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TSCA 8(E) SUPPLEMENTAL INFORMATION ON:
N-ethyl perfluorooctanesulfonamidoethanol (CASRN 1691-99-2); Docket Ref -0373

Dear Sir:

3M is submitting the final report from Covance Laboratories on the 2–year rat feeding study on N-ethyl perfluorooctanesulfonamidoethanol (CASRN 1691-99-2), originally notified to EPA in a letter dated December 4, 2000, with additional information provided in a letter dated May 24, 2001. In addition, two reports on serum and tissue analysis from samples collected during the study are enclosed.

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Sprague-Dawley rats were given N-ethyl perfluorooctanesulfonamido ethanol (98.1 %) in the diet at 0, 1, 3, 30, 100 & 300 ppm for up to 104 weeks. A recovery group at 100 ppm was dosed for 52 weeks, after which, untreated feed was given until termination. Interim sacrifices occurred at 4, 14 and 53 weeks for histopathology, metabolite quantitation, and cell proliferation. The 300-ppm groups were terminated after seven weeks of treatment. Through term, survival was unaffected by treatment, and bodyweights were lower in the 100 ppm dose group. Liver effects occurred at 100 ppm, with the exception of cystic degeneration in 30-ppm males. Hepatotoxicity was not present in 100-ppm recovery animals. In males, thyroid follicular cell adenoma was increased at the 100 ppm dose. For females, hepatocellular adenoma was increased in the 100-ppm and the 100-ppm-recovery groups.

For further information on this study contact Dr. John Butenhoff, 651-733-1962.

Sincerely,

Larry R. Johnson

Larry R. Johnson
Director, Corporate Toxicology

8EHQ-80-373

Enclosures

EPA-OTS
000811840M

Reports Submitted with Letter of February 8, 2002:

a. Final Report, 104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats, (Covance Study 6329-212), December 13, 2001.

b. Analytical Laboratory Report, Determination of the Presence and Concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556 and PFOSEA in Serum and Liver Samples of Crl:CD(SD) IGS BR Rats Exposed to N-Ethyl Perfluorooctanesulfonamido Ethanol, 3M Environmental Laboratory Report No. TOX-001, Laboratory Request No. U2103, 3M Refernce No. T-6316.1, June 6, 2001.

c. Analytical Laboratory Report, Determination of the Presence and Concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in Serum and Liver Samples of Crl:CD(SD) IGS BR Rats Exposed to N-Ethyl Perluorooctanesulfonamido Ethanol, 3M Environmental Laboratory Report No. TOX-003, Laboratory Request No. U2104, 3M Reference No. T-6316.1, June 6, 2001.

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
LRN-U2103

Study Title

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Analytical Laboratory Report Title

Determination of the Presence and Concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in Serum and Liver Samples of Crl:CD®(SD) IGS BR Rats Exposed to N-Ethyl Perfluorooctanesulfonamido Ethanol

Data Requirement

Not Applicable

Author

3M Environmental Laboratory

Study Completion Date

June 06, 2001

Performing Laboratories

Liver and Serum Analyses

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

Project Identification

3M Medical Department Study: T-6316.1
Covance In-Life Study: 6329-212
Analytical Report: FACT TOX-001
3M Laboratory Request No. U2103

Total Number of Pages

310

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
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This page has been reserved for specific country requirements.

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
LRN-U2103

GLP Compliance Statement

Analytical Laboratory Report Title: Determination of the Presence and Concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in Serum and Liver Samples of Crl:CD®(SD) IGS BR Rats Exposed to N-Ethyl Perfluorooctanesulfonamido Ethanol

Study Identification Numbers: T-6316.1, FACT TOX-001, LRN-U2103

This study was conducted in compliance with United States Food and Drug Administration (FDA) Good Laboratory Practice (GLP) Regulations 21 CFR Part 58, with the exceptions in the bulleted list below.

Exceptions to GLP compliance:

- There were two study directors in this study. This study was designed as two separate studies. The in-life phase study was considered to end at the generation and shipment of specimens. The analytical study was considered to start at the receipt of these specimens for analysis. This resulted in having two separate study directors, one for each phase of the same study. However, since the technical performance of each phase was entirely separate, no effect is expected from this exception.
- Sample storage stability will not be determined.
- Characterization of the analytical standards is underway, but has not yet been completed (21 CFR 58.105 (a)). Lot No. 59905 of the surrogate, THPFOS, will not be characterized for purity since quantities of this standard are exhausted.
- The electronic data systems in use have not been validated and there is not an electronic audit trail of corrections currently available (21 CFR 58.130 (e)). Authenticated hard copies of chromatograms and associated documents will be considered as the original raw data.
- Some changes in the raw data entries were not all made in accordance with 21 CFR 58.130 (e).
- Some reagents and solutions did not have expiration dates on the labels, as required according to 21 CFR 58.83.

(See next page for signatures)

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
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GLP Compliance Statement (continued)

June 6, 2001

John L. Butenhoff, Ph.D., *Study Director* Date

6 June 2001

Marvin T. Case, D.M.V., Ph.D., *Sponsor Representative* Date

05/31/01

Kristen J. Hansen, Ph.D., *Principal Analytical Investigator* Date

05/31/01

William K. Reagen, Ph.D., *Laboratory Manager* Date

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
LRN-U2103

GLP Study—Quality Assurance Statement

Analytical Laboratory Report Title: Determination of the Presence and Concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in Serum and Liver Samples of Crl:CD®(SD) IGS BR Rats Exposed to N-Ethyl Perfluorooctanesulfonamido Ethanol

Study Identification Numbers: T-6316.1, FACT TOX-001, LRN-U2103

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

Inspection Dates	Phase	Date Reported to	
		Management	Study Director
4/7/00–4/11/00	In-phase	5/8/00	5/8/00
1/15/01–2/4/01	Data	2/5/01	2/5/01
3/23/01, 3/26/01–3/30/01	Data	3/30/01	3/30/01
3/26/01–3/27/01, 3/29/01–3/30/01, 4/2/01–4/5/01	Data	4/6/01	4/6/01
4/3/01–4/6/01, 4/12/01–4/13/01, 4/17/01	Data	4/18/01	4/18/01
5/3/01, 5/7/01–5/10/01, 5/15/01–5/16/01	Draft Report	5/16/01	5/16/01

Ken Ost 6-11-01

QAU Representative Date

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*Contract lab professional service employees

Location of Archives

All original raw data, protocol, and analytical report have been archived at the 3M Environmental Laboratory. The test article and analytical reference standard reserve samples, as well as the specimens pertaining to the analytical phase of this study are archived at the 3M Environmental Laboratory.

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LRN-U2103

Introduction and Purpose

The purpose of the study is to determine the presence and concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in serum and liver specimens collected from Covance Study No.: 6329-212 titled: 104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats. The Covance in-life study was initiated on January 21, 1998. The analytical phase of the study was initiated on April 9, 1998.

Test System

A total of 410 males and 410 female rats were used as the test system. Table 1 outlines the rat population demographics and dosage levels for study 6329-212.

The test system species and strain selected was the Crl:CD®(SD) IGS BR rat received from Charles River Laboratories, Inc., identified using an implanted microchip device. At the initiation of treatment the rats were approximately 45–51 days old and weighed between approximately 100–310 g.

Table 1. Test System Population Demographics for Study 6329-212

Study Group	Number of Animals		Approximate Dietary Levels of EtFOSE-OH (ppm)
	Male	Female	
Group 1 Control	70	70	0
Group 2 Low	60	60	3
Group 3 Mid	60	60	30
Group 4 Mid-High	70	70	100
Group 5 High[a]	70	70	300
Group 6 Mid-High Recovery[b]	40	40	100
Group 7 High Recovery[a]	40	40	300

[a] Groups 5 and 7 were terminated during week 8 of treatment due to toxicity.
[b] Group 6 was placed on recovery after 52 weeks of treatment.

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Specimen Collection and Analysis

Specimens were collected by Covance (study 6329-212) and sent to the 3M Environmental Laboratory for analysis. Table 2 lists the sampling intervals and types of specimens that were collected for this study.

Table 2. Specimen Collection for FACT TOX-001

Week #	Number and Type of Specimens	Group # and Sex of Animal
4	50 serum and liver specimens	Groups 1–5 males and females
8	40 serum and 20 liver specimens	Groups 1 and 5 males and females
14	120 serum and 40 liver specimens	Groups 1–4 males and females
27	80 serum specimens	Groups 1–4 males and females
53	100 serum and 20 liver specimens	Groups 1 and 4 males and females
105	156 serum and 157 liver specimens	Groups 1–4 and Group 6 males and females

The total number and type of specimens collected for analyses in the analytical phase of this study are presented below.

Specimens Collected from Study Groups 1 through 5 (through 2/01/00):
Serum Specimens—524 specimens (423 samples were analyzed.)
Liver Specimens—265 specimens (All 265 samples analyzed.)

Specimens Collected from Study Group 6 (Recovery) on 2/01/00:
Serum Specimens—22 specimens (All 22 samples analyzed.)
Liver Specimens—22 specimens (All 22 samples analyzed.)

Liver and sera specimens were shipped to the 3M Environmental Laboratory frozen and on dry ice.

Sera and liver samples were extracted beginning on April 23, 1998 using an ion pairing reagent and either ethyl acetate or methyl-*tert*-butyl ether (MtBE). Liver samples were homogenized prior to the extraction procedure. Sample extracts were analyzed using high-pressure liquid chromatography-electrospray/tandem mass spectrometry (HPLC-ESMSMS) in the multiple reaction monitoring mode. PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA levels were evaluated by external calibration using extracted curves. Analytical details are included in this report.

Specimen Receipt and Maintenance

The 3M Environmental Laboratory received liver and serum specimens collected at predetermined time points during and at the end of the *in-life* phase of Covance Study 6329-212 from February 1998 through February 2000 from Covance. All specimens were received frozen on dry ice and were immediately transferred to storage at -20°C ±10°C.

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Control matrices used in liver and sera analyses performed during TOX-001 were obtained from commercial sources and are presented in Appendix A (see Tables 7 and 8). Samples analyzed at the 3M Environmental Laboratory will be maintained for a period of 10 years and will be stored at the laboratory at -20°C ±10°C to -80°C ±20°C.

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Chemical Characterization of the Reference Materials

Chemical characterization information on the analytical reference materials used in this study is presented in tabular form below.

Table 3. Characterization of the Analytical Reference Standards in Study FACT TOX-001

Reference Standard / Formula	Acronym	Source	Expiration Date	Storage Conditions	Chemical Lot Number	Physical Description	Purity
Potassium Perfluorooctanesulfonate $C_8F_{17}SO_3$-K+	KPFOS[a]	3M	08/31/01	Ambient temperature	171	Light colored powder	86.4%
		3M	2010	Ambient temperature	193	White crystals	88.0%
		3M	2010	Ambient temperature	215	White powder	TBD[c]
N-Ethyl Perfluorooctanesulfonamido ethyl alcohol $C_8F_{17}SO_2N(C_2H_5)CH_2CH_2OH$	EtFOSE-OH	3M	2010	Ambient temperature	936	Amber waxy solid	NA*
		3M	11/26/01	Ambient temperature	Unknown (SD013)	Amber waxy solid	88.9%
Sodium Perfluorooctanesulfonylamido(ethyl) acetate $C_8F_{17}SO_2N(CH_2CH_3)CH_2COO$-Na	PFOSAA[b]	3M	01/01/2010	Ambient temperature	617	Yellow to amber liquid	TBD[c]
Perfluorooctanesulfonylamido(ethyl)acid $C_8F_{17}SO_2N(CH_2CH_3)CH_2COO$- H		3M	01/01/2010	Ambient temperature	NB 112999-99	Tan waxy solid	TBD[c]
Perfluorooctanesulfonylamide $C_8F_{17}SO_2NH_2$	PFOSA	3M	01/01/2010	Ambient temperature	L2353	Amber to brown waxy solid	TBD[c]
		3M	2010	Ambient temperature	NR (TN-A-1886)	NR	TBD[c]
		3M	01/01/2010	Ambient temperature	L15709	Light yellow waxy solid	TBD[c]
Perfluorooctanesulfonylethylamide $C_8F_{17}SO_2NHCH_2CH_3$	PFOSEA	3M	01/01/2010	Ambient temperature	529	Amber waxy solid	TBD[c]
M556 $C_8F_{17}SO_2N(H)CH_2COOH$	M556	3M	01/01/2010	Ambient temperature	NB113047-80	White powder	TBD
1H, 1H, 2H, 2H-Tetrahydroperfluorooctanesulfonic acid $C_8H_4F_{13}SO_3H$	THPFOS	ICN	01/01/2010	Ambient temperature	59909	Brown powder	NA*
		ICN	01/01/2010	Ambient temperature	53406	Brown Waxy Solid	TBD[c]

*This lot is exhausted and cannot be characterized.
NR—Not recorded
NA—Not applicable
TBD—To be determined

[a]Target analyte is PFOS, $C_8F_{17}SO_3^-$
[b]Target analyte is $C_8F_{17}SO_2\{(CH_2CH_3)(CH_2COO^-)\}$
[c]Unless otherwise indicated, at the time of quantitation, the purity for all analytes was assumed to be 100%.

Dose Confirmation Analyses

Dose preparation methods and analysis were performed by Covance, using a validated analytical method provided by the Sponsor (MP-M312-MA), and are reported separately (Reference Covance 6329-212).

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Method Summaries

Following is a brief description of the methods used during this analytical study by the 3M Environmental Laboratory. Detailed descriptions of the methods used in this study are located in Appendix C.

Data collected prior to November 1999 was reworked in 2000 to accommodate improvements in data reduction methods. Both the original and "reworked" data are archived; reworked data is presented in the final results. The improved methods are documented in the form of method modifications.

As the present study progressed, more advanced methods evolved and these methods were used with deviations until amendments to the protocol were written. Protocol and method deviations are located in Appendix B of this report.

3M Environmental Laboratory

PREPARATORY METHODS

- **FACT-M-1.0**. "Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry." This method was used for week 4, week 8, and week 14 samples.
- **FACT-M-3.0**, "Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry." This method was used for week 4, week 8, and week 14 samples.
- **ETS-8-4.1**, "Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry." This method was used for week 27, week 53, and week 105 samples.
- **ETS-8-6.0**, "Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry." This method was used for week 53 and week 105 samples.

  An ion-pairing reagent was added to the sample and the analyte ion pair was partitioned into ethyl acetate (FACT-M-1.0 and FACT-M-3.0) or MtBE (ETS-8-4.1 and ETS-8-6.0). The extract was transferred to a centrifuge tube and put onto a nitrogen evaporator until dry. Each extract was reconstituted in 1.0 mL of methanol, and then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
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ANALYTICAL METHODS

- **FACT-M-2.0**, "Analysis of Liver Extracts for Fluorochemicals Using HPLC-Electrospray/Mass Spectrometry".
- **FACT-M-4.0**, "Analysis of Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry".
- **ETS-8-5.1**, "Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry".
- **ETS-8-7.0**, "Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry".

The analyses were performed by monitoring one or more product ions selected from a single primary ion characteristic of a particular fluorochemical using HPLC-ES/MS/MS. For example, molecular ion 499, selected as the primary ion for PFOS ($C_8F_{17}SO_3$-) analysis, was fragmented further to produce ion 99 (FSO_3-). The characteristic product ion 99 was monitored for quantitative analysis.

ANALYTICAL EQUIPMENT

The actual analytical equipment settings used in the present analytical phase of this study varied slightly during actual data collection. The following is representative of the settings used during the analytical phase of this study.

Liquid Chromatograph: Hewlett-Packard® Series 1100 Liquid Chromatograph system
Analytical column: Keystone® Betasil™ C_{18} 2x50 mm (5 µm)
Column temperature: Ambient
Mobile phase components:
Component A: 2mM ammonium acetate
Component B: methanol
Flow rate: 300 µL/min
Injection volume: 10 µL
Solvent Gradient: 13.5 minutes

Time (minutes)	*%B*
0.0	40%
8.5	90%
11.0	90%
12.0	40%
13.5	0%

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
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Mass Spectrometer: Micromass® API/Mass Spectrometer Quattro II™ Triple Quadrupole system
Software: Mass Lynx™ 2.3, 3.1, 3.2, 3.3, 3.4
Cone Voltage: 30–60 V
Collision Gas Energy: 25–45 eV
Mode: Electrospray Negative
Source Block Temperature: 150°C ±10°C
Electrode: Z-spray
Analysis Type: Multiple Reaction Monitoring (MRM)

Table 4. Target Ions Monitored in 3M Laboratory Analyses

Target Analyte	Primary Ion (AMU)	Product Ion (AMU)
PFOS	499	80, 99, 130
PFOSA	498	78
PFOSAA	584	83, 169
EtFOSE-OH	630	59
PFOSEA	526	65
M556	556	65, 78, 83, 169
THPFOS	427	80

Data Quality Objectives and Data Integrity

The following data quality objectives were indicated in the method performance section of ETS-8-5.1, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry and ETS-8-7.0, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry:

- **Linearity:** The coefficient of determination (r^2) equal to or greater than 0.980.
- **Limits of Quantitation (LOQ):** The LOQ is equal to the lowest acceptable standard in the calibration curve.
- **Acceptable Precision:** Precision is better than 30% for the method.
- **Acceptable Spike Recoveries:** 70–130%

Data Summary, Analyses, and Results

Data quality objectives for the analytical phase of this study outlined in the 3M Environmental Laboratory protocol for FACT TOX-001 (see Appendix B) were met with the exceptions noted in this report. See Appendix B for deviations.

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
LRN-U2103

Summary of Quality Control Analyses Results for PFOS, PFOSA, PFOSAA, M556, EtFOSE-OH and PFOSEA

- **Linearity:** The coefficient of determination (r^2) of the standard curve was ≥0.980.
- **Calibration Standards:** Quantitation of the target analytes was based on linear regression analysis weighted 1/x of a single, opening or two bracketing extracted matrix curves for each group of samples. Rat and rabbit sera were used for matrix curves for sera analyses, while rabbit liver was used for matrix curves for liver analyses. High or low points on the curve may have been deactivated to provide a better linear fit over the curve range most appropriate to the data. Low curve points with peak areas less than two times that of the extraction blanks were deactivated to disqualify a data range that may have been significantly affected by background levels of the analyte. Occasionally, a single mid-range curve point that was an obvious outlier may have been deactivated. Quantitation of each analyte was based on the response of one or more specific product ion(s) using the multiple reaction monitoring mode of the instrument (see Appendix C, Analytical Methods). Calibration standards were prepared to run, undiluted, approximately within the linear range of the instrument (approximately 5–1000 ng/g).
- **Limits of Quantitation (LOQ):** The LOQ is equal to the lowest acceptable standard in the calibration curve (defined as a standard within ±30% of the theoretical value), and is at least two times the analyte peak area detected in the surrogate matrix blanks. (see Appendix D).
- **Blanks:** All blanks were below the limit of quantitation for the compounds of interest, except PFOSA Week 8 H_2O + liver Blk-2 (0.0663 µg/g and 0.0414 µg/g) and PFOS Week 105 liver-Blk-2 (0.0163). To simplify analyses that were complicated by endogenous levels of fluorochemicals in unexposed rat sera and liver, rabbit sera and liver were selected as suitable surrogate matrices.
- **Precision, Instrumental:** Instrumental precision was determined by replicate injections of a single serum extract. Instrumental precision was determined for PFOS, PFOSAA, and M556; variation was less than 7.0 % for all targeted analytes.
- **Matrix Spikes:** Sera—Matrix spike samples were prepared from control rat sera along with each batch of sera samples. Samples were spiked between approximately 75–250 ng/mL, levels that approximate the levels detected in the Group 1—Group 5 samples, depending upon the analyte. All spikes were prepared to run, undiluted, within a ten-fold limit of the linear range of the calibration curve. For some analytes, samples for some high dose animals may be much higher than the range of these prepared spikes. Sera matrix spike recoveries are presented in tabular form in Table 5.

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Table 5. Matrix Spikes—Sera

	# of Spikes	Average Spike Recovery ± SD	Range	# of Spikes Deviating > ±30% and < ±50%	# of Spikes Deviating > ±50%
PFOS	28	**95 ± 25%**	65–183%	3	1
PFOSA	28	**103 ± 20%**	72–143%	4	0
PFOSAA	28	**111 ± 24%**	57–186%	3	1
EtFOSE-OH	22	**39 ± 29%**	18–127%	0	20
M556*	22	**126 ± 22%**	86–153%	8	3
PFOSEA	22	**63 ± 14%**	37–87%	8	6

*M556 was identified as a metabolite of EtFOSE-OH after initiation of the analytical work. Standard material was unavailable and had to be synthesized. No M556 data is provided for the Week 4 through Week 14.

NOTE: The matrix spikes for EtFOSE-OH and PFOSEA were inconsistent or were not conducted with all groups of samples and indicate that data presented for these two analytes should be considered qualitative only.

Liver—Matrix spike samples were prepared from control rat liver along with each batch of liver samples. Samples were spiked between approximately 100–1000 ng/g, levels that approximate the levels detected in the Group 1—Group 5 samples, depending on the analyte. All spikes were prepared to run, undiluted, within a ten-fold limit of the linear range of the calibration curve. For some analytes, samples for some high dose animals may be much higher than the range of these prepared spikes. Liver matrix spike recoveries are presented in tabular form in Table 6.

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Table 6. Matrix Spikes—Liver

	# of Spikes	Average Spike Recovery ± SD	Range	# of Spikes Deviating > ±30% and < ±50%	# of Spikes Deviating > ±50%
PFOS	24	90 ± 61%	0–196%	4	9
PFOS, revised (1)	18	**119 ± 38%**	60–196%	4	3
PFOSA	18	94 ± 27%	26–128%	0	2
PFOSA, revised (1)	16	**102 ± 17%**	71–128%	0	0
PFOSAA	20	86 ± 44%	0–147%	1	4
PFOSAA, revised (1)	16	**105 ± 19%**	74–147%	1	0
EtFOSE-OH	16	133 ± 166%	25–567%	1	8
EtFOSE, revised (1)	14	**148 ± 173%**	26–567%	1	6
M556*	12	**102 ± 15%**	85–141%	1	0
PFOSEA	10	**97 ± 12%**	80–117%	0	0

*M556 was identified as a metabolite of EtFOSE-OH after initiation of the analytical work. Standard material was unavailable and had to be synthesized. No M556 data or QC is provided for the Week 4 through Week 14 samples.

[1]NOTE: After matrix spikes had been prepared, unexpectedly high levels of the analytes were identified in the control animals. Spikes were originally prepared at either approximately 100 ng/g or 250 ng/g. Both of these levels were unacceptably low as they were less than 30% of the endogenous levels found in the control animals. Data from these MS/MSDs were not included in the revised data presented above.

NOTE: The matrix spikes for EtFOSE-OH and PFOSEA were inconsistent or were not conducted with all groups of samples and indicate that data presented for these two analytes should be considered qualitative only.

- **Surrogates:** The surrogate (THPFOS) was added to all samples and standards. THPFOS was not used for quantitation, but was used to monitor for gross instrument failure. After 11/04/99, the surrogate response of each analytical run was verified to determine that it did not vary more than ±50% from the mean within each analytical run. Deviations of greater than ± 50% are noted in the results table.

Statement of Data Quality

It is not possible to verify true recovery of endogenous analyte from tissues without radio-labeled reference material. The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. For example, in liver, PFOS data is accurate to 119 ± 38%. Please see bolded values in Tables 4 and 5 for specific values relating to data quality.

The results of quality control analyses (curve fit, CCVs, and MS/MSDs) for EtFOSE-OH and PFOSEA were inconsistent and indicate that data presented for these two analytes should be

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considered to be qualitative only. Values for these two analytes are presented in data tables in the spirit of full disclosure, but should not be used in any quantitative assessment of the data.

Summary of Sample Results

Some PFOS results (those obtained using lot 171) have been corrected for purity of the analytical reference material. Uncorrected results are noted in the data tables.

- **Samples from Control Animals:** The target analytes were often detected in the sera and liver samples from the control animals. These levels were usually lower than those found in the low dose test animals.
- **Samples from Dosed Animals:** In general, levels of the target analytes present in the sera and liver samples from the test animals increased with dose group, with the exception of Group 6, which was taken off the compound after 53 weeks. Detailed sample data tables are presented in Appendices D and E.

Statistical Methods and Calculations

Statistical methods were limited to the calculation of means and standard deviations. See Appendix F for example calculations used to generate the liver and serum sample data in FACT TOX-001.

Statement of Conclusion

Under the conditions of the present study, PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA were observed in the sera and liver of rats dosed with N-Ethyl Perfluoroctanesulfonamido Ethanol during the in-life phase of the study.

References

Covance Study No.: 6329-212, 104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluoroctanesulfonamido Ethanol in Rats

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Appendix A: Chemical Characterization and Control Matrices

Table 7. Characterization of the Control Matrices Used for Sera Analyses in Study FACT TOX-001

Control Matrix	Rat Serum	Rat Serum	Rabbit Serum
Source	Sigma	Sigma	Sigma
Expiration Date	2010	2010	2010
Storage Conditions	-20°C ±10°C	-20°C ±10°C	-20°C ±10°C
Chemical Lot #	17H9306	19H89291	47H4641
Physical Description	Rat Serum	Rat Serum	Rabbit Serum

NR—not recorded

Table 8. Characterization of the Control Matrices Used for Liver Analyses in Study FACT TOX-001

Control Matrix	Rabbit Liver	Rabbit Liver	Rabbit Liver	Rabbit Liver	Rabbit Liver
Source	Covance Laboratories, Inc.*	Covance Laboratories, Inc.*	Covance Laboratories, Inc.*	Covance Laboratories, Inc.*	Unknown
Expiration Date	2010	2010	12/99	2010	12/01/99
Storage Conditions	-20°C ±10°C	-20°C ±10°C	-20°C ±10°C	-20°C ±10°C	-20°C ±10°C
Chemical Lot #	F00014	F00008	F00007	F00016	NR (TCR-99062-22)
Physical Description	Rabbit Liver	Rabbit Liver	Rabbit Liver	Rabbit Liver	Rabbit Liver

NR—not recorded
*The control source is listed as CHW (Corning Hazelton Wisconsin) in the raw data. This is the former name of Covance Laboratories, Inc.

Table 9. Characterization of Test Article in Study FACT TOX-001

	Test Article
Chemical Name	**EtFOSE-OH** N-Ethyl Perfluorooctanesulfonamido ethanol
Source	3M
Expiration Date	11/26/01
Storage Conditions	Ambient temperature
Chemical Lot #	30035, 30037, 30039
Physical Description	Waxy solid
Purity	97.4%*

*This purity value is from the interim COA. The in-life report lists the purity as 98.1%; however, the COA is considered the more accurate purity.

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Appendix B: Protocol, Amendments and Deviations

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COVANCE
THE DEVELOPMENT SERVICES COMPANY

Sponsor:

3M
St. Paul, Minnesota

PROTOCOL

Study Title:

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Date:

January 21, 1998

Performing Laboratory:

Covance Laboratories Inc.
3301 Kinsman Boulevard
Madison, Wisconsin 53704

Laboratory Study Identification:

Proposal No. 6777

Covance 6329-212

Study
104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Purpose
To assess the carcinogenicity of the test material when administered in the diet to rats for at least 104 weeks

Sponsor
3M
Toxicology Services
Building 220-2E-02, 3M Center
St. Paul, Minnesota 55144-1000

Study Monitor
Andrew M. Seacat, PhD
3M
Telephone No.: 612.575.3161
Facsimile No.: 612.733.1773

Study Location
Covance Laboratories Inc.
3301 Kinsman Boulevard
Madison, Wisconsin 53704

Mailing Address: PO Box 7545
Madison, Wisconsin 53707

Study Director
Peter J. Thomford, PhD
Covance Laboratories Inc.
Telephone No.: 608.241.7207
Facsimile No.: 608.242.2736

Toxicologist
Thomas E. Ryan, BS
Covance Laboratories Inc.

Proposed Study Timetable
In-Life Start Date: January 26, 1998
In-Life End Date: January 31, 2000

Regulatory Compliance
This study will be conducted in compliance with the Food and Drug Administration Good Laboratory Practice Regulations as set forth in Title 21 of the US Code of Federal Regulations, Part 58, issued December 22, 1978 (effective June 20, 1979), and with any applicable amendments.

Animal Care and Use Statement
All procedures in this protocol are in compliance with the Animal Welfare Act Regulations, 9 CFR 1-4. In the opinion of the Sponsor and study director, the study does not unnecessarily duplicate any previous work.

Quality Assurance
The protocol, study conduct, and final report will be audited by the Covance Quality Assurance Unit (QAU). The proliferation cell nuclear antigen evaluation, data, and report will be audited by the QAU of Pathology Associates International.

Test Material

Identification
T-6316 (Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol, NEtFOSE)

Lot Numbers
The lot numbers will be maintained in the raw data.

Purity
98.1% NEtFOSE (w/w)

Stability
Responsibility of the Sponsor

Storage Conditions
At room temperature

Characteristics
Information on synthesis methods, composition, or other characteristics that define the test material is on file with the Sponsor.

Reserve (Archive) Samples
A reserve sample (approximately 5 g) will be taken and stored at room temperature. This sample will be transferred to the Sponsor after completion of the in-life phase to be retained in accordance with 21 CFR 58.195.

Disposition of Test Material
After authorization from the Sponsor, any remaining test material will be returned to:

Andrew M. Seacat, PhD
3M
Toxicology Services
Building 220-2E-02, 3M Center
St. Paul, Minnesota 55144-1000
Telephone No.: 612.575.3161
Facsimile No.: 612.733.1733

Animals

Species
Rat

Strain
Crl:CD®(SD)BR

Source
Charles River Laboratories, Inc., Raleigh, North Carolina

Age at Initiation of Treatment
Preferably 6 weeks of age, but not more than 8 weeks of age

Weight at Initiation of Treatment
100 to 300 g

Number and Sex
410 males and 410 females

Identification
Implantable microchip identification device

Husbandry

Housing
Individual (may be group-housed during acclimation)

Diet
Certified Rodent Diet #5002 (PMI® Feeds, Inc.) *ad libitum,* unless otherwise specified. The diet is routinely analyzed by the manufacturer for nutritional components and environmental contaminants. Specified nutrient and contaminant analyses are on file at Covance-Madison.

Water
Ad libitum. Samples of the water are routinely analyzed for specified microorganisms and environmental contaminants. The results are on file at Covance-Madison.

Contaminants
There are no known contaminants in the diet or water at levels that might interfere with this study.

Environment

Environmental controls for the animal room will be set to maintain 18 to 26°C, a relative humidity of 30 to 70%, a 12-hour light/12-hour dark cycle, and minimum of 10 room air changes/hour. The light/dark cycle may be interrupted to accommodate study-related activities.

Acclimation

At least 1 week

Randomization

Selection of animals for the study will be based on body weights, clinical observations, and other data as appropriate. Animals will be assigned to treatment groups using a computerized blocking procedure designed to achieve body weight balance with respect to treatment groups. At the time of randomization, the weight variation of the animals of each sex used will not exceed ±2 standard deviations of the mean weight, and the mean body weight for each group of each sex will not be statistically different at the 5.0% probability level.

Justification

Rats historically have been used in safety evaluation studies and are recommended by appropriate regulatory agencies.

Group Designations and Dietary Levels

Group	Number of Animals Male	Number of Animals Female	Dietary Levels (ppm NEtFOSE)[a]
1 (Control)[b,c,d]	70	70	0
2 (Low)[c]	60	60	3
3 (Mid)[c]	60	60	30
4 (Mid-High)[c,d]	70	70	100
5 (High)[c,d]	70	70	300
6 (Mid-High Recovery)[e]	40	40	100
7 (High Recovery)[e]	40	40	300

a T-6316 is 98.1% n-ethyl perfluorooctanesulfonamido ethanol (NEtFOSE); dose levels are expressed as ppm of NEtFOSE.
b The control animals will receive the basal diet only.
c Five animals/sex in Groups 1 through 5 will be sacrificed during Weeks 3 and 14 for hepatocellular proliferation rate measurements and biochemical analyses (palmitoyl-CoA oxidation).
d Ten animals/sex in Groups 1, 4, and 5 will be designated as interim sacrifice animals and will be sacrificed after at least 78 weeks of treatment.
e Animals in Groups 6 and 7 will be treated for at least 78 weeks, then treatment will be discontinued, and the animals will be observed for reversibility, persistence, or delayed occurrence of toxic effects for at least 26 weeks posttreatment. During recovery, the animals will receive basal diet only.

Dosing Procedures

Method of Administration

Dietary. Animals in Groups 1 through 5 will receive test diet for at least 104 weeks. Animals in Groups 6 and 7 will receive test diet for 78 weeks only.

Reason for Dosing Route

The potential human exposure is by the oral route.

Dose Preparation

Before initiation of treatment, all dose preparations will be mixed once, and low- and high-dose preparations will be mixed four times for determination of homogeneity and stability.

During treatment, all dose preparations will be mixed at least once every 4 weeks according to the study-specific mixing procedure developed by Covance. Dose concentrations will be based on the NEtFOSE content as supplied; the Sponsor has stated the T-6316 is 98.1% NEtFOSE (w/w) as supplied. All dose preparations will be stored at room temperature.

Retention Samples

Samples (approximately 100 g) will be taken from each dose preparation during the in-life phase and stored at room temperature. Unless used for analyses, these samples will be discarded at least 1 month after completion of the in-life phase.

Dose Analyses

By Covance using a method supplied by the Sponsor and validated by Covance

Homogeneity

Homogeneity will be determined for all dose level preparations once pretest and for the preparations for Weeks 1 through 3; for two additional pretest high-dose level preparations; and for four additional pretest low-dose level preparations. One sample (approximately 100 g) each from the top, middle, and bottom of the dose preparations mixed for homogeneity analyses will be collected, divided into three subsamples for extraction and analysis, and analyzed for test material content. All samples will be stored at room temperature until analyzed within 7 days of mixing. Homogeneity analysis will be repeated if batch size changes by more than 30%.

Stability

Four sets of samples (approximately 100 g each) will be taken from the low- and high-dose level concentrations of diet preparations mixed pretest to establish stability. One set will be analyzed on the day of mixing. One set will be stored at room temperature for at least 19 days, then analyzed. The third set will be stored at room temperature after at least 32 days, then analyzed. The remaining set will be stored in a freezer set to maintain -10 to -30°C for 8 weeks, then analyzed.

In addition, samples (approximately 100 g each) will be taken from the low- and high-dose level concentrations of diet preparations mixed pretest. The samples will be stored at room temperature for 5 days, then for 7 days under animal room conditions (room temperature in a feeding container), then analyzed.

Dose Confirmation

Samples (approximately 100 g each) will be collected from all dose preparations and analyzed in duplicate. Homogeneity samples collected from the middle of the dose preparations for Weeks 1 through 3 will be used for dose confirmation results. All samples will be stored at room temperature until analyzed.

Observation of Animals

Clinical Observations

Each animal will be observed twice daily (a.m. and p.m.) for mortality and moribundity, recording findings as they are observed.

Once prior to treatment and weekly thereafter, each animal will be removed from its cage and examined; abnormal findings or an indication of normal will be recorded. The following information on each grossly visible or palpable mass will be recorded.

time of onset
location
size (small or large)
appearance
progression

Body Weights

Prior to treatment (at randomization), weekly for Weeks 1 through 17, once every 4 weeks thereafter, and at Week 105

Food Consumption
Weekly for Weeks 1 through 16 and once every 4 weeks thereafter

Clinical Pathology

Frequency and Number of Animals

Unscheduled Collection
When possible, a blood film will be made and held for possible future examination from animals sacrificed at unscheduled intervals.

Scheduled Collections
Hematology, clinical chemistry, urinalysis, urine chemistry, and serum sampling will be done on 10 animals/sex/group in Groups 1 through 5 during Weeks 14, 27, and 53.

A blood film will be made and held for possible future examination for animals at scheduled sacrifices after at least 78 and 104 weeks of treatment.

Method of Collection

Hematology, Clinical Chemistry, Urinalyses, Urine Chemistry, Serum Samples
Animals will be fasted overnight; blood will be collected from a jugular vein. The anticoagulant will be potassium EDTA for hematology tests. Samples for clinical chemistry and serum samples will be collected without anticoagulant. Urine will be collected chilled overnight (approximately 16 hours).

Blood Films
Blood films will be taken as part of the necropsy procedure.

Tests

Hematology

red blood cell (erythrocyte) count
hemoglobin
hematocrit
mean corpuscular volume
mean corpuscular hemoglobin
mean corpuscular hemoglobin concentration
platelet count
white blood cell (leukocyte) count
differential blood cell count
blood cell morphology
reticulocyte smear (made, but not examined)

Clinical Chemistry

glucose
urea nitrogen
creatinine
total protein
albumin
globulin
cholesterol
total bilirubin
alanine aminotransferase
gamma glutamyltransferase
aspartate aminotransferase
calcium
inorganic phosphorus
sodium
potassium
chloride

Urinalysis

appearance
volume
specific gravity
pH
protein
urobilinogen
glucose
ketones
bilirubin
blood
microscopic examination of sediment

Urine Chemistry

sodium
potassium
16 hour excretion of:
sodium
potassium

Serum Samples

Serum not used for clinical chemistry will be stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to:

Kris J. Hansen, PhD
3M Environmental Technology and Safety Services
935 Bush Avenue
Building 2-3E-09
St. Paul, Minnesota 55133-3331
Telephone No.: 612.778.6018
Facsimile No.: 612.778.6176

Samples will be retained by the Sponsor for possible future analysis.

Hepatocellular Proliferation and Biochemical Analyses

Frequency and Number of Animals

Five animals/sex in Groups 1 through 5 during Weeks 3 and 14

Cell Proliferation Tissue Collection and Immunohistochemical Evaluation

At each interval, animals will be fasted overnight, anesthetized with sodium pentobarbital, weighed, and exsanguinated. The abdominal cavity of each animal will be opened, and the liver will be removed, weighed, and representative samples of left lateral, right median, and right lateral lobes of the liver and any macroscopic lesions of the liver will be collected and preserved in zinc formalin.

After fixation, each sample of liver will be embedded in paraffin, and the paraffin blocks will be shipped to:

Sandra R. Eldridge, PhD
Pathology Associates International
15 Worman's Mill Court, Suite I
Frederick, Maryland 21701
Telephone No.: 301.663.1644, ext. 2201
Facsimile No: 301.663.8994

Proliferation cell nuclear antigen (PCNA) evaluation will be done on the samples. Results will be provided for inclusion in the final report.

Palmitoyl-CoA Oxidase Tissue Collection and Analyses
A sample (approximately 500 mg) of the right lateral lobe of the liver will also be collected from each animal and flash-frozen in liquid nitrogen. The liver tissue will be stored in a freezer set to maintain -60 to -80°C until analyzed by Covance for palmitoyl-CoA oxidase activity.

Animal Disposition
Animals will be discarded after liver collection.

Termination

Unscheduled Sacrifices and Deaths
Necropsies will be done. Animals to be sacrificed will be anesthetized with sodium pentobarbital, weighed, and exsanguinated. A blood film will be taken as part of the necropsy procedure for sacrificed animals.

Scheduled Sacrifices

Interim Sacrifice
After at least 78 weeks of treatment, 10 animals/sex from Groups 1, 4, and 5 will be fasted overnight, anesthetized with sodium pentobarbital, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Terminal Sacrifice
After at least 104 weeks of treatment, the remaining animals in Groups 1 through 5 will be fasted overnight, then anesthetized with sodium pentobarbital, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Recovery Sacrifice
After at least 78 weeks of treatment and 26 weeks without treatment, the remaining animals in Groups 6 and 7 will be fasted overnight, then anesthetized with sodium pentobarbital, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Postmortem Procedures

Necropsy

The necropsy will include an examination of the external features of the carcass; all external body orifices; the abdominal, thoracic, and cranial cavities; organs; and tissues.

Organ Weights

At the scheduled sacrifices, the following organs (when present) will be weighed; paired organs will be weighed separately:

adrenal (2)	ovary (2)
brain	spleen
kidney (2)	testes
liver	thyroid (2) with parathyroid
lung	uterus with cervix

Organ-to-body weight percentages and organ-to-brain weight ratios will be calculated.

Bone Marrow Smear

From the femur of each animal at scheduled sacrifices only; made but not examined

Tissue Preservation

The following tissues (when present) from each animal will be preserved in 10% neutral-buffered formalin:

adrenal (2)
brain
cecum
cervix
colon
duodenum
epididymis (2)
esophagus
eye (2)
femur with bone marrow (articular surface of the distal end)
Harderian gland
heart
ileum
jejunum
kidney (2)
lesions
liver
lung with mainstem bronchi
lymph node (mesenteric)
mammary gland (females only)
ovary (2)
pancreas
pituitary
prostate
rectum
salivary gland [mandibular (2)]
sciatic nerve
seminal vesicle (2)
skeletal muscle (thigh)
skin
spinal cord (cervical, thoracic, and lumbar)
spleen
sternum with bone marrow
stomach
testis (2)
thymus
thyroid (2) with parathyroid
trachea
urinary bladder
uterus
vagina

Histopathology

Tissues (as appropriate) from each animal in Groups 1, 5, and 7 and from each animal that dies or is sacrificed at an unscheduled interval will be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically.

Macroscopic lesions will also be examined microscopically from each animal in Groups 2, 3, 4, and 6.

Reports

One copy of each draft report will be sent to the Sponsor. The report will include the following information:

Experimental Design and Methods

Results
dose analyses
mortality
clinical observations
body weights
body weight changes
food consumption
test material consumption
clinical pathology results
palmitoyl CoA oxidase activities
macroscopic observations
microscopic observations
cell proliferation assessments (provided by the Sponsor's designee)

Statistical Evaluation
body weights
body weight changes
food consumption
survival rates
clinical pathology values
palmitoyl CoA oxidase activities
neoplastic and nonneoplastic lesions

Statistical methods will be those presented in Attachments Nos. 1 and 2. For each sex, Groups 2 through 7 will be compared to Group 1 (Control).

At the end of 1 year after issuance of the audited draft report, if no requested revisions or instructions to finalize have been communicated by the Sponsor, then the audited draft report will be considered 'final' and issued as the final report, signed by the study director, and submitted to the Sponsor.

Any modifications or changes to the audited draft report requested 1 year after issuance will be performed at additional cost to the Sponsor.

Two copies of the signed final report (one unbound and one bound) will be sent to the Sponsor.

Record Retention

All raw data, documentation, records, protocol, specimens, and final report generated as a result of this study, including those items listed below, will be archived in the storage facilities of Covance-Madison for a period of 1 year following submission of the final report to the Sponsor. All raw data stored on magnetic media, the protocol and protocol amendments, study correspondence, and the original report will be retained by Covance. One year after submission of the final report, all of the aforementioned materials will be sent to the Sponsor, and a return fee will be charged. The Sponsor may elect to have the materials retained in the Covance archives for an additional period of time, and Covance will charge a storage fee. If the Sponsor chooses to have Covance dispose of the materials, a disposal fee will be charged.

protocol and protocol amendments
dose preparation records
in-life records
  animal receipt
  acclimation
  animal room maintenance
  randomizations
  dose administration
  clinical observations
  body weights
  food consumption
  sample collection
clinical pathology records
anatomical pathology records
statistical analyses
study correspondence
tissue specimens (wet and in paraffin)
blood, bone marrow, and tissue slides
final report (original signed copy)

The following supporting records will be retained at Covance-Madison but will not be archived with the study data.

feed analysis records
water analysis records
animal room environment records
refrigerator and freezer temperature records
room temperature records for test material storage
instrument calibration and maintenance records

PCNA evaluation data and paraffin blocks and tissue slides for PCNA will be retained by Pathology Associates International.

Serum samples sent to the Sponsor will be retained by the Sponsor

PROTOCOL APPROVAL

Andrew M. Seacat, PhD
Study Monitor
3M

Date 1/27/98

Peter J. Thomford, PhD
Study Director
Department of Toxicology
Covance Laboratories Inc.

Date 1/21/98

Attachment No. 1

Statistical Analyses

The statistical methods that will be used are described below. Only data collected on or after the first day of treatment will be analyzed statistically.

One-way analysis of variance [ANOVA (Winer, 1971)] will be used (if applicable) to analyze body weights; body weight changes; food consumption; clinical chemistry and hematology values (except blood cell morphology); urine specific gravity, pH, and volume; urine chemistry values; palmitoyl CoA oxidase activities; organ weights; organ-to-body weight percentages; and organ-to-brain weight ratios.

Levene's test (Levene, 1960) will be done to test for variance homogeneity. In the case of heterogeneity of variance at $p \leq 0.05$, transformations will be used to stabilize the variance. ANOVA will be done on the homogeneous or transformed data. If the ANOVA is significant, Dunnett's t-test (Dunnett, 1964) will be used for pairwise comparisons between treated and control groups.

If the ANOVA shows significance for body weights at Week 1, one-way analysis of covariance [ANCOVA (Winer, 1971)] will be used to analyze body weights, with initial body weights as the covariate, using untransformed data. If the ANCOVA is significant, least squares means t-test (Winer, 1971) will be used for pairwise comparisons between treated and control groups.

Group comparisons will be evaluated at the 5.0%, two-tailed probability level.

References

Dunnett, C. W., "New Tables for Multiple Comparisons with a Control," Biometrics, 20:482-491 (1964).

Levene, H., "Robust Tests for Equality of Variances," Contributions to Probability and Statistics, (eds.) I. Olkin et al., Ch. 25, pp. 278-292, Stanford University Press: Stanford, California (1960).

Winer, B. J., Statistical Principles in Experimental Design, Second Ed., McGraw-Hill: New York, New York (1971b).

Attachment No. 2

Adjusted survival data are analyzed by the National Cancer Institute (NCI) lifetable package (Thomas, 1977). The tests include: Graphical (Kaplan-Meier product-limit estimation curves), Cox-Tarone binary regression methods for trend and heterogeneity, and Gehan-Breslow nonparametric methods for trend and heterogeneity.

Non-neoplastic lesions. Non-neoplastic lesions are analyzed by the Cochran-Armitage test for trend and the Fisher-Irwin exact test for heterogeneity (Thakur, 1985).

Neoplastic lesions. Incidental tumors are analyzed by Dinse-Lagakos logistic prevalence methods (Dinse, 1983) for trend and heterogeneity. Rapidly lethal and palpable tumors are analyzed in the same manner as survival.

In the cases where the study pathologist can assign particular occult neoplastic lesions as the cause of death in the animals, such information will be taken into appropriate analysis as in the IARC document (Peto et al, 1980).

References

Thomas, D. G., Breslow, N., and Gart, J. J., "Trend and Homogeneity Analyses of Proportions and Life Table Data," Comput. Biomed. Res., 10:373-381 (1977).

Thakur, A. K., Berry, K. J., and Mielke, Jr., P. W., "A FORTRAN Program for Testing Trend and Homogeneity in Proportions," Comput. Progr. Biomed., 19:229-233 (1985).

Dinse, G. E., and Lagakos, S. W., "Regression Analysis of Tumor Prevalence Data," J. Roy. Stat. Soc. Series C (Appl. Stat.), 32:236-248 (1983).

Peto, R., Pike, M. C., Day, N. E., Gray, R. G., Lee, P. N., Parish, S., Peto, J., Richards, S., and Wahrendorf, J., "Guidelines for Simple Sensitive Significance Tests for Carcinogenic Effects in Long-term Animal Experiments, in Long-term and Short-term Screening Assays for Carcinogens: A Critical Appraisal," Lyon: International Agency for Cancer Research, pp. 311-426 (1980).

COVANCE
THE DEVELOPMENT SERVICES COMPANY

PROTOCOL AMENDMENT NO. 1

Covance 6329-212

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Sponsor:	3M, St. Paul, Minnesota
Study Monitor:	Andrew M. Seacat, PhD
Testing Facility:	Covance Laboratories Inc., Madison, Wisconsin
Study Director:	Peter J. Thomford, PhD

This amendment modifies the following portions of the protocol:

Effective January 21, 1998

1. **Page 4, Animals, Strain.** To correct the strain of the animals used in this study, delete the text in this section and replace with the following:

   Crl:CD®(SD)IGS BR

Effective February 6, 1998

2. **Page 7, Group Designations and Dietary Levels, Footnote C.** To reflect the decision to delay the Week 3 collection, delete the text in this section and replace with the following:

   c Five animals/sex/group in Groups 1 through 5 will be sacrificed during Weeks 4 and 14 for hepatocellular proliferation rate measurements and biochemical analyses (palmitoyl-CoA oxidation).

3. **Page 12, Hepatocellular Proliferation and Biochemical Analyses, Frequency and Number of Animals.** To reflect the decision to delay the Week 3 collection, delete the text in this section and replace with the following:

   Five animals/sex/group in Groups 1 through 5 during Weeks 4 and 14

4. **Page 13, Hepatocellular Proliferation and Biochemical Analyses.** To reflect the decision to perform mitochondrial analyses, add the following section after "Palmitoyl-CoA Oxidase Tissue Collection and Analyses":

   Mitochondrial Analyses
   At the Week 4 collection, a sample (approximately 1.5 g) from the remaining portion of the liver will be collected from selected animals. Samples will be placed on wet ice and transferred to the Sponsor for mitochondrial analysis. Results will be reported separately by the Sponsor.

OK - to Keep

Effective February 17, 1998

5. **Page 12.** To reflect the decision to collect serum samples for possible future analysis, add the following section after "Clinical Pathology".

   Serum Analyses

   Frequency and Number of Animals
   Five animals/sex/group in Groups 1 through 5 during Weeks 4 and 14 (animals selected for hepatocellular proliferation and biochemical analyses); five animals/sex/group from Groups 1, 4, and 5 (from animals selected for interim sacrifice) after at least 78 weeks of treatment; and five animals/sex/group from Groups 1 through 5 at the terminal sacrifice

   Method of Collection
   Animals will be fasted overnight; blood (approximately 2 mL) will be collected from a jugular vein. Samples will be collected without anticoagulant.

Sample Handling
Blood samples will be allowed to clot at room temperature and centrifuged. Serum samples will be harvested and stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

6. **Page 13, Hepatocellular Proliferation and Biochemical Analyses.** To reflect the decision to collect liver samples for possible future analyses, add the following section after "Palmitoyl-CoA Oxidase Tissue Collection and Analyses":

Additional Liver Sample Collection
At the collections during Weeks 4 and 14, the remaining liver will be stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

7. **Page 14, Postmortem Procedures.** To reflect the decision to collect liver samples for possible future analyses, add the following section after "Bone Marrow Smear":

Additional Liver Sample Collection
A portion of the liver will be collected from five animals/sex/group from Groups 1, 4, and 5 at the interim sacrifice and from five animals/sex/group from Groups 1 through 5 at the terminal sacrifice and stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

8. **Page 17, Record Retention, Paragraph 4.** To indicate that frozen liver samples sent to the Sponsor will be retained by the Sponsor, delete this paragraph and replace with the following:

Frozen liver samples and serum samples sent to the Sponsor will be retained by the Sponsor.

Effective March 13, 1998

9. **Page 7, Group Designations and Dietary Levels.** To reflect the decision to sacrifice five animals/sex/group from Groups 1 and 5 and to terminate the remaining animals in Group 5 and 7 during Week 8, delete the text in this section and replace with the following:

Group	Number of Animals Male	Number of Animals Female	Dietary Levels (ppm NEtFOSE)[a]
1 (Control)[b,c,d,e]	70	70	0
2 (Low)[c]	60	60	3
3 (Mid)[c]	60	60	30
4 (Mid-High)[c,e]	70	70	100
5 (High)[c,d]	70	70	300
6 (Mid-High Recovery)[f]	40	40	100
7 (High Recovery)[d]	40	40	300

a T-6316 is 98.1% n-ethyl perfluorooctanesulfonamido ethanol (NEtFOSE); dose levels are expressed as ppm of NEtFOSE.

b The control animals will receive the basal diet only.

c Five animals/sex/group in Groups 1 through 5 will be sacrificed during Week 4 and five animals/sex/group in Groups 1 through 4 will be sacrificed during Week 14 for hepatocellular proliferation rate measurements and biochemical analyses (palmitoyl-CoA oxidation).

d Five animals/sex/group in Groups 1 and 5 will be sacrificed during Week 8; the remaining animals in Groups 5 and 7 will be sacrificed and discarded during Week 8.

e Ten animals/sex/group in Groups 1 and 4 will be designated as interim sacrifice animals and will be sacrificed after at least 78 weeks of treatment.

f Animals in Group 6 will be treated for at least 78 weeks, then treatment will be discontinued, and the animals will be observed for reversibility, persistence, or delayed occurrence of toxic effects for at least 26 weeks posttreatment. During recovery, the animals will receive basal diet only.

10. **Page 7, Dosing Procedures, Method of Administration.** To reflect the decision to terminate Groups 5 and 7 during Week 8, delete the text in this section and replace with the following:

    Dietary. Animals in Groups 1 through 4 will receive test diet for at least 104 weeks. Animals in Group 6 will receive test diet for 78 weeks only. Animals in Groups 5 and 7 will receive test diet for at least 7 weeks.

11. **Page 9, Observation of Animals, Body Weights.** To reflect the decision to record body weights before sacrifice during Week 8, add the following to this section:

    Body weights will also be recorded for all animals in Groups 5 and 7 before sacrifice during Week 8.

12. **Page 10, Clinical Pathology, Frequency and Number of Animals, Scheduled Collections.** To reflect the decision to collect clinical pathology samples from animals in Groups 1 and 5 during Week 8 and to terminate Groups 5 and 7 during Week 8, delete the text in this section and replace with the following:

    Hematology, clinical chemistry, urinalysis, and urine chemistry will be done on five animals/sex/group from Groups 1 and 5 during Week 8. Hematology, clinical chemistry, urinalysis, urine chemistry, and serum sampling will be done on 10 animals/sex/group in Groups 1 through 4 during Weeks 14, 27, and 53.

    A blood film will be made and held for possible future examination for animals at scheduled sacrifices after at least 78 and 104 weeks of treatment.

13. **Page 12, Serum Analyses, Frequency and Number of Animals.** To reflect the decision to collect serum samples from animals in Groups 1 and 5 during Week 8 and to terminate Groups 5 and 7 during Week 8, delete the text in this section and replace with the following:

Five animals/sex/group in Groups 1 through 5 during Week 4 (animals selected for hepatocellular proliferation and biochemical analyses); five animals/sex/group in Groups 1 and 5 during Week 8; five animals/sex/group in Groups 1 through 4 during Week 14 (animals selected for hepatocellular proliferation and biochemical analyses); five animals/sex/group from Groups 1 and 4 (from animals selected for interim sacrifice) after at least 78 weeks of treatment; and five animals/sex/group from Groups 1 through 4 at the terminal sacrifice

14. **Page 12, Hepatocellular Proliferation and Biochemical Analyses, Frequency and Number of Animals.** To reflect the decision to perform analyses on livers from animals in Groups 1 and 5 during Week 8 and to terminate Groups 5 and 7 during Week 8, delete the text in this section and replace with the following:

    Five animals/sex/group in Groups 1 through 5 during Week 4; five animals/sex/group in Groups 1 and 5 during Week 8; and five animals/sex/group in Groups 1 through 4 during Week 14

15. **Page 13, Hepatocellular Proliferation and Biochemical Analyses, Additional Liver Sample Collection.** To reflect the decision to collect livers from animals in Groups 1 and 5 during Week 8, delete the text in this section and replace with the following:

    At the collections during Weeks 4, 8, and 14, a portion of the remaining liver will be stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

16. **Page 12, Hepatocellular Proliferation and Biochemical Analyses, Cell Proliferation Tissue Collection and Immunohistochemical Evaluation, Paragraph 1.** To reflect the decision to collect only the left lateral lobe of the liver at the Week 8 and 14 collections, delete the text in this paragraph and replace with the following:

At each interval, animals will be fasted overnight, anesthetized with sodium pentobarbital, weighed, and exsanguinated. The abdominal cavity of each animal will be opened, and the liver will be removed and weighed. At the Week 4 collection, representative samples of left lateral, right median, and right lateral lobes of the liver and any macroscopic lesions of the liver will be collected and preserved in zinc formalin. At the Week 8 and 14 collections, representative samples of the left lateral lobe of the liver and any macroscopic lesions of the liver will be collected and preserved in zinc formalin.

17. **Page 13, Termination, Scheduled Sacrifices.** To reflect the decision to sacrifice five animals/sex/group from Groups 1 and 5, to terminate the remaining animals in Groups 5 and 7 during Week 8, and to sacrifice five animals/sex/group from Groups 1 through 4, delete the text in this section and replace with the following:

Interim Sacrifices

During Week 8, five animals/sex/group from Groups 1 and 5 will be fasted overnight, bled for clinical pathology and serum analyses, anesthetized with sodium pentobarbital, weighed, exsanguinated, and necropsied. Liver samples will be collected for palimitoyl CoA, PCNA, and frozen samples for possible analysis. The remaining animals in Groups 5 and 7 will be euthanatized with carbon dioxide and discarded without necropsy.

During Week 14, five animals/sex/group from Groups 1 through 4 will be fasted overnight, bled for clinical pathology and serum analyses, anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. Liver samples will be collected for palimitoyl CoA, PCNA, and frozen samples for possible analysis.

After at least 78 weeks of treatment, five animals/sex/group from Group 1 and 10 animals/sex from Group 4 will be fasted overnight, anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Terminal Sacrifice
After at least 104 weeks of treatment, the remaining animals in Groups 1 through 4 will be fasted overnight, then anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Recovery Sacrifice
After at least 78 weeks of treatment and 26 weeks without treatment, the remaining animals in Group 6 will be fasted overnight, then anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

18. **Page 14, Postmortem Procedures, Additional Liver Sample Collection.** To reflect the decision to sacrifice five animals/sex/group from Groups 1 and 5 and to terminate the remaining animals in Group 5 and 7 during Week 8, delete the text in this section and replace with the following:

A portion of the liver will be collected from five animals/sex/group from Groups 1 and 4 at the Week 79 sacrifice and from five animals/sex/group from Groups 1 through 4 at the terminal sacrifice and stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

19. **Page 15, Postmortem Procedures, Histopathology.** To reflect the decision to terminate Groups 5 and 7 early, to examine tissues from animals sacrificed during Weeks 8 and 14, and to examine tissues from Groups 4 and 6, delete the text in this section and replace with the following:

Tissues (as appropriate) from each animal in Groups 1, 4, and 6 sacrificed at the Week 79 interim sacrifice and terminal sacrifice and from each animal that dies or is sacrificed at an unscheduled interval will be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically. Adrenals, brain, eyes, kidneys, liver, mesenteric lymph node, pancreas, spleen, testes, and

ovaries from the animals necropsied at Weeks 8 and 14 interim sacrifice will also be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically.

Macroscopic lesions will also be examined microscopically from each animal in Groups 2 and 3 sacrificed at terminal necropsy.

20. **Page 16, Reports, Results.** To reflect the decision to include results from Covance 6329-228 as an appendix to this report, add the following:

The study report for Covance 6329-228 will be included as an appendix to this report.

Effective March 30, 1998

21. **Page 12, Hepatocellular Proliferation and Biochemical Analyses, Cell Proliferation Tissue Collection and Immunohistochemical Evaluation, Paragraph 3.** To include the microscopic examinations of liver sections stained with hematoxylin and eosin and to indicate that evaluations will be done on the left lateral lobe of the liver only, delete the text in this section and replace with the following:

Proliferation cell nuclear antigen (PCNA) evaluation will be done on the samples (left lateral lobe only). In addition, sections of the left lateral lobe of the liver will be stained with hematoxylin and eosin and examined microscopically. Results will be provided for inclusion in the final report.

AMENDMENT APPROVAL

Andrew M. Seacat, PhD
Study Monitor
3M

Date 4/21/98

Peter J. Thomford, PhD
Study Director
Covance Laboratories Inc.

Date 4/15/98

COVANCE
THE DEVELOPMENT SERVICES COMPANY

PROTOCOL AMENDMENT NO. 2

Covance 6329-212

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Sponsor:	3M, St. Paul, Minnesota
Study Monitor:	Andrew M. Seacat, PhD
Testing Facility:	Covance Laboratories Inc., Madison, Wisconsin
Study Director:	Peter J. Thomford, PhD

This amendment modifies the following portions of the protocol:

Effective April 23, 1998

1. **Page 13, Termination, Unscheduled Sacrifices and Deaths.** To reflect the decision to anesthetize animals with carbon dioxide for unscheduled sacrifices, delete the text in this section and replace with the following:

   Necropsies will be done. Animals to be sacrificed will be anesthetized, weighed, and exsanguinated. Animals sacrificed before Week 13 will be anesthetized with sodium pentobarbital, animals sacrificed during or after Week 13 will be anesthetized with carbon dioxide. A blood film will be taken as part of the necropsy procedure for sacrificed animals.

Effective April 28, 1998

2. **Page 2, Alternate Study Monitor.** To include alternate study monitor in the protocol, add the following after "Study Monitor:"

Alternate Study Monitor
Marvin T. Case, DVM, PhD
3M Toxicology Services
Telephone No.: 612.733.5180
Facsimile No.: 612.733.1773

3. **Page 7, Group Designations and Dietary Levels.** To reflect the decision to begin recovery after 52 weeks of treatment and to change the Week 79 interim sacrifice to Week 53, delete the text in this section and replace with the following:

Group	Number of Animals Male	Number of Animals Female	Dietary Levels (ppm NEtFOSE)[a]
1 (Control)[b,c,d,e]	70	70	0
2 (Low)[c]	60	60	3
3 (Mid)[c]	60	60	30
4 (Mid-High)[c,e]	70	70	100
5 (High)[c,d]	70	70	~~300~~
6 (Mid-High Recovery)[f]	40	40	100
7 (High Recovery)[d]	40	40	300

a T-6316 is 98.1% n-ethyl perfluorooctanesulfonamido ethanol (NEtFOSE); dose levels are expressed as ppm of NEtFOSE.

b The control animals will receive the basal diet only.

c Five animals/sex/group in Groups 1 through 5 will be sacrificed during Week 4, and five animals/sex/group in Groups 1 through 4 will be sacrificed during Week 14 for hepatocellular proliferation rate measurements and biochemical analyses (palmitoyl-CoA oxidation).

d Five animals/sex/group in Groups 1 and 5 will be sacrificed during Week 8; the remaining animals in Groups 5 and 7 will be sacrificed and discarded during Week 8.

e Ten animals/sex/group in Groups 1 and 4 will be designated as interim sacrifice animals and will be sacrificed after at least 52 weeks of treatment.

f Animals in Group 6 will be treated for at least 52 weeks, then treatment will be discontinued, and the animals will be observed for reversibility, persistence, or delayed occurrence of toxic effects for at least 52 weeks posttreatment. During recovery, the animals will receive basal diet only.

4. **Page 7, Dosing Procedures, Method of Administration.** To reflect the decision to begin recovery after 52 weeks of treatment, delete the text in this section and replace with the following:

   Dietary. Animals in Groups 1 through 4 will receive test diet for at least 104 weeks. Animals in Group 6 will receive test diet for 52 weeks only. Animals in Groups 5 and 7 will receive test diet for at least 7 weeks.

5. **Page 10, Clinical Pathology, Frequency and Number of Animals, Scheduled Collections, Paragraph 2.** The Week 79 interim sacrifice will be moved to Week 53, therefore blood films will be prepared from animals sacrificed after 52 weeks of treatment. To reflect this decision, delete the text in this paragraph and replace with the following:

   A blood film will be made and held for possible future examination for animals at scheduled sacrifices after at least 52 and 104 weeks of treatment.

6. **Page 12, Serum Analyses, Frequency and Number of Animals.** The Week 79 interim sacrifice will be moved to Week 53, therefore serum samples will be collected from animals sacrificed after 52 weeks of treatment. To reflect this decision, delete the text in this section and replace with the following:

   Five animals/sex/group in Groups 1 through 5 during Week 4 (animals selected for hepatocellular proliferation and biochemical analyses); five animals/sex/group in Groups 1 and 5 during Week 8; five animals/sex/group in Groups 1 through 4 during Week 14 (animals selected for hepatocellular proliferation and biochemical analyses); five animals/sex/group from Groups 1 and 4 (from animals selected for interim sacrifice) after at least 52 weeks of treatment; and five animals/sex/group from Groups 1 through 4 at the terminal sacrifice.

7. **Page 13, Termination, Scheduled Sacrifices.** To reflect the decision to move the Week 79 interim sacrifice to Week 53 and to begin recovery after 52 weeks of treatment, delete the text in this section and replace with the following:

Interim Sacrifices
During Week 8, five animals/sex/group from Groups 1 and 5 will be fasted overnight, bled for clinical pathology and serum analyses, anesthetized with sodium pentobarbital, weighed, exsanguinated, and necropsied. Liver samples will be collected for palimitoyl CoA analysis and PCNA evaluation; additional liver samples will be frozen for possible analysis. The remaining animals in Groups 5 and 7 will be euthanatized with carbon dioxide and discarded without necropsy.

During Week 14, five animals/sex/group from Groups 1 through 4 will be fasted overnight, bled for clinical pathology and serum analyses, anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. Liver samples will be collected for palimitoyl CoA analysis and PCNA evaluation; additional liver samples will be frozen for possible analysis for possible analysis.

After at least 52 weeks of treatment, five animals/sex/group from Group 1 and 10 animals/sex from Group 4 will be fasted overnight, anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied.

Terminal Sacrifice
After at least 104 weeks of treatment, the remaining animals in Groups 1 through 4 will be fasted overnight, then anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Recovery Sacrifice
After at least 52 weeks of treatment and 52 weeks without treatment, the remaining animals in Group 6 will be fasted overnight, then anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

8. **Page 14, Postmortem Procedures, Organ Weights.** To reflect the decision to record organ weights at the Week 8, 14, and 53 interim sacrifices only, delete the text in this section and replace with the following:

At the Week 8, 14, and 53 interim sacrifices, the following organs (when present) will be weighed; paired organs will be weighed separately:

adrenal (2)	ovary (2)
brain	spleen
kidney (2)	testis (2)
liver	thyroid (2) with parathyroid
lung	uterus with cervix

Organ-to-body weight percentages and organ-to-brain weight ratios will be calculated.

9. **Page 14, Postmortem Procedures, Additional Liver Sample Collection.** The Week 79 interim sacrifice will be moved to Week 53, therefore liver samples will be collected from animals sacrificed after 52 weeks of treatment. To reflect this decision, delete the text in this section and replace with the following:

A portion of the liver will be collected from five animals/sex/group from Groups 1 and 4 at the Week 53 sacrifice and from five animals/sex/group from Groups 1 through 4 at the terminal sacrifice and stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

10. **Page 15, Postmortem Procedures, Histopathology, Paragraph 1.** The Week 79 interim sacrifice will be moved to Week 53, therefore histopathology will be done for animals sacrificed after 52 weeks of treatment. To reflect this decision, delete the text in this section and replace with the following:

Tissues (as appropriate) from each animal in Groups 1, 4, and 6 sacrificed at the Week 53 interim sacrifice and terminal sacrifice and from each animal that dies or is sacrificed at an unscheduled interval will be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically. Adrenals, brain, eyes, kidneys, liver, mesenteric lymph node, pancreas, spleen, testes, and ovaries from the animals necropsied at the Week 8 and 14 interim sacrifices will also be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically.

11. **Page 15, Reports, Paragraph 1.** To reflect the decision to provide unaudited summary reports after the Week 14 and 53 interim sacrifices, delete the text in this paragraph and replace with the following:

After the Week 14 and 53 interim sacrifices, unaudited summary reports will be sent to the Sponsor. The summary reports will include a brief description of methods and results and summary tables of in-life data, clinical pathology data, and anatomical pathology data. After completion of the study, one copy of the draft report will be sent to the Sponsor. The draft report will include the following information:

Effective May 15, 1998

12. Page 12, Serum Analyses, Method of Collection. To reflect the decision to increase the volume collected for serum analyses at the Week 53 and 105 sacrifices, delete the text in this section and replace with following:

Animals will be fasted overnight; blood (approximately 2 mL at the Week 4, 8 and 14 sacrifices, approximately 3 mL at the Week 53 and 105 sacrifices) will be collected from a jugular vein. Samples will be collected without anticoagulant.

AMENDMENT APPROVAL

Andrew M. Seacat, PhD
Study Monitor
3M

Date 6/8/98

Peter J. Thomford, PhD
Study Director
Covance Laboratories Inc.

Date 6/2/98

COVANCE
THE DEVELOPMENT SERVICES COMPANY

PROTOCOL AMENDMENT NO. 3

Covance 6329-212

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Sponsor:	3M, St. Paul, Minnesota
Study Monitor:	Andrew M. Seacat, PhD
Testing Facility:	Covance Laboratories Inc., Madison, Wisconsin
Study Director:	Peter J. Thomford, PhD

This amendment modifies the following portions of the protocol:

Effective January 21, 1998

1. **Page 4.** To include the vehicle used to dissolve the test material before mixing with the diet, add the following section.

Vehicle

Identification
Acetone

Lot Numbers
The lot numbers will be maintained in the raw data.

Purity
On file with the manufacturer

Stability
On file with the manufacturer

Storage Conditions
At room temperature

Characteristics
Information on synthesis methods, composition, or other characteristics that define the vehicle is on file with the manufacturer.

2. **Page 16, Reports, Results.** To include organ weights as a result to be reported, add "organ weights" to this section.

3. **Page 16, Reports, Statistical Evaluation.** To include statistical evaluation of organs weights, add "organ weights (Weeks 8, 14, and 53 interim sacrifices only)"

Effective April 28, 1998

4. **Page 7, Group Designations and Dietary Levels, Footnote e.** To correct the number of Group 1 animals to be sacrificed at the Week 53 interim sacrifice, delete this sentence and replace with the following:

   e Five animals/sex in Group 1 and 10 animals/sex in Group 4 will be designated as interim sacrifice animals and will be sacrificed after at least 52 weeks of treatment.

Effective June 30, 1998

5. **Page 8, Dosing Procedure, Retention Samples.** To reflect the decision to send retention samples of control diet to the Sponsor, delete the text in this section and replace with the following:

   Samples (approximately 100 g) will be taken from each dose preparation during the in-life phase and stored at room temperature. Unless used for analyses, retention samples from the treated groups will be discarded at least 1 month after completion of the in-life phase. Samples from control diets prepared for Weeks 24 and before will be shipped to:

Kris J. Hansen, PhD
3M Environmental Technology and Safety Services
935 Bush Avenue
Building 2-3E-09
St. Paul, Minnesota 55133-3331
Telephone No.: 651.778.6018
Facsimile No.: 651.778.6176

Effective July 20, 1998

6. **Page 2, Study Monitor and Alternate Study Monitor.** To indicate the change in the area code, delete the text in these sections and replace with the following:

Study Monitor
Andrew M. Seacat, PhD
3M
Telephone No.: 651.575.3161
Facsimile No.: 651.733.1773

Alternate Study Monitor
Marvin T. Case, DVM, PhD
3M Toxicology Services
Telephone No.: 651.733.5180
Facsimile No.: 651.733.1773

7. **Page 4, Disposition of Test Material.** To indicate the change in the area code, delete the text in these section and replace with the following:

After authorization from the Sponsor, any remaining test material will be returned to:

Andrew M. Seacat, PhD
3M
Toxicology Services
Building 220-2E-02, 3M Center
St. Paul, Minnesota 55144-1000
Telephone No.: 651.575.3161
Facsimile No.: 651.733.1733

8. **Page 12, Clinical Pathology, Serum Samples.** To indicate the change in the area code, delete the text in these section and replace with the following:

Serum not used for clinical chemistry will be stored in a freezer set to maintain -60 to -80 C. Samples will be packed on dry ice and shipped to:

Kris J. Hansen, PhD
3M Environmental Technology and Safety Services
935 Bush Avenue
Building 2-3E-09
St. Paul, Minnesota 55133-3331
Telephone No.: 651.778.6018
Facsimile No.: 651.778.6176

Samples will be retained by the Sponsor for possible future analysis.

AMENDMENT APPROVAL

Andrew M. Seacat

Andrew M. Seacat, PhD
Study Monitor
3M

6/14/99
Date

Peter J. Thomford

Peter J. Thomford, PhD
Study Director
Covance Laboratories Inc.

02 June 1999
Date

COVANCE
THE DEVELOPMENT SERVICES COMPANY

PROTOCOL AMENDMENT NO. 4

Covance 6329-212

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Sponsor:	3M, St. Paul, Minnesota
Study Monitor:	Andrew M. Seacat, PhD
Testing Facility:	Covance Laboratories Inc., Madison, Wisconsin
Study Director:	Peter J. Thomford, PhD

This amendment modifies the following portion of the protocol.

Effective January 13, 2000

1. **Page 14, Postmortem Procedures, Tissue Preservation.** The Sponsor has requested that a subset of tissues be collected for possible electron microscopy. Therefore, add the following to this section

   At the terminal sacrifice, sections of the heart and liver will be collected from 10 animals/sex/group in Groups 1, 3, 4, and 6 and preserved in 2.0% paraformaldehyde/2.5% glutaraldehyde in 0.1 M phosphate buffer. These tissues will be processed and embedded in epoxy blocks for possible future examination by electron microscopy.

AMENDMENT APPROVAL

Andrew M. Seacat

Andrew M. Seacat, PhD
Study Monitor
3M

1/21/2000
Date

Peter J. Thomford

Peter J. Thomford, PhD
Study Director
Covance Laboratories Inc.

20 Jan 2000
Date

COVANCE
THE DEVELOPMENT SERVICES COMPANY

PROTOCOL AMENDMENT NO. 5

Covance 6329-212

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Sponsor:	3M, St. Paul, Minnesota
Study Monitor:	Andrew M. Seacat, PhD
Testing Facility:	Covance Laboratories Inc., Madison, Wisconsin
Study Director:	Peter J. Thomford, PhD

This amendment modifies the following portion of the protocol.

Effective January 21, 2000

1. **Page 10, Clinical Pathology, Frequency and Number of Animals, Scheduled Collections.** The Sponsor has requested that specific clinical chemistry parameters be determined for animals at terminal and recovery sacrifice. To reflect this decision, add the following to this section.

   In addition, samples for clinical chemistry parameters (cholesterol and triglycerides) will be collected from all remaining animals at terminal and recovery sacrifice.

2. **Page 12, Serum Analyses, Frequency and Number of Animals.** The Sponsor has requested that serum be collected from all animals at terminal and recovery sacrifice. To reflect this decision, delete the text in this section and replace with the following.

   Five animals/sex/group in Groups 1 through 5 during Week 4 (animals selected for hepatocellular proliferation and biochemical analyses); five animals/sex/group in Groups 1 and 5 during Week 8; five animals/sex/group in Groups 1 through 4 during Week 14 (animals selected for hepatocellular proliferation and biochemical analyses); five animals/sex/group from Groups 1 and 4 (from animals selected for interim sacrifice) after at least 52 weeks of treatment; and all remaining animals at terminal and recovery sacrifice.

3. **Page 14, Postmortem Procedures, Additional Liver Sample Collection.** The Sponsor has requested that frozen liver samples be collected from all animals at terminal and recovery sacrifice. Therefore, delete the text in this section and replace with the following.

A portion of the liver will be collected from five animals/sex/group from Groups 1 and 4 at the Week 53 sacrifice and from all remaining animals at the terminal and recovery sacrifice and stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

Effective January 26, 2000

4 **Page 15, Postmortem Procedures, Histopathology.** The Sponsor has requested that all tissues from animals in Groups 1 and 4 at terminal sacrifice and selected tissues from animals in Groups 2, 3, and 6 at the terminal and recovery sacrifice be examined microscopically. To reflect this decision, delete the text in this section and replace with the following.

Tissues (as appropriate) from each animal in Groups 1 and 4 sacrificed at the terminal sacrifice and from each animal that dies or is sacrificed at an unscheduled interval will be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically.

Tissues (as appropriate) from each animal in Groups 1, 4, and 6 sacrificed at the Week 53 interim sacrifice will be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically.

Lesions, liver, lungs, kidneys, pancreas, thyroid, testes, and mammary glands (females) from each animal in Groups 2, 3, and 6 sacrificed at the terminal and recovery sacrifice will be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically. Parathyroids will be processed with the thyroids, but will not be examined.

Adrenals, brain, eyes, kidneys, liver, mesenteric lymph node, pancreas, spleen, testes, and ovaries from the animals necropsied at the Week 8 and 14 interim sacrifices will also be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically.

AMENDMENT APPROVAL

Andrew M. Seacat, PhD
Study Monitor
3M

2/16/2000
Date

Peter J. Thomford, PhD
Study Director
Covance Laboratories Inc.

31 Jan 2000
Date

3M Environmental Laboratory

Protocol - Analytical Study Phase

104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

In-Vivo Study Reference Number: Covance # 6329-212

Study Number: AMDT-092597.1
Test Material: T-6316

Name and Address of Sponsor: 3M Toxicology Services
Building 220-2E-02, 3M Center
St. Paul, MN 55144-1000

Name and Address of Testing Facility: 3M Environmental Technology and Services
935 Bush Avenue
St. Paul, MN 55106

Proposed Initiation Date: April 7, 1998
Proposed Completion Date: October, 31, 1998

Method Numbers and Revisions
FACT-M-1.0, Extraction of Perfluorooctanesulfonate from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry
FACT-M-2.0, Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry
FACT-M-3.0, Extraction of Perfluorooctanesulfonate from Sera for Analysis using HPLC-Electrospray/Mass Spectrometry
FACT-M-4.0, Analysis of Sera Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry

Author: Lisa Clemen

4/9/98
Kris J. Hansen, PhD Date
Study Director

4/9/98
Dale Bacon Date
Study Director Management

7/30/98
Andrew M. Seacat, PhD Date
Sponsor Representative

1.0 PURPOSE

1.1 According to this analytical protocol, the 3M Environmental Laboratory will analyze the tissue and fluid samples from the Covance study number 6329-212, "104-Week Dietary Carcinogenicity with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats." The collected data will be given to the sponsor for their use in the assessment of toxicological effects of the test material when administered in the diets of rats for at least 104 weeks.

1.2 Data collected in the Environmental Laboratory will be considered non-quantitative screening data until future studies have been conducted to determine absolute recoveries of specific or general fluorochemical compounds.

2.0 REGULATORY COMPLIANCE

2.1 This analytical phase of the study will be conducted in accordance with the FDA Good Laboratory Practices Regulations 21 CFR 58, with the following exceptions:

2.1.1 The analytical phase is being conducted as a separate study and therefore has a separate Study Director, protocol, and final report, from those listed in the Covance protocol 6329-212.

2.1.2 The characterization of the reference material, including purity, identity, and stability, are the responsibility of the sponsor.

2.1.3 Sample storage stability will not be determined.

3.0 TEST MATERIALS

3.1 Control, and reference Materials and Matrices

3.1.1 Analytical Reference Material: T-6316, from 3M ICP/PCP Division

3.1.2 Analytical Reference Matrix: Rat liver, from Covance and rat serum, from Sigma Chemical Company.

3.1.3 Analytical Control Material: None.

3.1.4 Analytical Control Matrix: Rat liver, from Covance and rat serum, from Sigma Chemical Company.

3.2 Number of Test and Control Samples: Liver and serum from 680 test animals and 140 control animals will be made available; samples will be analyzed as requested by the sponsor or the study director. Other biological tissues (kidney, bile, dermal application site, and cellular fraction) will be available for analysis if deemed appropriate.

3.3 Identification of Test and Control Samples: The samples will be identified using the Covance animal identification number which consists of a letter and five digit number, plus the tissue identity and day identity (serum).

3.4 Purity and Identity of Reference Material: To be determined by Sponsor.

3.5 Stability of Reference Material: To be determined by Sponsor.

3.6 Storage Conditions for Reference Materials: Reference materials will be stored at room temperature (3.1.1), and samples will be stored at -20 ± 10°C (3.1.2, 3.1.4). Test and Control samples will be received according to AMDT-S-10-0.

3.7 Disposition of Specimens: Biological tissues and fluids will be retained per GLP Regulation for the time period required for studies longer than 28 days.

3.8 Safety Precautions: Refer to appropriate MSDS. Wear appropriate laboratory attire. Use caution when handling knives for cutting tissue samples.

4.0 EXPERIMENTAL - Overview

4.1 The tissues from animals dosed as described (Covance# 6329-212), will be available for analysis for fluorine-containing compounds. At the discretion of the Study Director, a series of analytical tests can be performed. All high dose and control sera and livers will be analyzed initially using HPLC-electrospray mass spectrometry to identify fluorine-containing compounds of interest present in the sera and liver (if any). The screening for fluoride in liver via combustion may be performed to present definitive data for fluorine in the liver. Based on the findings from these analyses, additional samples, tissues, or fluids may be analyzed at the discretion of the Study Director to determine the presence of fluorochemicals in these matrices.

5.0 EXPERIMENTAL - Methods

5.1 Methods (attached):

- **5.1.1 FACT-M-1.0,** "Extraction of Perfluorooctanesulfonate from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry"
- **5.1.2 FACT-M-2.0,** "Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry"
- **5.1.3 FACT-M-3.0,** "Extraction of Perfluorooctanesulfonate from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry"
- **5.1.4 FACT-M-4.0,** "Analysis of Serum Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry"

6.0 DATA ANALYSIS

6.1 Quality Control: Matrix spikes will be extracted and analyzed to determine accuracy of the method. Also, continuing calibration checks will be analyzed to determine response bias.

6.2 Transformations: Any transformations performed on data collected during the analytical phase of the study will be documented in the final report.

6.3 Statistics: At the discretion of the Study Director, statistics used may include regression analysis of serum concentrations with time, averages, and standard deviations of concentrations for the different dose groups. If necessary, simple tests such as the Student's t-test may be applied to determine statistical difference. Any statistical analysis performed will be documented in the final report.

6.4 Data Reporting: A final data package will be submitted to 3M Toxicology Services. The data package will include the following with additional data included as deemed appropriate.

6.4.1 A summary of individual sample results, reported as a concentration (weight/weight, weight/volume) of fluoride per tissue or fluid, or as the mass of a specific fluorochemical (HPLC-electrospray mass spectrometry) per unit of tissue or fluid.

6.4.2 A summary of quality control results (continuing calibration checks, method blanks, instrument blanks, matrix spikes, and matrix spike duplicates).

6.4.3 Certified copies or originals of the written validated methods.

6.4.4 Certified copies or originals of sample identification sheets sent from Covance.

6.4.5 Certified copies or originals of study specific raw data.

6.4.6 A summary of key personnel involved with the analytical phase of the study.

6.4.7 A signed QAU statement listing the dates of inspections and reports of findings to management and Study Director.

7.0 MAINTENANCE OF RAW DATA AND RECORDS

7.1 The following raw data and records (or certified copies thereof) will be maintained in the study folder in the archives according to appropriate SOPs.

7.1.1 Approved protocol
7.1.2 Approved methods
7.1.3 Data summaries
7.1.4 Study correspondence
7.1.5 Shipping records
7.1.6 Raw data
7.1.7 Electronic copies of data

7.2 Supporting records to be retained separately from the study folder in the archives according to 3M ET & SS SOPs, will include, but not necessarily be limited to the following:

7.2.1 Approved validation reports
7.2.2 Training records
7.2.3 Calibration records
7.2.4 Instrument maintenance logs
7.2.5 Standard operating procedures, equipment procedures, and methods
7.2.6 Appropriate specimens

8.0 REFERENCES

8.1 AMDT-S-1, "Chemical Tracking"
8.2 AMDT-S-2, "Solutions and Standards Making"
8.3 AMDT-S-3, "Training"
8.4 AMDT-S-4, "General Lab Documentation Systems"
8.5 AMDT-S-5, "GLP-related Documentation Systems"
8.6 AMDT-S-6, "General Lab Records"
8.7 AMDT-S-7, "GLP-Related Records"
8.8 AMDT-S-8, "Archives"
8.9 AMDT-S-9, "GLP Program and Responsibilities"
8.10 AMDT-S-10, "Sample Tracking System"

8.11 AMDT-S-12, "Analytical Method Validation"

8.12 AMDT-S-13, "Equipment Installation"

8.13 AMDT-S-14, "Quality Assurance Unit"

8.14 AMDT-S-15, "Laboratory Practices and Data Management"

8.15 AMDT-S-17, "Analytical Equipment Systems Documentation"

8.16 AMDT-S-18, "Routine Calibration Checks of Balances"

8.17 AMDT-S-20, "Daily Calibration Check of Automatic Pipettors"

8.18 AMDT-S-21, "Annual Calibration Checks of Automatic Pipettors"

8.19 AMDT-S-25, "Labeling, Storage, and Use of Test, Control, and Reference Substances"

8.20 AMDT-S-30, "Project Control and Data Review in the AMDT"

9.0 ATTACHMENTS

9.1 FACT-M-1.0, Extraction of Perfluorooctanesulfonate from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry

9.2 FACT-M-2.0, Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry

9.3 FACT-M-3.0, Extraction of Perfluorooctanesulfonate from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry

9.4 FACT-M-4.0, Analysis of Serum Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry

Study Title

104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

PROTOCOL AMENDMENT NO. 1

Amendment Date:

20 January 2000

Performing Laboratory

3M Environmental Technology & Safety Services
3M Environmental Laboratory
935 Bush Avenue
St. Paul, MN 55106

Laboratory Project Identification

ET&SS LRN-U2103
FACT TOX-001
Covance Study: 6329-212
3M Medical Department Study: T-6316.1

This amendment modifies the following portion(s) of the protocol:

1. ***PROTOCOL READS:***
   The following methods will be used:
   FACT-M-1.0, Extraction of Perfluorooctanesulfonate from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry
   FACT-M-2.0, Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry
   FACT-M-3.0, Extraction of Perfluorooctanesulfonate from Sera for Analysis using HPLC-Electrospray/Mass Spectrometry
   FACT-M-4.0, Analysis of Sera Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry

   AMEND TO READ:
   The following methods will be used:
   ETS-8-4.1, Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry
   ETS-8-5.1, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry
   ETS-8-6.0, Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry
   ETS-8-7.0, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry

   REASON:
   The methods originally listed were superseded during the course of the study.

2. ***PROTOCOL READS:***
   There is no independent section of the protocol that addresses sample retention.
   AMEND TO READ:
   Specimens will be maintained in the 3M Environmental Laboratory specimen archives. Any specimens sent to sub-contract laboratories will be returned to the 3M Environmental Laboratory upon completion of analysis and submission of the sub-contract laboratory(s) final report. Specimens analyzed at sub-contract laboratories will be returned with the following documentation: the signed original chain of custody and records of storage conditions while at the sub-contract facility.
   REASON:
   To define in detail the appropriate disposition of specimens analyzed at subcontract laboratories.

3. ***PROTOCOL READS:***
Section 7 states that the following raw data and records will be retained in the study folder in the archives according to AMDT-S-8: Approved protocol and amendments; approved methods; data summaries; study correspondence; shipping records; raw data; and electronic copies of data. Additionally, Section 7 states that supporting records to be retained separately from the study folder in the archives according to AMDT-S-8 will include at least the following: Approved validation reports; training records; calibration records; instrument maintenance logs; Standard Operating Procedures, Equipment Procedures, and Methods; and appropriate specimens.
AMEND TO READ:
Section 7 states: "The original data, or copies thereof, will be available at the 3M Environmental Laboratory to facilitate audits of the study during its progress and before acceptance of the final report. When the final report is completed, all original paper data, including: approved protocol and amendments, study correspondence, shipping records, raw data, approved final report, and electronic copies of data will be retained in the archives of the 3M Environmental Laboratory. All corresponding training records, calibration records, instrument maintenance logs, standard operating procedures, equipment procedures, and methods will be retained in the archives of the facility performing each analysis."
REASON:
To direct subcontract laboratories in the disposition of the items listed above.

4. ***PROTOCOL READS:***
The study director for the present study was identified in the protocol as Kristen J. Hansen, Ph.D.
AMEND TO READ:
The role of study director for the present study was reassigned to John L. Butenhoff, Ph.D., as of 20 January 2000. The previous study director, Kristen Hansen, has been reassigned to the role of Principle Analytical Investigator.
REASON:
The role of study director was reassigned in an effort to ensure compliance with Good Laboratory Practice Standards that outline study personnel requirements (refer to 21 CFR Part 58).

5. ***PROTOCOL READS:***
The sponsor for the present study was identified as Andrew M. Seacat, Ph.D.
AMEND TO READ:
The role of sponsor for the present study was reassigned to Marvin T. Case, D.V.M., Ph.D., as of 20 January 2000.
REASON:
The change was made at the request of the sponsor.

Amendment Approval

Andrew M. Seacat 2/10/2000
Andrew M. Seacat, Ph.D., Outgoing Sponsor Representative Date

Kristen Hansen 11-Feb-2000
Kristen J. Hansen, Ph.D., Outgoing Study Director Date

Marvin T Case 10 February 2000
Marvin T. Case, D.V.M., Ph.D., Incoming Sponsor Representative Date

John L. Butenhoff February 10, 2000
John L. Butenhoff, Ph.D., Incoming Study Director Date

Study Title

104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

PROTOCOL AMENDMENT NO. 2

Amendment Date:

January 8, 2001

Performing Laboratory

3M Environmental Technology & Safety Services
3M Environmental Laboratory
935 Bush Avenue
St. Paul, MN 55106

Laboratory Project Identification

ET&SS LRN-U2103
FACT TOX-001
Covance 6329-212
3M Medical Department Study: T-6316.1

This amendment modifies the following portion(s) of the protocol:

1. PROTOCOL READS:

1.2 Data collected in the Environmental Laboratory will be considered non-quantitative screening data until future studies have been conducted to determine absolute recoveries of specific or general fluorochemical compounds.

AMEND TO READ:

1.2 If matrix spike studies provide accurate representation of recovery of endogenous levels of PFOS, PFOSA, PFOSAA, PFOSEA, M556, and EtFOSE-OH, the 3M Environmental Laboratory will provide semi-quantitative data for sera and liver samples collected from test animals.

REASON:

Due to improved analytical methods, the decision was made to change the purpose of the study results from non-quantitative to semi-quantitative.

3M Environmental Laboratory

Amendment Approval

Marvin T Case — 10 Jan 2001

Marvin T. Case, D.V.M., Ph.D., Sponsor Representative — Date

John L. Butenhoff — 10 JAN 2001

John L. Butenhoff, Ph.D., Study Director — Date

Study Title

104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

PROTOCOL AMENDMENT NO. 3

Amendment Date:

February 15, 2001

Performing Laboratory

3M Environmental Technology & Safety Services
3M Environmental Laboratory
935 Bush Avenue
St. Paul, MN 55106

Laboratory Project Identification

ET&SS LRN-U2103
FACT TOX-001
Covance 6329-212
3M Medical Department Study: T-6316.1

3M Environmental Laboratory

This amendment modifies the following portion(s) of the protocol:

1. PROTOCOL READS:

Specimens will be maintained in the Environmental Laboratory specimen archives. Any specimen sent to sub-contract laboratories will be returned to the 3M Environmental Laboratory upon completion of analysis and submission of the sub-contracted laboratory(s) final report. Specimens analyzed at sub-contracted laboratories will be returned with the following documentation: the signed original chain of custody and records of storage conditions while at the sub-contracted facility.

AMEND TO READ:

Specimens will be maintained in the Environmental Laboratory specimen archives. Specimens sent to sub-contract laboratories for analysis of PFOS will be returned to the 3M Environmental Laboratory upon completion of analysis and submission of the sub-contracted laboratory(s) final report. Specimens sent to sub-contract laboratories for analysis of compounds other than PFOS will not be returned. Specimens returned by sub-contracted laboratories will be returned with the following documentation: the signed original chain of custody and records of storage conditions while at the sub-contracted facility.

REASON:

Some specimens sent to sub-contract laboratories will not be returned. Portions of these specimens have been retained at the 3M Environmental Laboratory.

Amendment Approval

Marvin T Case 21 Feb 2001

Marvin T. Case, D.V.M., Ph.D., Sponsor Representative Date

John L. Butenhoff 21 FEB 2001

John L. Butenhoff, Ph.D., Study Director Date

3M Environmental Laboratory

Study Title

104–Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

PROTOCOL AMENDMENT NO. 4

Amendment Date:

April 2, 2001

Performing Laboratory

3M Environmental Technology & Safety Services
3M Environmental Laboratory
935 Bush Avenue
St. Paul, MN 55106

Laboratory Project Identification

ET&SS FACT TOX-001
3M Laboratory Request No. U2103

This amendment modifies the following portion(s) of the protocol:

1. ***THE AMENDED PROTOCOL READS:***
(Amendment #1) The following methods will be used:
ETS-8-4.1, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry"
ETS-8-5.1, "Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry"
ETS-8-6.0, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry"
ETS-8-7.0, "Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry"

AMEND TO READ:
The following methods will be used:
ETS-8-4.1, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry"
ETS-8-5.1, "Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry" *with the following exception*: (Section 14.5.1) "Matrix spike recoveries must be within ± 30% of the spiked concentration for all analytes except M556, which must be within ±50%.
ETS-8-6.0, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry"
ETS-8-7.0, "Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry" *with the following exception*: (Section 14.5.1) "Matrix spike recoveries must be within ±30% of the spiked concentration for all analytes except PFOS, which must be ±50%.

REASON: The analytical method and resulting QC data support a 50%–150% acceptable range for spike recoveries for these analytes and matrices.

Amendment Approval

John L Butenhoff 4/4/2001

John L. Butenhoff, Ph.D., Study Director — Date

Marvin T Case 9 April 2001

Marvin T. Case, D.V.M., Ph.D., Sponsor Representative — Date

3M Environmental Laboratory

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) ☐ SOP X Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number(s): FACT-M-2.0, FACT-M-4.0, ETS-8-5.0, ETS-8-7.0

Date(s) of occurrence: Entire Study

II. Description:

Required Procedure/process:
These methods state: Typically the analytical batch run sequence begins and ends with a set of extracted matrix standards.

Actual Procedure/process:
In many analytical runs, only the initial extracts matrix curve was used to evaluate the extracts. Either the second curve was not injected or the bracketing curve was deactivated since this calibraton curve was divergent.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written and a few method modifications were also written stating that the second bracketing calibration curve may be deactivated if instrumental drift affects the data. The first curve and acceptable calibration checks shall then bracket usable data.

Recorded By: Lisa A. Clemen Date: 05/17/01

IV. Impact on Study / Project

No adverse impact on the outcome of this study since data were sufficiently bracketed by calibration checks. 05/27/01

Authorized By *(Study Director / Project Lead)*: John L. Butenhoff 5/24/01 Date:

SD: John Butenhoff Sponsor Rep: Marv Case
Marvin T Case 31 May 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 1
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) ❒ SOP X Method ❒ Equipment Procedure ❒ Protocol ❒ Other:

Document Number(s): FACT-M-3.0

Date(s) of occurrence: 04/23/98, 04/28/98, 05/11/98

II. Description:

Required Procedure/process:
Section 11.1.2 states: If the majority of the serum samples are less than 1.0 mL, extract the standards in the same volume as the samples.

Actual Procedure/process:
Initial sera volumes for weeks 4, 8, and 14 were below 1.0 mL (0.6 mL, 0.5 mL, & 0.5 mL respectively) The curves prepared for these samples were made using 1.0 mL of serum.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written.

Recorded By: Kim A Clemen Date: 02/13/01

IV. Impact on Study / Project

No adverse impact on the study. KJH 02/13/01

Authorized By *(Study Director / Project Lead)* Date

Sponsor Representative: Marvin Case Marvin T Case 15 Feb 2001
Study Director: John Butenhoff John R. Butenhoff 16 FEB 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 2
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one): ❒ SOP X Method ❒ Equipment Procedure ❒ Protocol ❒ Other:

Document Number(s): ETS-8-5.1

Date(s) of occurrence: 05/05/98

II. Description:

Required Procedure/process:
Acceptable data must be bracketed by the curve and passing CCVs.

Actual Procedure/process:
The Matrix spike (MSD 4/28/98) analyzed on 05/05/98 was above the range of the calibration curve.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written.

Recorded By: Lisa A Clemen Date: 02/13/01

IV. Impact on Study / Project

The sample result will be flagged in the data as an estimate. KH 02/13/01

Authorized By *(Study Director / Project Lead)* Date

Sponsor Representative: Marvin Case Marvin T Case 15 Feb 2001
Study Director: John Butenhoff John Z. Butenhoff 16 FEB 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 3
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No.
TOX001 (LIMS #U2103)

Deviation type *(Check one)*: ❒ SOP X Method ❒ Equipment Procedure ❒ Protocol ❒ Other:

Document number FACT-M-4.0 and ETS-8-5.1

Date(s) of occurrence
05/05/98, 4/20/00, 4/21/00, 5/02/00, 5/24/00, 5/30/00, 8/23/00, 02/28/00, 03/15/00, 02/27/00, 04/17/00, 04/18/00, 04/19/00, 05/31/00, 06/19/00, 06/20/00, 06/21/00, 08/02/00, 04/06/00

II. Description

Required procedure/process:
Section 10.1.2: Analyze a method blank and a matrix blank prior to each calibration curve.

Actual procedure/process:
On several occasions, the blanks were either a) analyzed after the calibration curves or b) not analyzed at all for a particular MS run.

III. Actions Taken
(such as amendment issued, SOP revision, etc.)

Deviation written.

Recorded by: kjh Date: 04/10/01

IV. Impact on Study / Project
(completed by Study Director or Project Lead)

All blanks were analyzed at least once during the course of the study. When blanks were run after the calibration curve, all control-of-bias practices were followed. In the instances where blanks were not run at all, the samples being analyzed were usually well above the typical run LOQ. In addition, the vast majority of samples run without blanks were high level dilutions. The function of the blank is to determine if contamination was introduced during extraction; any low-level contamination would be inconsequential to extracts requiring dilution. This deviation has no adverse affect on the study data.

kjh 04/10/01

Authorized by: Date:

Sponsor Representative: Marvin Case Marvin T. Case 30 April 2001
Study Director: John Butenhoff John L. Butenhoff APRIL 23 2001

Deviation No. 4

Record of Deviation

I. Identification

Study / Project No.
TOX001 (LIMS #U2103) Covance 6329-212

Deviation type (Check one): ❒ SOP X Method ❒ Equipment Procedure ❒ Protocol ❒ Other:

Document number: ETS-8-5.1 (Modified method) ETS-8-7.0 (Modified method)

Date(s) of occurrence: 05/05/98 (ETS-8-5.1), 02/18/99, 12/06/00 (ETS-8-7.0)

II. Description

Required procedure/process:
Section 14.3.6: A valid calibration curve must contain at least 5 active points

Actual procedure/process:
A suitable calibration curve (i.e. with all points within 30% of theoretical and with an r^2>0.980) could not be derived from 5 points; four points were used for the calibration curve for PFOS and PFOSAA (5/5/98), for Et-FOSE-OH (2/18/99), and for PFOSAA (12/06/00).

III. Actions Taken
(such as amendment issued, SOP revision, etc.)

Deviation written. Data evaluated against 4 point curve is specifically marked in the table of final results.

Recorded by: Lisa A Clemen Date: 02/13/01

IV. Impact on Study / Project
(completed by Study Director or Project Lead)

The data quality of samples evaluated against the four-point curve is adversely affected.

kjh 02/13/01

Authorized by Date

Sponsor Representative: Marvin Case Marvin T Case 15 Feb 2001 **Deviation No.** 5
(assigned by Study Director or Project Lead at the end of study or project)

Study Director: John Butenhoff John L. Butenhoff 16 FEB 2001

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) ☐ SOP ☐ Method ☐ Equipment Procedure X Protocol ☐ Other:

Document Number(s): FACT-TOX-001

Date(s) of occurrence: 05/06/98, 05/13/98, 05/14/98, 05/15/98,. 05/17/98, 06/03/98, 06/05/98, 06/12/98, 06/14/98, 06/18/98, 06/25/98, 06/26/98, 07/06/98, 07/09/98, 12/14/98, 12/29/98, 01/06/99, 01/18/99, 02/16/99, 02/18/99

II. Description:

Required Procedure/process:
The protocol states: Methods FACT-M-2.0 and FACT-M-4.0 are to be used to analyze liver and sera extracts.

Actual Procedure/process:
Methods were not documented or the incorrect methods were listed for analyses of these extracts.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written.

Recorded By: Lisa A. Clemen Date: 05/17/01

IV. Impact on Study / Project

These analyses were reviewed and these data were analyzed according to methods as listed in the protocol. No adverse impact on the outcome of this study. bjh 05/22/01

Authorized By *(Study Director / Project Lead)*: John Z. Butenhoff 5/24/01 Date:

SD: John Butenhoff Sponsor: Marv Cast 31 May 2001

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Deviation No. 6
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one): ❒ SOP X Method ❒ Equipment Procedure ❒ Protocol ❒ Other:

Document Number(s): ETS-8-5.1

Date(s) of occurrence: 05/14/98

II. Description:

Required Procedure/process:

The continuing calibration verification (CCV) must be within +/- 30% of the theoretical concentration.

Actual Procedure/process:

The Week 8, Sera, PFOS and PFOSAA MS, MSD, H2O Blank, and Sera Blank data were entered even though CCVs did not meet criteria.

The CCV analyzed prior to the PFOS data was within criteria and the curve immediately following these data were within criteria. Three CCVs in a row, prior to the good CCV, failed the 30% criteria.

Two CCVs analyzed prior to the PFOSAA data were within criteria and the curve immediately following these data were within criteria. Two CCVs prior to the good CCVs failed the 30% criteria.

III. Actions Taken:

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

This deviation was written and data will be flagged as tentative in the raw data.

Recorded By: Lisa A Clemen Date: 01/08/01

IV. Impact on Study / Project

Affected data will not be within +/-30% criteria.

Authorized By *(Study Director / Project Lead)*: Marvin T. Case 10 Jan 2001 John L. Butenhoff 10 JAN 2001 kh 01/08/01

Sponsor Representative: Marvin Case Study Director: John Butenhoff

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Deviation No. 7
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) ❒ SOP X Method ❒ Equipment Procedure ❒ Protocol ❒ Other:

Document Number(s): ETS-8-7.0

Date(s) of occurrence: 05/29/98, 06/01/98

II. Description:

Required Procedure/process:
A five point curve or better is used for plotting the calibration curve.

Actual Procedure/process:
A four point curve was used for plottting the EtFOSE calibration curve for the Week 14, Liver curve, generated on 12/14/98.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
These data were flagged in the raw data and this deviation was written.

Recorded By: Lisa A Clemen

Date: 02/19/01

IV. Impact on Study / Project

EtFOSE-OH data is flagged as qualitative, so this data will not be adversely affected.
KH 02/19/01

Authorized By *(Study Director / Project Lead)*

Date

Sponsor Representative: Marvin Case Marvin T Case 21 Feb 2001
Study Director: John Butenhoff John L. Butenhoff 21 FEB 2001

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Form ETS-4-8.0

Deviation No. 8
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) ❒ SOP ❒ Protocol X Method ❒ Equipment Procedure ❒ Other:

Document Number(s): ETS-8-7.0

Date(s) of occurrence: 06/02/98, 01/14/99, 01/17/99

II. Description:

Required Procedure/process:
Section 14.4.1 states: matrix spike percent recoveries must be within ± 30% of the spiked concentration.

Actual Procedure/process:
The week 14, liver matrix spike and matrix spike duplicate samples prepared for PFOS and PFOSAA analysis (extracted 06/02/98) weren't within criteria. The non-compliance of the PFOS and PFOSAA data on 06/02/98 can be attributed to inappropriate preparation: spikes were prepared at 100 ng/g, a level not distinguishable from background levels of the analyte in the controls. Spikes were reprepped at a higher level (250 ng/g) on 01/14/99, but again, for PFOS analysis, the spike level was not distinguishable from background levels; PFOSAA was inadvertently omitted from analysis. Spikes for the week 14 liver samples were prepared a third time on 01/17/99 at 750 ng/g. Results of the third analysis showed an average PFOS recovery of 132%; one of the two matrix spikes was recovered outside the stated 130% criteria (138%). Results of matrix spike recovery from PFOSAA at the 750 ng/g level were acceptable.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written. A note will be added to the week 14 liver data stating that one PFOS MS was recovered at 132% and that week 14 PFOS analytical results may be biased high.

Recorded By: Lisa A Clemen Date: 02/19/01

IV. Impact on Study / Project

PFOS results for week 14 liver samples may be biased high. hjh 02/19/01

Authorized By *(Study Director / Project Lead)* Date

Sponsor Representative: Marvin Case Marvin T Case 21 Feb 2001
Study Director: John Butenhoff John Butenhoff 21 FEB 2001

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Form ETS-4-8.0

Deviation No. 9
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) ❒ SOP X Method ❒ Equipment Procedure ❒ Protocol ❒ Other:

Document Number(s): ETS-8-7.0 | Date(s) of occurrence: 06/03/98

II. Description:

Required Procedure/process:
Section 14.4.1 states: matrix spike percent recoveries must be within ± 30% of the spiked concentration.

Actual Procedure/process:
All week 4 liver MS recoveries are outside criteria, however, the matrix spike and sample data appeared to be mislabeled.
When the MS and sample labels are corrected, all recoveries are within criteria with the Exception of PFOS and EtFOSE. Average PFOS recoveries are 192% and average EtFOSE Recoveries are 133%.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
The MS and sample data labels were corrected in the raw data, the PFOS data were flagged in the spreadsheet, sample results are noted to have a potential high bias, and this deviation was written.

Recorded By: Lisa A Clemen | Date: 02/19/01

IV. Impact on Study / Project

PFOS data in week 4 liver samples is noted to be potentially biased high. EtFOSE-OH data Are already designated as qualitative only and is not further affected by the deviant recoveries.
kjh 02/19/01

Authorized By *(Study Director / Project Lead)* | Date

Sponsor Representative: Marvin Case Marvin T Case 21 Feb 2001
Study Director: John Butenhoff John L Butenhoff 21 FEB 2001

3M Environmental Laboratory
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Deviation No. 10
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one): ☐ SOP ☐ Protocol **X** Method ☐ Equipment Procedure ☐ Other:

Document Number(s): ETS-8-7.0

Date(s) of occurrence: 12/29/98

II. Description:

Required Procedure/process:
Section 14.5.1 states: Continuing calibration verification percent recoveries must be within +/-30% of the spiked concentration.

Actual Procedure/process:
The continuing calibration verification samples for PFOS, PFOSA, and PFOSAA analyzed on 12-29-98 had percent recoveries greater than 130% of expected.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written; affected data is noted in the data tables

Recorded By: [signature] Date: 2.13.01

IV. Impact on Study / Project

Affected data may be biased high. kh 02/13/01

Authorized By *(Study Director / Project Lead)* Date

Sponsor Representative: Marvin Case [signature] 15 Feb 2001
Study Director: John Butenhoff [signature] 16 FEB 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 11
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) ❒ SOP X Method ❒ Equipment Procedure ❒ Protocol ❒ Other:

Document Number(s): ETS-8-7.0 Date(s) of occurrence: 01/18/99

II. Description:

Required Procedure/process:
Section 14.5.1 states: Continuing calibration verification percent recoveries must be within ± 30% of the spiked concentration.

Actual Procedure/process:
The Week 14, liver samples C91281F, C91288F, C91293F, C91299F, and C91304F were bracketed by a continuing calibration verification that did not meet the +/- 30% criteria. (-31%)

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
These data were flagged in the raw data as having a potential low bias and this deviation was written.

Recorded By: Lisa A Clemer Date: 02/19/01

IV. Impact on Study / Project

Results for these analyses may be biased low.

Authorized By *(Study Director / Project Lead)* Date

Sponsor Representative: Marvin Case Marvin T Case 21 Feb 2001
Study Director: John Butenhoff John R. Butenhoff 21 FEB 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 12
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) ☐ SOP ☐ Method ☐ Equipment Procedure X Protocol ☐ Other:

Document Number(s): FACT-TOX-001

Date(s) of occurrence: 02/07/00, 02/08/00, 02/09/00

II. Description:

Required Procedure/process:
The protocol states: Method FACT-M-3.0 is to be used when extracting the serum samples.

Actual Procedure/process:
Method ETS-8-4.1 was used to extract these serum samples.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written.

Recorded By: John A. Clemen

Date: 05/17/01

IV. Impact on Study / Project

No adverse impact on the outcome of this study. Sera method ETS-8-4.1 is an improvement over sera method FACT-M-3.0.

MJ 05/22/01

Authorized By *(Study Director / Project Lead)*: John Z Bartholomew 5/24/01

Date:

Marvin Case Sponsor Marvin T Case 31 May 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 13
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212 LIMS U2103

Deviation Type (Check one) ☐ SOP X Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number(s): ETS-8-7.0

Date(s) of occurrence: 02/22/00, 04/24/00, 04/26/00, 04/28/00, 05/24/00, 07/28/00, 08/01/00, 08/14/00, 08/15/00, 08/21/00, 08/23/00, 03/22/01

II. Description:

Required Procedure/process:

Section 10.1.2 states: Analyze a method blank and a matrix blank prior to each calibration curve.

Actual Procedure/process:

On several occasions, the blanks were either a) analyzed after the calibration curves or b) not analyzed At all for a particular MS analysis.

III. Actions Taken:

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

This deviation was written.

KH 04/19/01

Recorded By | Date

IV. Impact on Study / Project

All blanks were analyzed at least once during the course of the study. When blanks were analyzed after the calibration curve, all control-of-bias practices were followed. In the instances where blanks were not analyzed at all, the samples being analyzed were usually well above the typical run LOQ. In addition, the vast majority of samples analyzed without blanks were high level dilutions. The function of the blank is to determine if contamination was introduced during extraction; any low-level contamination would be inconsequential to extracts requiring dilutions. This deviation has no adverse affect on these study data.

KH 04/19/01

Authorized By *(Study Director / Project Lead)* | Date

Sponsor Representative: Marvin Case Marvin T Case 23 April 2001

Study Director: John Butenhoff

John Z. Butenhoff 23/APRIL/2001

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Deviation No. 14
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No.
FACT- TOX-001 Covance 6329-212

Deviation Type (Check one) ☐ SOP ☒ Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number	Date(s) of occurrence
ETS-8-5.1	06/21/00

II. Description:

Required Procedure/process:

Method states: Analyze a mid-range calibration standard after every tenth sample, with a minimum of one per batch.
Continuing calibration verification percent recoveries must be ±30% of the spiked concentration.

Actual Procedure/process:

Data for the group 6 extracts (wk 105) were reported for PFOSAA and EtFOSE-OH even though two consecutive continuing calibration verifications were ≥30%. These extracts were analyzed 3 times with the same results each time. It appears to be a matrix effect.
Instead of 10 samples bracketed there were 15 samples bracketed by continuing calibration checks within the set criteria.

III. Actions Taken:

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

Data were footnoted in the raw data and this deviation was written.

Recorded By	Date
Lisa A Clemen	06/26/00

IV. Impact on Study / Project

No Impact. kjn 08/28/00

Authorized By *(Study Director / Project Lead)*	Date
John Z. Butenhoff 9/15/00 / Maurice T Case 15 Sept 2000	

Study Director: John Butenhoff Sponsor Rep: Marv Case

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 15
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-001 Covance 6329-212

Deviation Type (Check one) [] SOP [] Method [] Equipment Procedure X Protocol [] Other:

Document Number(s): FACT-TOX-001

Date(s) of occurrence: 07/28/00, 08/01/00, 08/14/00, 08/15/00, 08/21/00, 08/23/00, 08/28/00, 08/29/00

II. Description:

Required Procedure/process:
The protocol states: Method ETS-8-7.0 is to be used to analyze liver extracts.

Actual Procedure/process:
Method ETS-8-6.0 was incorrectly listed as the method used to analyze these liver extracts.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written.

Recorded By: Lisa A. Clemen

Date: 05/17/01

IV. Impact on Study / Project

ETS-8-6.0 is the extraction method, ETS-8-7.0 is the analytical method. These analyses were reviewed and data were analyzed according to ETS-8-7.0. No adverse impact on the outcome of this study. MH 05/22/01

Authorized By *(Study Director / Project Lead)*: John R. Butenhoff May 24, '01

Date:

SD: John Butenhoff

Sponsor: Mary Case Marvin Case 31 May 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 16
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. TOX001

Deviation Type (Check one) ☐ SOP ☒ Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number: ETS 8-7.0

Date(s) of occurrence: 3-23-01

II. Description:

Required Procedure/process:

CCVs must be within the ±30% Criteria.

Actual Procedure/process:

The CCV was effected by carryover from the two preceding samples and was outside acceptance criteria.

III. Actions Taken:

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

The data is bracketed by the final curve. The PFOS value for C910746-17 grb df:10 will be reported.

Recorded By: Marlene Thy

Date: 3-28-01

IV. Impact on Study / Project

The reported data was not affected by carryover + sufficient QC in the form of the calibration curve, adequately brackets the data. No adverse affect.

Authorized By *(Study Director / Project Lead)*: Gh 03/23/01

Date:

3M Environmental Laboratory
Form ETS-4-8.0
Study Director : John Butenhoff

John R. Butenhoff April 9, 2001

Deviation No. 17
(assigned by Study Director or Project Lead at the end of study or project)

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
LRN-U2103

Appendix C: Extraction and Analytical Methods

This appendix includes the following methods:

Preparatory Methods

FACT-M-1.0. Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry, (8 pages)

FACT-M-3.0, Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry, (8 pages)

ETS-8-4.1, Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry, (14 pages)

ETS-8-6.0, Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry, (14 pages)

Analytical Methods

FACT-M-2.0, Analysis of Liver Extracts for Fluorochemicals Using HPLC-Electrospray/Mass Spectrometry, (8 pages)

FACT-M-4.0, Analysis of Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry, (8 pages)

ETS-8-5.1, Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry, (9 pages)

ETS-8-7.0, Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry, (10 pages)

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER ANIONIC FLUOROCHEMICAL SURFACTANTS FROM LIVER FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: FACT-M-1.0

Adoption Date: 5/26/98

Revision Date: N/A

Author: Lisa Clemen

Approved By:

Laboratory Manager — Date: 5/26/98

Group Leader — Date: 5/26/98

Technical Reviewer — Date: 5/27/98

Initial: DSD
Date: 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the extraction of Potassium Perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from liver.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds.

1.3 Matrices: Rabbit, rat, bovine, and monkey livers or other livers as designated in the validation report.

2.0 Summary of Method

2.1 This method describes how to extract potassium perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from liver using ion pairing reagent and 5.0 mLs of ethyl acetate. An ion pairing reagent is added to each sample and partitioned into ethyl acetate. Four mLs of extract is removed to a centrifuge tube and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 1.0 mL methanol then filtered through a 3 cc plastic syringe attached to a 0.2 µm filter into glass autovials.

3.0 Definitions

3.1 None.

4.0 Warnings and Cautions

4.1 Health and Safety Warnings:

4.1.1 Use universal precautions when handling animal livers, they may contain pathogens.

5.0 Interferences

5.1 There are no known interferences at this time.

6.0 Equipment

6.1 The following equipment is used while carrying out this method. Equivalent equipment is acceptable.

6.1.1 Ultra-Turrax T25 Grinder for grinding liver samples
6.1.2 Vortex mixer, VWR, Vortex Genie 2
6.1.3 Centrifuge, Mistral 1000 or IEC
6.1.4 Shaker, Eberbach or VWR
6.1.5 Nitrogen Evaporator, Organomation
6.1.6 Balance

7.0 Supplies and Materials

7.1 Gloves
7.2 Dissecting scalpels
7.3 Eppendorf or disposable pipettes
7.4 Nalgene bottles, capable of holding 250 mL and 1 L
7.5 Glass, type A, volumetric flasks
7.6 40 mL glass I-CHEM vials
7.7 Plastic sampule vials, Wheaton, 6 mL
7.8 Polypropylene centrifuge tubes, 15 mL
7.9 Labels

7.10 Syringes, capable of measuring 10 µL to 50 µL

7.11 Glass, type A, volumetric pipettes

7.12 Graduated pipettes

7.13 Electronic pipettor, Eppendorf or equivalent

7.14 Timer

7.15 Disposable plastic 3 cc syringes

7.16 Filters, nylon syringe filters, 0.2 µm, 25 mm

7.17 Crimp cap autovials

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 Reagents and Standards

8.1 Reagents

8.1.1 Sodium Hydroxide (J.T Baker or equivalent), (NaOH) 10N: weigh approximately 200 grams NaOH. Pour into a 1000 mL beaker containing 500 liters (L) Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L nalgene bottle.

8.1.2 Sodium Hydroxide (J.T Baker or equivalent), (NaOH) 1N. Dilute 10N 1:10. Measure 10 mL of the 10N NaOH solution into a 100 mL volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 mL nalgene bottle.

8.1.3 Tetrabutylammonium hydrogen sulfate (Kodak or equivalent), (TBA) 0.5M: Weigh approximately 169 grams of TBA into a 1 L volumetric containing 500 L Milli-Q™ water. Adjust to pH 10 using approximately 64 mL 10N NaOH and dilute to volume with Milli-Q™ water. Add NaOH slowly while adding the last 1 mL of NaOH because the pH changes abruptly. Store in a 1 L nalgene bottle.

8.1.3.1 TBA requires a check prior to each use to ensure pH = 10. Adjust as needed using 1N NaOH solution.

8.1.4 Sodium carbonate/Sodium Bicarbonate Buffer (J.T. Baker or equivalent), (Na_2CO_3/$NaHCO_3$) 0.25M: Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and dilute to volume with Milli-Q™ water. Store in a 1 L nalgene bottle.

8.1.5 PFOS (3M Specialty Chemical Division), molecular weight = 538.

8.1.6 Ethyl Acetate, Omnisolv, glass distilled or HPLC grade.

8.1.7 Methanol, Omnisolv, glass distilled or HPLC grade.

8.1.8 Liver and control liver, received frozen from testing laboratory.

8.1.9 Milli-Q™ water, all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus system.

8.2 Standards

8.2.1 Prepare PFOS standards for the standard curve.

8.2.2 Weigh approximately 100 mg of PFOS into a 100 mL volumetric flask and record the actual weight.

8.2.3 Bring to volume with methanol for a stock standard of approximately 1000 ppm (µg/mL).

8.2.4 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.2.5 Dilute the stock solution with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.2.6 Dilute the stock solution with methanol for a working standard 3 solution of approx. 0.50 ppm.

9.0 SAMPLE HANDLING

9.1 All livers are received frozen and must be kept frozen until the extraction is performed.

10.0 QUALITY CONTROL

10.1 Matrix Spikes

10.1.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.1.2 Prepare each spike using liver chosen by the analyst, usually a control liver.

10.1.3 Expected concentrations will fall in the mid-range of the initial calibration curve.

10.2 Continuing Calibration Checks

10.2.1 Prepare and analyze continuing calibration check samples to determine the continued linearity of the initial calibration curve.

10.2.2 One check is prepared per group of ten samples. For example, if a sample set = 34, four checks are prepared and extracted.

10.2.3 Prepare each continuing calibration check from the same liver homogenate used to prep the initial curve.

10.2.4 The expected concentration will fall within the mid-range of the initial calibration curve.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare Liver Homogenate to Use for Standards

11.1.1 Weigh approximately 40 g of liver into a 250 mL Nalgene bottle containing 200 mLs Milli-Q™ water. Grind to a homogeneous solution.

11.1.2 If 40 g is not available, use appropriate amounts of liver and water in keeping with a 1:5 ratio.

11.1.3 See section 13.0 to calculate the actual density of liver.

11.1.4 Add 1 mL of homogeneous solution to a 15 mL centrifuge tube. Re-suspend homogeneous solution by shaking between aliquots while preparing a total of sixteen 1 mL aliquots of homogeneous solution in 15 mL centrifuge tubes.

11.1.5 Two 1 mL aliquots serve as matrix blanks. Use the standard concentrations and spiking amounts listed in table 1 to spike, in duplicate, two standard curves for a total of fourteen samples.

Table 1
Approximate Spiking Amounts for Calibration Standards

Working Standard (Approx. Conc.)	µL	Approx. final conc. of PFOS in liver
-	-	Blank
0.50 ppm	4	0.010 ppm
0.50 ppm	20	0.050 ppm
0.50 ppm	40	0.100 ppm
5.0 ppm	10	0.250 ppm
5.0 ppm	20	0.500 ppm
5.0 ppm	30	0.750 ppm
50 ppm	4	1.000 ppm

11.1.1 See section 13.0 to calculate actual concentrations of PFOS in calibration standards.

11.2 Extract spiked liver homogenates following **12.14-12.24** of this method. Use these standards to establish each initial curve on the mass spectrometer.

12.0 PROCEDURES

12.1 Obtain frozen liver samples. In spent tissue, note that the liver has not been packaged with other tissues.

12.2 Cut approximately 1 g of liver using a dissecting scalpel.

12.3 Weigh the sample directly into a tared plastic sampule vial.

12.4 Record the liver weight in the study notebook.

12.5 Label the sampule vial with the study number, weight, liver ID, date and analyst initials.

12.6 Add 2.5 mLs of water to sampule vial.

12.7 Grind the sample. Put the grinder probe in the sample and grind for about 2 minutes, or until the sample is homogeneous.

12.8 Rinse the probe into the sample with 2.5 mLs water using a pipette.

12.9 Take the grinder apart and clean it with methanol after each sample. Follow AMDT-EP-22.

12.10 Cap the sample and vortex for 15 seconds.

12.11 Pipette 1 mL homogenate into a 15 mL polypropylene centrifuge tube. Label the centrifuge tube with the identical information as the sampule vial. (See Worksheet for documenting the remaining steps.)

12.12 Spike liver homogenates with the appropriate amount of PFOS standard as described in section 11.1 or Table 1.

12.13 Pipette two 1 mL aliquots of Milli-Q™ water to centrifuge tubes. These will serve as instrument blanks.

12.14 Add 1 mL 0.5 M TBA and 2 mL of the 0.25 M sodium carbonate/sodium bicarbonate buffer.

12.15 Using a volumetric pipette, add 5 mLs ethyl acetate.

12.16 Cap each sample and put on the shaker for 20 minutes.

12.17 Centrifuge for 20 to 25 minutes, until layers are well separated. Set power on the centrifuge to approximately 3500 rpm.

12.18 Remove 4 mLs of organic layer, using a 5 mL graduated glass pipette, to a clean 15 mL centrifuge tube. Label this fresh tube with the same information as in **12.5**.

12.19 Put each sample on the analytical nitrogen evaporator until dry, approximately 2 to 3 hours.

12.20 Add 1.0 mL of methanol to each centrifuge tube using a graduated pipette.

12.21 Vortex mix for 30 seconds.

12.22 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 mL glass autovial.

12.23 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, and analyst(s) who performed the extraction.

12.24 Cap and hold for electrospray mass spectrometry analysis.

12.25 Complete the worksheet and tape to page of study notebook.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.1 Calculate the density of liver (mg) in 1.0 mL homogenate using the following equation:

$$\frac{\text{g of Liver} \times \text{Average weight of ten 1 mL aliquots (mg)}}{(\text{g of Liver} + \text{g of Water})}$$

13.1.2 Calculate actual concentrations of PFOS in calibration standards using the following equation:

$$\frac{\mu L \text{ of Standard} \times \text{Concentration } (\mu g/mL)}{\text{mg Liver}^{*} / 1 \text{ mL homogenate}} = \text{Final Concentration } (\mu g/g \text{ or } mg/kg) \text{ of PFOS in Liver}$$

*Average weight of liver in solution as determined in **13.1.1**, by weighing ten 1 mL homogenates of approximately 40 mg liver in 200 mL of Milli-Q water.

14.0 METHOD PERFORMANCE

14.1 The method detection limit is equal to half the lowest standard in the calibration curve.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

16.0 RECORDS

16.1 Complete the extraction worksheet and tape into the study notebook.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 The validation report associated with this method is FACT-M-1.0 & 2.0-V-1.

18.0 REFERENCES

18.1 AMDT-EP-22, "Routine Maintenance of Ultra-Turrax T-25"

19.0 AFFECTED DOCUMENTS

19.1 FACT-M-2, "Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

Extraction Worksheet for FACT-M-1

Study #	Sample Number set #	PFOS approx. 0.5 ppm actual ppm #W_____	PFOS approx. 5 ppm actual ppm #W_____	PFOS approx. 50 ppm actual ppm #W_____	Date and Initials for Std.
-	H_2O Blank	-	-	-	
-	_____ Liver Blank	-	-	-	
-					
-					
-					
-					
-					
-					
-					
-					
[1]		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
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		-	-	-	
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		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

[1] Study number where the original worksheet is located.

Blank Liver Homogenate: Std # Liver amount = g

Liver Extraction Method :	**Date & Initials**
Vortex 15 sec.	
Pipette 1 mL of Liver Solution	
Pipette 1 mL of †0.5 M TBA, pH 10. Std. #_____	
Pipette 2 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ Buffer Std. #_____	
Pipette 5 mL of Ethyl Acetate TN-A-_____	
Shake 20 min.	
Centrifuge 20-25 min. Centrifuge Speed	
Remove a 4 mL aliquot of organic layer	
Put on Nitrogen Evaporator to dryness Evaporator Temperature	
Add 1.0 mL of Methanol TN-A-_____	
Vortex 30 sec.	
Filter using a 3cc B-D syringe with a 0.2µm SRI filter into a 1.5 mL autosample vial	

MS/MSD/___ Cont. Checks: Spiked _____ uL of a _____ ppm std (_____) for a final concentration of _____ ppm. MS/MSD used sample _____. Cont. Checks used same homogenate as for std curve.

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER ANIONIC FLUOROCHEMICAL SURFACTANTS FROM SERUM FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: FACT-M-3.0 **Adoption Date:** 4/22/98

Revision Date: N/A

Author: Lisa Clemen

Approved By:

Laboratory Manager 4/22/98 Date

Group Leader 4/22/98 Date

Technical Reviewer 4/22/98 Date

Exact Copy of Original
Initial RSD Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the extraction of potassium perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from serum.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds.

1.3 Matrices: Rabbit, rat, and bovine serum or other sera as designated in the validation report.

2.0 Summary of Method

2.1 This method describes how to extract potassium perfluorooctanesulfonate (PFOS) or other anionic fluorochemical surfactants from serum using an ion pairing reagent and 5.0 mL of ethyl acetate. An ion pairing reagent is added to the sample and the analyte ion pair is partitioned into ethyl acetate. Four mL of extract are removed and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 1.0 mL of methanol, then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

3.0 Definitions

3.1 None.

4.0 Warnings and Cautions

4.1 Health and Safety Warnings:

4.1.1 Use universal precautions, especially laboratory coats, goggles, and gloves when handling animal serum, it may contain pathogens.

5.0 Interferences

5.1 There are no known interferences at this time.

6.0 Equipment

6.1 The following equipment is used while carrying out this method. Equivalent equipment is acceptable.

6.1.1 Vortex mixer, VWR, Vortex Genie 2
6.1.2 Centrifuge, Mistral 1000 or IEC
6.1.3 Shaker, Eberbach or VWR
6.1.4 Nitrogen evaporator, Organomation
6.1.5 Balance, (± 0.100 gm)

7.0 Supplies and Materials

7.1 Gloves
7.2 Eppendorf or disposable pipettes
7.3 Nalgene bottles, capable of holding 250 mL and 1 L
7.4 Glass, type A, volumetric flasks
7.5 40 mL glass I-CHEM vials
7.6 Polypropylene centrifuge tubes, 15 mL
7.7 Labels
7.8 Syringes, capable of measuring 10 µL to 50 µL
7.9 Glass, type A, volumetric pipettes
7.10 Graduated pipettes

7.11 Electronic pipettor, Eppendorf or equivalent

7.12 Timer

7.13 Disposable plastic 3 cc syringes

7.14 Filters, nylon syringe filters, 0.2 µm, 25 mm

7.15 Crimp cap autovials

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 Reagents and Standards

8.1 Reagents

8.1.1 Sodium hydroxide (J.T Baker or equivalent), (NaOH) 10N: weigh approximately 200 grams NaOH. Pour into a 1000 mL beaker containing 500 liters (L) Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L Nalgene bottle.

8.1.2 Sodium hydroxide (J.T Baker or equivalent), (NaOH) 1N. Dilute 10N 1:10. Measure 10 mL of 10N NaOH solution into a 100 mL volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 mL Nalgene bottle.

8.1.3 Tetrabutylammonium hydrogen sulfate (Kodak or equivalent), (TBA) 0.5M: Weigh approximately 169 grams of TBA into a 1 L volumetric containing 500 L Milli-Q™ water. Adjust to pH 10 using approximately 64 mL of 10N NaOH and dilute to volume with Milli-Q™ water. Add NaOH slowly while adding the last mL of NaOH because the pH changes abruptly. Store in a 1 L Nalgene bottle.

8.1.3.1 TBA requires a check prior to each use to ensure pH = 10. Adjust as needed using 1N NaOH solution.

8.1.4 Sodium carbonate/sodium bicarbonate buffer (J.T. Baker or equivalent), (Na_2CO_3/$NaHCO_3$) 0.25M: Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and bring to volume with Milli-Q™ water. Store in a 1 L nalgene bottle.

8.1.5 PFOS (3M Specialty Chemical Division), molecular weight = 538.

8.1.6 Other fluorochemicals, as appropriate.

8.1.7 Ethyl Acetate, Omnisolv, glass distilled or HPLC grade.

8.1.8 Methanol, Omnisolv, glass distilled or HPLC grade.

8.1.9 Serum, frozen liquid from Sigma.

8.1.10 Control serum received with each sample set.

8.1.11 Milli-Q™ water, all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus system.

8.2 Standards

8.2.1 Prepare PFOS standards for the standard curve.

8.2.2 Prepare other fluorochemical standards, as appropriate.

8.2.3 Weigh approximately 100 mg of PFOS into a 100 mL volumetric flask and record the actual weight.

8.2.4 Bring to volume with methanol for a stock standard of approximately 1000 ppm (µg/mL).

8.2.5 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.2.6 Dilute the stock solution with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.2.7 Dilute the stock solution with methanol for a working standard 3 solution of approx. 0.50 ppm.

9.0 SAMPLE HANDLING

9.1 All sera are received frozen and must be kept frozen until the extraction is performed.

10.0 QUALITY CONTROL

10.1 Matrix Blanks and Method Blanks

10.1.1 Two 1.0 mL aliquots of the serum are extracted following this procedure and used as matrix blanks. See section 11.1.2.

10.1.2 Two 1.0 mL aliquots of Milli-Q™ water are extracted following this procedure and used as method blanks.

10.2 Matrix Spikes

10.2.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.2.2 Prepare each spike using serum chosen by the analyst, usually control serum received with each sample set.

10.2.3 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spikes may be included and may fall in the low-range of the initial calibration curve.

10.3 Continuing Calibration Checks

10.3.1 Prepare and analyze continuing calibration check samples to determine the continued linearity of the initial calibration curve.

10.3.2 One check is prepared per group of ten samples. For example, if a sample set = 34, four checks are prepared and extracted.

10.3.3 Prepare each continuing calibration check from the same serum used to prep the initial curve.

10.3.4 The expected concentration will fall within the mid-range of the initial calibration curve.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare Serum Standards

11.1.1 Transfer 1 mL of serum to a 15 mL centrifuge tube.

11.1.2 If the majority of serum sample volumes are less than 1.0 mL, extract standards using serum volumes in the standards equal to the serum volumes in samples. Do not extract below 0.50 mL of serum. Record the serum volume on the extraction sheet.

11.1.3 Mix or shake between aliquots while preparing a total of sixteen aliquots of serum in 15 mL centrifuge tubes.

11.1.4 Two 1 mL or appropriate aliquots serve as matrix blanks. Typically use the standard concentrations and spiking amounts listed in table 1 to spike, in duplicate, two standard curves for a total of fourteen samples.

11.1.5 Refer to the validation report FACT-M-3.0-V-1 and FACT-M-4.0-V-1 which lists the working ranges for calibration curves.

Table 1
Approximate Spiking Amounts for Standards and Spikes Using 1.0 mL of Serum

Working Standard (Approx. Conc.)	µL	Approx. final conc. of PFOS in serum
-	-	Blank
0.500 ppm	20	0.010 ppm
5.00 ppm	5	0.025 ppm
5.00 ppm	10	0.050 ppm
5.00 ppm	20	0.100 ppm
50.0 ppm	5	0.250 ppm
50.0 ppm	10	0.500 ppm
50.0 ppm	15	0.750 ppm

11.1.4 See section 13.0 to calculate actual concentrations of PFOS in calibration standards.

11.2 Extract spiked serum standards following **12.6-12.16** of this method. Use these standards to establish each initial curve on the mass spectrometer.

12.0 PROCEDURES

12.1 Obtain frozen serum samples and allow to thaw.

12.2 Vortex mix for 15 seconds then remove 1.0 mL or appropriate volume to a 15 mL polypropylene centrifuge tube.

12.3 Return serum samples to freezer after extraction amount has been removed.

12.4 Record the serum volume on the extraction worksheet. The final methanol volume will equal the initial serum volume.

12.5 Label the tube with the study number, serum ID, date and analyst initials. See attached worksheet for documenting the remaining steps.

12.6 Spike serum with the appropriate amount of PFOS standard as described in section 11.1 or Table I for the calibration curve standards. Also spike matrix spikes and continuing calibration standards.

12.7 Vortex mix the standard curve samples, matrix spike samples, and continuing calibration samples for 15 seconds.

12.8 Add 1 mL 0.5 M TBA and 2 mL of the 0.25 M sodium carbonate/sodium bicarbonate buffer.

12.9 Using a volumetric pipette, add 5 mL ethyl acetate.

12.10 Cap each sample and put on the shaker for 20 minutes.

12.11 Centrifuge for 20 to 25 minutes, until layers are well separated. Set power on the centrifuge to approximately 3500 rpm.

12.12 Transfer 4 mL of organic layer, using a 5 mL graduated glass pipette, to a clean 15 mL centrifuge tube. Label this fresh tube with the same information as in **12.5**.

12.13 Put each sample on the analytical nitrogen evaporator until dry, approximately 2 to 3 hours.

12.14 Add 1.0 mL or appropriate volume of methanol to each centrifuge tube using a graduated pipette. (This volume equals the initial volume of serum used for the extraction.)

12.15 Vortex mix for 30 seconds.

12.16 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 mL glass autovial.

12.17 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, and analyst(s) who performed the extraction.

12.18 Cap and hold for HPLC-electrospray/mass spectrometry analysis. Extracts may be stored at 4° C until analysis.

12.19 Complete the extraction worksheet, attached to this document, and tape to page of study notebook.

13.0 Data Analysis and Calculations

13.1 Calculations:

13.1.1 Calculate actual concentrations of PFOS, or other appropriate fluorochemical, in calibration standards using the following equation:

$$\frac{\text{mL of Standard x Concentration } (\mu g/mL)}{\text{mL of Standard + Initial Serum Volume (mL)}} = \text{Final Concentration } (\mu g/mL) \text{ of PFOS in Serum}$$

14.0 Method Performance

14.1 The method detection limit is equal to half the lowest standard in the calibration curve.

15.0 Pollution Prevention and Waste Management

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

16.0 Records

16.1 Complete the extraction worksheet attached to this method, and tape into the study notebook.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 The validation report associated with this method is FACT-M-3.0 & 4.0-V-1.

18.0 References

18.1 None

19.0 Affected Documents

19.1 FACT-M-4, "Analysis of Serum Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

20.0 Revisions

Revision Number.	Reason For Revision	Revision Date

Extraction Worksheet for FACT-M-3

Study #	Sample Number set #	PFOS approx. 0.5 ppm actual ppm #W_____	PFOS approx. 5 ppm actual ppm #W_____	PFOS approx. 50 ppm actual ppm #W_____	Date and Initials for Std. or Comments
-	H_2O Blank	-	-	-	
-	_____Serum Blank	-	-	-	
-					
-					
-					
-					
-					
-					
-					
[1]		-	-	-	
		-	-	-	
		-	-	-	
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		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

[1] Study number where the original worksheet is located.

Blank Serum Std # Serum amount = g

Serum Extraction Method : **Date & Initials**

Vortex 15 sec.

Pipette Serum Volume mL

Pipette 1 mL of 0.5 M TBA, pH 10. Std. #_____

Pipette 2 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ buffer Std. #_____

Pipette 5 mL of ethyl acetate TN-A-_____

Shake 20 min.

Centrifuge 20-25 min. Centrifuge speed:

Remove a 4 mL aliquot of organic layer

Put on Nitrogen Evaporator to dryness Evaporator #: Temperature:

Add methanol Volume_____mL TN-A-_____

Vortex 30 sec.

Filter using a 3cc B-D syringe with a 0.2µm SRI filter into a 1.5 mL autosample vial

MS/MSD/___ Cont. Checks: Spiked _____ uL of a _____ ppm std (_____) for a final concentration of _____ ppm. MS/MSD used sample _____. Cont. Checks used same serum as for std curve.

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICAL COMPOUNDS FROM SERUM FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-4.1 **Adoption Date:** 03/01/99

Revision Date: 4/27/99

Author: Lisa Clemen, Glenn Langenburg

Approved By:

Laboratory Manager	Date 4/27/99
Group Leader	Date 4/26/99
Technical Reviewer	Date 04/26/99

Exact Copy of Original
Initial RSV
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 **Scope:** This method is for the extraction of potassium perfluorooctanesulfonate (PFOS) or other fluorochemical compounds from serum.

1.2 **Applicable compounds:** Fluorochemical surfactants or other fluorinated compounds.

1.3 **Matrices:** Rabbit, rat, bovine, monkey, and human serum or other fluids as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the procedure for extracting potassium perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from serum, or other fluids, using an ion pairing reagent and methyl-*tert*-butyl ether (MtBE). In this method, seven fluorochemicals were extracted: PFOS, PFOSA, PFOSAA, EtFOSE-OH, PFOSEA, M556, and surrogate standard (see *3.0 **Definitions***). An ion pairing reagent is added to the sample and the analyte ion pair is partitioned into MtBE. The MtBE extract is removed and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 1.0 mL of methanol, then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

2.2 These sample extracts are analyzed following method ETS-8-5.1 or other appropriate methods.

3.0 Definitions

3.1 PFOS: perfluorooctanesulfonate (anion of potassium salt) $C_8F_{17}SO_3^-$

3.2 PFOSA: perfluorooctane sulfonylamide $C_8F_{17}SO_2NH_2$

3.3 PFOSAA: perfluorooctane sulfonylamido (ethyl)acetate $C_8F_{17}SO_2N(CH_2CH_3)CH_2CO_2^-$

3.4 EtFOSE-OH: 2(N-ethylperfluorooctane sulfonamido)-ethyl alcohol $C_8F_{17}SO_2N(CH_2CH_3)CH_2CH_2OH$

3.5 PFOSEA: perfluorooctane sulfonyl ethylamide $C_8F_{17}SO_2N(CH_2CH_3)H$

3.6 M556: $C_8F_{17}SO_2N(H)(CH_2COOH)$

3.7 Surrogate standard: 1H-1H-2H-2H perfluorooctane sulfonic acid

4.0 Warnings and Cautions

4.1 **Health and safety warnings**

4.1.1 Use universal precautions, especially laboratory coats, goggles, and gloves when handling animal tissue, which may contain pathogens.

5.0 Interferences

5.1 There are no interferences known at this time.

6.0 Equipment

6.1 The following equipment is used while performing this method. Equivalent equipment is acceptable.

6.1.1 Vortex mixer, VWR, Vortex Genie 2

6.1.2 Centrifuge, Mistral 1000 or IEC

6.1.3 Shaker, Eberbach or VWR

6.1.4 Nitrogen evaporator, Organomation

6.1.5 Balance (± 0.100 g)

7.0 Supplies and Materials

7.1 Gloves

7.2 Eppendorf or disposable pipettes

7.3 Nalgene bottles, capable of holding 250 mL and 1 L

7.4 Volumetric flasks, glass, type A

7.5 I-CHEM vials, glass, 40 mL glass

7.6 Centrifuge tubes, polypropylene, 15 mL

7.7 Labels

7.8 Oxford Dispenser – 3.0 to 10.0 mL

7.9 Syringes, capable of measuring 5 µL to 50 µL

7.10 Graduated pipettes

7.11 Syringes, disposable plastic, 3 cc

7.12 Syringe filters, nylon, 0.2 µm, 25 mm

7.13 Timer

7.14 Crimp cap autovials and caps

7.15 Crimpers

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 Reagents and Standards

8.1 Type I reagent grade water, Milli-Q™ or equivalent; all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus™ system

8.2 Sodium hydroxide (NaOH), J.T Baker or equivalent

8.3 Tetrabutylammonium hydrogen sulfate(TBA), Kodak or equivalent

8.4 Sodium carbonate (Na_2CO_3), J.T. Baker or equivalent

8.5 Sodium bicarbonate ($NaHCO_3$), J.T. Baker or equivalent

8.6 Methyl-T-Butyl Ether, Omnisolv, glass distilled or HPLC grade

8.7 Methanol, Omnisolv, glass distilled or HPLC grade

8.8 Serum or blood, frozen from supplier

8.9 **Fluorochemical standards**

8.9.1 PFOS (3M Specialty Chemical Division), molecular weight = 538

8.9.2 PFOSA (3M Specialty Chemical Division), molecular weight = 499

8.9.3 PFOSAA (3M Specialty Chemical Division), molecular weight = 585

8.9.4 EtFOSE-OH (3M Specialty Chemical Division), molecular weight = 570

8.9.5 PFOSEA (3M Specialty Chemical Division), molecular weight = 527

8.9.6 M556 (3M Specialty Chemical Division), molecular weight = 557

8.9.7 Surrogate standard: 4-H, perfluorooctane sulfonic acid (1-H,1-H, 2-H, 2-H $C_8F_{13}SO_3H$) molecular weight = 428

8.9.8 Other fluorochemicals, as appropriate

8.10 Reagent preparation

NOTE: When preparing larger volumes than listed in reagent, standard, or surrogate preparation, adjust accordingly.

8.10.1 10 N sodium hydroxide (NaOH): Weigh approximately 200 g NaOH. Pour into a 1000 mL beaker containing 500 mL Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L Nalgene bottle.

8.10.2 1 N sodium hydroxide (NaOH): Dilute 10 N NaOH 1:10. Measure 10 mL of 10 N NaOH solution into a 100 mL volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 mL Nalgene bottle.

8.10.3 0.5 M tetrabutylammonium hydrogen sulfate (TBA): Weigh approximately 169 g of TBA into a 1 L volumetric containing 500 mL Milli-Q™ water. Adjust to pH 10 using approximately 44 to 54 mL of 10 N NaOH (While adding the last mL of NaOH, add slowly because the pH changes abruptly). Dilute to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.10.3.1 TBA requires a check prior to each use to ensure pH = 10. Adjust as needed using 1 N NaOH solution.

8.10.4 0.25 M sodium carbonate/sodium bicarbonate buffer ($Na_2CO_3/NaHCO_3$): Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and bring to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.11 Standards preparation

8.11.1 Prepare PFOS standards for the standard curve.

8.11.2 Prepare other fluorochemical standards, as appropriate. Multicomponent fluorochemical standards are acceptable (for example, one working standard solution containing 1.00 ppm PFOS, 1.02 ppm PFOSA, 0.987 ppm PFOSAA, and 1.10 ppm EtFOSE-OH.)

8.11.3 Weigh approximately 100 mg of PFOS into a 100 mL volumetric flask and record the actual weight.

8.11.4 Bring to volume with methanol for a stock standard of approximately 1000 ppm (µg/mL).

8.11.5 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.11.6 Dilute working standard 1 with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.11.7 Dilute working standard 1 with methanol for a working standard 3 solution of approx. 0.50 ppm.

8.12 Surrogate stock standard preparation

8.12.1 Weigh approximately 50-60 mg of surrogate standard 1-H,1-H, 2-H, 2-H, $C_8F_{13}SO_3H$ into a 50 mL volumetric flask and record the actual weight.

8.12.2 Bring to volume with methanol for a surrogate stock of approximately 1000-1200 ppm.

8.12.3 Prepare a surrogate working standard. Transfer approximately 1 mL of surrogate stock to a 10 mL volumetric flask and bring to volume with methanol for a working standard of 100 ppm. Record the actual volume transferred.

9.0 SAMPLE HANDLING

9.1 All samples are received frozen and must be kept frozen until the extraction is performed.

9.2 Allow samples to thaw to room temperature prior to extraction.

10.0 QUALITY CONTROL

10.1 Solvent Blanks, Method blanks and matrix blanks

10.1.1 An aliquot of 1.0 mL methanol is used as a solvent blank.

10.1.2 Extract two 1.0 mL aliquots of Milli-Q™ water following this procedure and use as method blanks.

10.1.3 Extract two 1.0 mL aliquots of the serum following this procedure and use as matrix blanks. See **11.1.4**.

10.2 Matrix spikes

10.2.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.2.2 Prepare each spike using a sample chosen by the analyst, usually the control matrix received with each sample set.

10.2.3 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spikes may be included and may fall in the low-range of the initial calibration curve.

10.2.4 Prepare one matrix spike and matrix spike duplicate per 40 samples, with a minimum of 2 matrix spikes per batch.

10.3 Continuing calibration checks

10.3.1 Prepare continuing calibration check samples to ensure the accuracy of the initial calibration curve.

10.3.2 Prepare, at a minimum, one continuing check per group of 10 samples. For example, if a sample set = 34, four checks are prepared and extracted.

10.3.3 Prepare each continuing calibration check from the same matrix used to prepare the initial curve.

10.3.4 The expected concentrations will fall within the mid-range of the initial calibration curve. Additional spikes may be included that fall in the low-range of the initial calibration curve. This is necessary if the analyst must quantitate using only the low end of the calibration curve (for example, 5 ppb – 100 ppb, rather than
5 ppb – 1000 ppb).

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare matrix calibration standards

11.1.1 Transfer 1 mL of serum to a 15 mL centrifuge tube.

11.1.2 If most sample volumes are less than 1.0 mL, extract standards with matrix volumes equal to the sample volumes. Do not extract less than 0.50 mL of matrix. Record each sample volume on the extraction sheet.

11.1.3 While preparing a total of twenty aliquots in 15 mL centrifuge tubes, mix or shake between aliquots.

11.1.4 Two 1 mL aliquots, or other appropriate volume, serve as matrix blanks. Typically use the standard concentrations and spiking amounts listed in Table 1, at the end of this section, to spike, in duplicate, two standard curves, for a total of eighteen standards, two matrix blanks, and two method blanks.

11.1.5 Refer to validation report **ETS-8-4.0 & ETS-8-5.0-V-1,** which lists the working ranges and the Linear Calibration Range (LCR) for calibration curves.

11.1.6 Use Attachment D as an aid in calculating the concentrations of the working standards. See Section **13.0** to calculate actual concentrations of PFOS in calibration standards.

11.2 To each standard, blank, or continuing check, add appropriate amount of surrogate working standard for the concentration to fall within the calibration curve range 5 ppb - 1000 ppb.

11.3 Extract spiked matrix standards following **12.6-12.16** of this method. Use these standards to establish each initial curve on the mass spectrometer.

Table 1 Approximate spiking amounts for standards and spikes Using 1.0 mL of matrix		
Working standard (approx. conc.)	µL	Approx. final conc. of analyte in matrix
-	-	Blank
0.500 ppm	10	0.005 ppm
0.500 ppm	20	0.010 ppm
5.00 ppm	5	0.025 ppm
5.00 ppm	10	0.050 ppm
5.00 ppm	20	0.100 ppm
50.0 ppm	5	0.250 ppm
50.0 ppm	10	0.500 ppm
50.0 ppm	15	0.750 ppm
50.0 ppm	20	1.00 ppm

12.0 PROCEDURE

12.1 Obtain frozen samples and allow to thaw at room temperature or in a lukewarm waterbath.

12.2 Vortex mix for 15 seconds, then transfer 1.0 mL or other appropriate volume to a 15 mL polypropylene centrifuge tube.

12.3 Return unused samples to freezer after extraction amounts have been removed.

12.4 Record the initial volume on the extraction worksheet.

12.5 Label the tube with the study number, sample ID, date and analyst initials. See attached worksheet for documenting the remaining steps.

12.6 Spike all samples, including blanks and standards, ready for extraction with surrogate standard as described in **11.2.**

12.7 Spike each matrix with the appropriate amount of standard as described in **11.1,** or **Table 1** in that section, for the calibration curve standards. Also prepare matrix spikes and continuing calibration standards.

12.8 Vortex mix the standard curve samples, matrix spike samples, and continuing calibration samples for 15 seconds.

12.9 Check to ensure the 0.5 M TBA reagent is at pH 10. If not, adjust accordingly.

12.10 To each sample, add 1 mL 0.5 M TBA and 2 mL of 0.25M sodium carbonate/sodium bicarbonate buffer.

12.11 Using an Oxford Dispenser, add 5 mL methyl-*tert*-butyl ether.

12.12 Cap each sample and put on the shaker at a setting of 300 rpm, for 20 minutes.

12.13 Centrifuge for 20 to 25 minutes at a setting of 3500 rpm, or until layers are well separated.

12.14 Label a fresh 15 mL centrifuge tube with the same information as in **12.5**.

12.15 Remove 4.0 mL of the organic layer to this clean 15 mL centrifuge tube.

12.16 Put each sample on the analytical nitrogen evaporator until dry, approximately 1 to 2 hours.

12.17 Add 1.0 mL of methanol to each centrifuge tube using a graduated pipette.

12.18 Vortex mix for 30 seconds.

12.19 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 mL glass autovial or low-volume autovial when necessary.

12.20 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, and analyst(s) performing the extraction.

12.21 Cap and store extracts at room temperature or at approximately 4 °C until analysis.

12.22 Complete the extraction worksheet, attached to this document, and tape in the study notebook or include in study binder, as appropriate.

13.0 Data Analysis and Calculations

13.1 Calculations

13.1.1 Calculate actual concentrations of PFOS, or other applicable fluorochemical, in calibration standards using the following equation:

$$\frac{\text{mL of standard x concentration of standard (µg /mL)}}{\text{mL of standard + mL of surrogate standard + initial matrix volume (mL)}} =$$

Final Concentration (µg/mL) of PFOS in matrix

14.0 Method Performance

14.1 The method detection limit (MDL) is analyte and matrix specific. Refer to MDL report for specific MDL and limit of quantitation (LOQ) values (see **Attachments B and C**).

14.2 The following quality control samples are extracted with each batch of samples to evaluate the quality of the extraction and analysis.

14.2.1 Method blanks and matrix blanks.

14.2.2 Matrix spike and matrix spike duplicate samples to determine accuracy and precision of the extraction.

14.2.3 Continuing calibration check samples to determine the continued accuracy of the initial calibration curve.

14.3 Refer to section 14 of ETS-8-5.1 for method performance criteria.

15.0 Pollution Prevention and Waste Management

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

16.0 RECORDS

16.1 Complete the extraction worksheet attached to this method, and tape in the study notebook or include in the 3-ring study binder, as appropriate.

17.0 ATTACHMENTS

17.1 Attachment A, Extraction worksheet

17.2 Attachment B, MDL/LOQ values and summary

17.3 Attachment C, Calibration standard concentration worksheet

18.0 REFERENCES

18.1 The validation report associated with this method is **ETS-8-4.0 & 5.0-V-1.**

18.2 FACT-M-3.1, "Analysis of Serum or Other Fluid Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

19.0 AFFECTED DOCUMENTS

19.1 ETS-8-5.1, "Analysis of Serum or Other Fluid Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

20.0 REVISIONS

Revision Number	Reason For Revision	Revision Date
1	Section 12.21 Changed to include sample storage at room temperature. Section 12.13 Added the shaker speed. Section 12.17 Final volume is 1.0 mL; not adjusted for initial volumes less than 1.0 mL.	04/02/99

Extraction Worksheet ETS-8-4.1

Study #_____ Matrix_____ Box #_____ Wk/Day_____ DateSpiked/Analyst	Surrogate Std approx. ppm actual ppm #_____	FC-Mix approx. 0.5 pm actual ppm #_____	FC-Mix approx. 5 ppm actual ppm #_____	FC-Mix approx. 50 ppm actual ppm #_____	Comments
CCV					
MS					
MSD					
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
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		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

Blank	Std #	amount =	mL
Serum Extraction Method	:		**Date & Initials**
Vortex 15 sec.			
Pipette Matrix	Volume	mL	
Pipette 1 mL of 0.5 M TBA, pH 10. pH = _____	Std. # _____		
Pipette 2 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ buffer	Std. # _____		
Dispense 5 mL of methyl-t-butyl ether	TN-A- _____		
Shake 20 min.	Shaker speed:		
Centrifuge 20-25 min.	Centrifuge speed:		
Remove a 4 mL aliquot of organic layer			
Put on Nitrogen Evaporator to dryness	Temperature:		
Add methanol	Volume _____ mL	TN-A- _____	
Vortex 30 sec.			
Filter using a 3cc B-D syringe with a 0.2µm filter into a 1.5 mL autosample vial			

Cont. Cal. Verifications used same matrix as for std curve.

MDL/LOQ values for rabbit serum

Compound	MDL (ppb)	LOQ (ppb)	Linear Calibration Range (LCR) Approximate concentrations to be used for preparing the Standard Calibration Curve
PFOS	1.74	5.55	5 ppb - 1000 ppb
PFOSA	1.51	4.79	5 ppb - 1000 ppb
PFOSAA	3.46	20.5	5 ppb - 1000 ppb
EtFOSE-OH	11.4	36.2	5 ppb - 1000 ppb
M556	6.03	19.2	5 ppb - 1000 ppb
PFOSEA	5.71	18.2	5 ppb - 1000 ppb

MDL/LOQ values in rat, bovine, monkey, and human serum, and monkey plasma were not statistically determined. Two curves in each of these matrices were extracted and analyzed with the rabbit serum curves to determine equivalence. Responses in the rat, bovine, monkey, and human were equivalent to the rabbit responses, therefore, their MDL and LOQ will be the same values as determined in rabbit serum.

Please see LOQ Summary and MDL study in ETS-8-4.0 & 5.0-V-1 for further information.

Compound: PFOS

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.995 - 978	24.8 - 978	83-108	4.67-11.0
Low Curve	4.94 - 248	4.94 - 248	85-104	5.34-12.0
High curve	97.8 - 978	97.8 – 978	85-106	4.84-9.80
1/X	0.995 - 978	4.94 - 978	94-111	4.60-10.5

Compound: PFOSA

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.993 - 976	4.93 - 976	88-103	5.10-14.7
Low Curve	4.93 - 97.6	4.93 - 97.6	87-105	9.85-14.7
High curve	24.8 - 976	24.8 - 978	93-102	5.08-13.9
1/X	0.993 - 976	4.93 - 976	94-103	5.10-14.5

Compound: PFOSAA

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.991 - 974	24.7 - 974	81-111	4.18-10.6
Low Curve	4.92 - 247	9.74 - 247	97-107	6.38-21.8
High curve	49.2 - 974	97.4 - 974	85-108	4.33-12.5
1/X	0.991 - 974	9.74 - 974	95-115	4.11-23.2

Compound: EtFOSE-OH

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.993 - 976	49.3 - 976	77-110	11.2-25.5
Low Curve	4.93 – 97.6	9.76 – 97.6	97-107	14.1-21.3
High curve	49.3 - 976	97.6 - 976	90-109	11.5-19.6
1/X	0.993 - 493	9.76 - 976	86-111	11.1-21.2

Compound: PFOSEA

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.993 - 976	24.8 - 976	96-106	10.1-16.2
Low Curve	4.93 - 248	9.76 - 248	91-110	11.8-19.5
High curve	49.3 - 976	49.3 - 976	86-106	10.2-18.2
1/X	0.993 - 976	9.76 - 976	95-117	10.1-19.1

Compound: M556

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.993 - 976	24.8 - 976	88-106	4.82-17.9
Low Curve	4.93 - 97.6	9.76 – 97.6	100-105	5.95-18.2
High curve	97.6 - 976	97.6 - 976	81-111	5.11-9.74
1/X	0.993 - 976	9.76 - 976	97-110	4.77-19.5

Ion Pair Standard Curves – Fluids

Prep date(s):
Analyte(s):
Sample matrix:

Standard number:
Equipment number:
Final solvent and TN:
Blank fluid/identifier:

Method/revision:
Target analyte(s):
FC mix std approx. 0.500 ppm:
FC mix std approx. 5.00 ppm:
FC mix std approx. 50.0 ppm:
Surrogate std approx. 100 ppm:

Actual concentrations of standards in the FC mix

PFOS Std conc ug/mL	PFOSA Std conc ug/mL	PFOSAA Std conc ug/mL	EtFOSE Std conc ug/mL	PFOSEA Std conc ug/mL	M556 Std conc ug/mL	All Am't spiked mL	All Final vol mL
0.500	0.507	0.532	0.501	0.521	0.501	0.010	1.015
0.500	0.507	0.532	0.501	0.521	0.501	0.020	1.025
5.00	5.07	5.32	5.01	5.21	5.01	0.005	1.010
5.00	5.07	5.32	5.01	5.21	5.01	0.010	1.015
5.00	5.07	5.32	5.01	5.21	5.01	0.020	1.025
50.0	50.1	53.2	50.1	52.1	50.1	0.005	1.010
50.0	50.1	53.2	50.1	52.1	50.1	0.010	1.015
50.0	50.1	53.2	50.1	52.1	50.1	0.015	1.020
50.0	50.1	53.2	50.1	52.1	50.1	0.020	1.025

Calculated concentrations of standards in the sample matrix

PFOS Final conc ng/mL	PFOSA Final conc ng/mL	PFOSAA Final conc ng/mL	EtFOSE Final conc ng/mL	PFOSEA Final conc ng/mL	M556 Final conc ng/mL	Surrogate Std conc ng/mL	All Am't spiked mL
4.93	5.00	5.24	4.94	5.01	5.13	100	0.005
9.76	9.89	10.4	9.78	9.93	10.2		
24.8	25.1	26.3	24.8	25.2	25.8	**Surrogate**	
49.3	50.0	52.4	49.4	50.1	51.3	Final conc	
97.6	98.9	104	97.8	99.3	102	ng/mL	
248	251	263	248	252	258	500	
493	500	524	494	501	513		
735	746	782	737	749	766		
976	989	1038	978	993	1017		

Validated ranges – approximate concentrations

Serum	PFOS	PFOSA	PFOSAA	EtFOSE-OH	PFOSEA	M556
Rabbit	5.00-1000	5.00-1000	5.00-1000	5.00-1000	5.00-1000	5.00-1000
Bovine	Estimates only. Use values for rabbit.					
Rat	Estimates only. Use values for rabbit.					
Monkey & Plasma	Estimates only. Use values for rabbit.					
Human	Estimates only. Use values for rabbit.					

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICAL COMPOUNDS FROM LIVER FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-6.0 **Adoption Date:** 07/22/99

Revision Date: NA

Author: Lisa Clemen, Robert Wynne

Approved By:

Laboratory Manager — Date 7/22/99

Group Leader — Date 7/14/99

Technical Reviewer — Date 07/14/99

Exact Copy of Original
Initial RSD
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the extraction of potassium perfluorooctanesulfonate (PFOS) or other fluorochemical compounds from liver.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds.

1.3 Matrices: Rabbit, rat, bovine, and monkey livers or other tissues as designated in the validation report.

2.0 SUMMARY OF METHOD

2.1 This method describes the procedure for extracting potassium perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from liver, or other tissues, using an ion pairing reagent and methyl-*tert*-butyl ether (MtBE). In this method, seven fluorochemicals can be extracted: PFOS, PFOSA, PFOSAA, EtFOSE-OH, PFOSEA, M556, and surrogate standard. An ion pairing reagent is added to the sample and the analyte ion pair is partitioned into MtBE. The MtBE extract is transferred to a centrifuge tube and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 1.0 mL methanol then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

2.2 These sample extracts are analyzed following method ETS-8-7.0 or other appropriate methods.

3.0 DEFINITIONS

3.1 PFOS: perfluorooctanesulfonate (anion of potassium salt) $C_8F_{17}SO_3$

3.2 PFOSA: perfluorooctane sulfonylamide $C_8F_{17}SO_2NH_2$

3.3 PFOSAA: perfluorooctane sulfonylamido (ethyl)acetate $C_8F_{17}SO_2N(CH_2CH_3)CH_2CO_2$

3.4 EtFOSE-OH: 2(N-ethylperfluorooctane sulfonamido)-ethyl alcohol $C_8F_{17}SO_2N(CH_2CH_3)CH_2CH_2OH$

3.5 PFOSEA: perfluorooctane sulfonyl ethylamide $C_8F_{17}SO_2N(CH_2CH_3)H$

3.6 M556: $C_8F_{17}SO_2N(H)(CH_2COOH)$

3.7 Surrogate standard: 1H-1H-2H-2H perfluorooctane sulfonic acid

4.0 WARNINGS AND CAUTIONS

4.1 Health and Safety Warnings:

4.1.1 Use universal precautions, especially laboratory coats, goggles, and gloves when handling animal tissue, which may contain pathogens.

5.0 INTERFERENCES

5.1 There are no interferences known at this time.

6.0 EQUIPMENT

6.1 The following equipment is used while performing this method. Equivalent equipment is acceptable.

6.1.1 Ultra-Turrax T25 Grinder for grinding liver samples

6.1.2 Vortex mixer, VWR, Vortex Genie 2

6.1.3 Centrifuge, Mistral 1000 or IEC

6.1.4 Shaker, Eberbach or VWR

6.1.5 Nitrogen Evaporator, Organomation

6.1.6 Balance (sensitivity to 0.100 g)

7.0 Supplies and Materials

7.1 Gloves

7.2 Dissecting scalpels

7.3 Eppendorf or disposable pipettes

7.4 Nalgene bottles, capable of holding 250 mL and 1 L

7.5 Volumetric flasks, glass, type A

7.6 I-CHEM vials, 40 mL glass

7.7 Plastic sampule vials, Wheaton, 6 mL (or appropriate size)

7.8 Centrifuge tubes, polypropylene, 15 mL

7.9 Labels

7.10 Oxford Dispensor – 3.0 to 10.0 ml

7.11 Syringes, capable of measuring 5 µL to 50 µL

7.12 Graduated pipettes

7.13 Syringes, disposable plastic, 3 cc

7.14 Syringe filters, nylon, 0.2 µm, 25 mm

7.15 Timer

7.16 Crimp cap autovials and caps

7.17 Crimpers

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 Reagents and Standards

8.1 Type I reagent grade water, Milli-Q™ or equivalent; all water used in this method should be Milli-Q™ water and be provided by a Milli-Q TOC Plus™ system

8.2 Sodium hydroxide (NaOH), J.T Baker or equivalent

8.3 Tetrabutylammonium hydrogen sulfate(TBA), Kodak or equivalent

8.4 Sodium carbonate (Na_2CO_3), J.T. Baker or equivalent

8.5 Sodium bicarbonate ($NaHCO_3$), J.T. Baker or equivalent

8.6 Methyl-*tert*-butyl ether, Omnisolv, glass distilled or HPLC grade

8.7 Methanol, Omnisolv, glass distilled or HPLC grade

8.8 Liver, frozen from supplier

8.9 Dry ice from supplier

8.10 **Fluorochemical standards**

8.10.1 PFOS (3M Specialty Chemical Division), molecular weight = 538

8.10.2 PFOSA (3M Specialty Chemical Division), molecular weight = 499

8.10.3 PFOSAA (3M Specialty Chemical Division), molecular weight = 585

8.10.4 EtFOSE-OH (3M Specialty Chemical Division), molecular weight = 570

8.10.5 PFOSEA (3M Specialty Chemical Division), molecular weight = 527

8.10.6 M556 (3M Specialty Chemical Division), molecular weight = 557

8.10.7 Surrogate standard: 4-H, perfluorooctane sulfonic acid (1-H,1-H, 2-H, 2-H $C_8F_{13}SO_3H$) molecular weight = 428

8.10.8 Other fluorochemicals, as appropriate

8.11 Reagent preparation

NOTE: When preparing larger volumes than listed in reagent, standard, or surrogate preparation, adjust accordingly.

8.11.1 10 N sodium hydroxide (NaOH): Weigh approximately 200 g NaOH. Pour into a 1000 mL beaker containing 500 mL Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L Nalgene bottle.

8.11.2 1 N sodium hydroxide (NaOH): Dilute 10 N NaOH 1:10. Measure 10 mL of 10 N NaOH solution into a 100 mL volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 mL Nalgene bottle.

8.11.3 0.5 M tetrabutylammonium hydrogen sulfate (TBA): Weigh approximately 169 g of TBA into a 1 L volumetric containing 500 mL Milli-Q™ water. Adjust to pH 10 using approximately 44 to 54 mL of 10 N NaOH (While adding the last mL of NaOH, add slowly because the pH changes abruptly). Dilute to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.11.3.1 TBA requires a check prior to each use to ensure pH = 10. Adjust as needed using 1 N NaOH solution.

8.11.4 0.25 M sodium carbonate/sodium bicarbonate buffer ($Na_2CO_3/NaHCO_3$): Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and bring to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.12 Standards preparation

8.12.1 Prepare PFOS standards for the standard curve.

8.12.2 Prepare other fluorochemical standards, as appropriate. Multicomponent fluorochemical standards are acceptable (for example, one working standard solution containing 1.00 ppm PFOS, 1.02 ppm PFOSA, 0.987 ppm PFOSAA, and 1.10 ppm EtFOSE-OH.)

8.12.3 Weigh approximately 100 mg of PFOS into a 100 mL volumetric flask and record the actual weight.

8.12.4 Bring to volume with methanol for a stock standard of approximately 1000 ppm (µg/mL).

8.12.5 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.12.6 Dilute the stock solution with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.12.7 Dilute the stock solution with methanol for a working standard 3 solution of approx. 0.50 ppm.

8.13 Surrogate stock standard preparation

8.13.1 Weigh approximately 50-60 mg of surrogate standard 1-H,1-H, 2-H, 2-H, $C_8F_{13}SO_3H$ into a 50 ml volumetric flask and record the actual weight.

8.13.2 Bring to volume with methanol for a surrogate stock of approximately 1000-1200 ppm.

8.13.3 Prepare a surrogate working standard. Transfer approximately 1.0 ml of surrogate stock to a 10 ml volumetric flask and bring to volume with methanol for a working standard of 10-20 ppm. Record the actual volume transferred.

9.0 SAMPLE HANDLING

9.1 All samples are received frozen and must be kept frozen until the extraction is performed.

10.0 QUALITY CONTROL

10.1 Matrix blanks and method blanks

10.1.1 An aliquot of 1.0 mL methanol is used as a solvent blank.

10.1.2 Extract two 1.0 mL aliquots of Milli-Q™ water following this procedure and use as method blanks.

10.1.3 Extract two 1.0 mL aliquots of liver homogenate following this procedure and use as matrix blanks. Refer to **11.1.6**.

10.2 Matrix spikes

10.2.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.2.2 Prepare each spike using a sample chosen by the analyst, usually a control liver received with each sample set.

10.2.3 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spikes may be included and may fall in the low-range of the initial calibration curve.

10.2.4 Prepare one matrix spike and matrix spike duplicate per 40 samples, with a minimum of 2 matrix spikes per batch.

10.3 Continuing calibration verifications

10.3.1 Prepare continuing calibration verification samples to ensure the accuracy of the initial calibration curve.

10.3.2 Prepare, at a minimum, one continuing calibration verification sample per group of 10 samples. For example, if a sample set = 34, four verifications are prepared and extracted.

10.3.3 Prepare each continuing calibration verification from the same matrix used to prepare the initial curve.

10.3.4 The expected concentrations will fall within the mid-range of the initial calibration curve. Additional spikes may be included that fall in the low-range of the initial calibration curve. This is necessary if the analyst must quantitate using only the low end of the calibration curve (for example, 5 ppb – 100 ppb, rather than 5 ppb – 1000 ppb).

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare matrix calibration standards

11.1.1 Weigh approximately 40 g of liver into a 250 mL Nalgene bottle containing 200 mLs Milli-Q™ water. Grind to a homogeneous solution.

11.1.2 If 40 g is not available, use appropriate amounts of liver and water to ensure a 1:5 ratio.

11.1.3 Refer to **13.0** to calculate the actual density of liver homogenate and the concentration of solid liver tissue dispersed in 1.0 mL of homogenate solution.

11.1.5 Add 1 mL of homogenate to a 15 mL centrifuge tube. Re-suspend solution by shaking between aliquots while preparing a total of eighteen 1 mL aliquots of homogeneous solution in 15 mL centrifuge tubes.

11.1.6 Two 1 mL aliquots, or other appropriate volume, serve as matrix blanks.

11.1.7 Typically use the standard concentrations and spiking amounts listed in Table 1, at the end of this section, to spike, in duplicate, two standard curves, for a total of eighteen samples, two matrix blanks, and two method blanks.

11.1.8 Refer to validation reports **ETS-8-6.0 and ETS-8-7.0-V-1** or **Attachment B**, which lists the working ranges and the Linear Calibration Range (LCR) for calibration curves.

11.1.9 Use **Attachment C** as an aid in calculating the concentrations of the working standards. Refer to **13.0** to calculate actual concentrations of PFOS in calibration standards.

11.2 To each working standard, blank, or continuing verification, add appropriate amount of surrogate working standard for the concentration to fall within the calibration curve range 5 ppb – 1000ppb.

11.3 Extract spiked liver homogenates following **12.14-12.25** of this method. Use these standards to establish each initial curve on the mass spectrometer.

Table 1
Approximate Spiking Amounts for Calibration Standards

Working Standard (Approx. Conc.)	µl	Approx. final conc. of PFOS in liver
-	-	Blank
0.50 ppm	2	0.005 ppm
0.50 ppm	4	0.010 ppm
0.50 ppm	10	0.025 ppm
0.50 ppm	20	0.050 ppm
0.50 ppm	40	0.100 ppm
5.0 ppm	10	0.250 ppm
5.0 ppm	20	0.500 ppm
5.0 ppm	30	0.750 ppm
50 ppm	4	1.00 ppm

12.0 PROCEDURE

12.1 Obtain frozen liver samples.

12.2 Cut approximately 1 g of liver using a dissecting scalpel. This part of the procedure is best performed quickly, not allowing the liver to thaw.

12.3 Weigh the sample directly into a tared plastic sampule vial.

12.4 Record the liver weight in the study notebook.

12.5 Return unused liver portions to freezer.

12.6 Add 2.5 mLs of water to sampule vial.

12.7 Grind the sample. Put the grinder probe in the sample and grind for about 2 minutes, or until the sample is homogeneous.

12.8 Rinse the probe into the sample with 2.5 mLs water using a pipette.

12.9 Take the grinder apart and clean it with methanol after each sample. Refer to AMDT-EP-22.

12.10 Cap the sample and vortex for 15 seconds. Label the sampule vial with the study number, weight, liver ID, date and analyst initials.

12.11 Pipette 1.0 mL, or other appropriate volume, of homogenate into a 15 mL polypropylene centrifuge tube. Label the centrifuge tube with the identical information as the sampule vial. Refer to attached worksheet for documenting the remaining steps.

12.12 Pipette two 1 mL aliquots of Milli-Q™ water to centrifuge tubes. These will serve as method blanks.

12.13 Spike all samples, including blanks and standards ready for extraction with surrogate standard as described in section **11.2**.

12.14 Spike each matirx with the appropriate amount of standard as described in 11.1, or Table 1 of that section, for the calibration curve standards. Also prepare matrix spikes and continuing calibration standards.

12.15 Vortex mix the standard curve samples, matrix spike samples, and continuing calibration samples for 15 seconds.

12.16 Check to ensure 0.5 M TBA reagent is at pH 10. If not, adjust accordingly.

12.17 To each sample, add 1 mL 0.5 M TBA and 2 mL of the 0.25 M sodium carbonate/sodium bicarbonate buffer.

12.18 Using an Oxford Dispenser, add 5 mL methyl-*tert*-butyl ether.

12.19 Cap each sample and put on the shaker at a setting of 300 rpm, for 20 minutes.

12.20 Centrifuge for 20 to 25 minutes at a setting of 3500 rpm, or until layers are well separated.

12.21 Label a fresh 15 mL centrifuge tube with the same information as in **12.10**.

12.22 Remove 4.0 mL of the organic layer to the fresh 15 mL centrifuge tube.

12.23 Put each sample on the analytical nitrogen evaporator until dry, approximately 1 to 2 hours.

12.24 Add 1.0 mL to each centrifuge tube using a graduated pipette.

12.25 Vortex mix for 30 seconds.

12.26 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 mL glass autovial or low-volume autovial when necessary.

12.27 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, and analyst(s) performing the extraction.

12.28 Cap and store extracts at room temperature or at approximately 4 °C until analysis.

12.29 Complete the extraction worksheet, attached to this document, and tape in study notebook or include in study binder, as appropriate.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.1 Calculate the average density of the liver homogenate by recording each mass of ten separate 1.0 mL aliquots of homogenate.

$$\text{Average density (mg/mL)} = \frac{\text{Average mass (mg) of the aliquots}}{\text{1.0 mL aliquot}}$$

13.1.2 Calculate the amount of liver (mg) per 1.0 mL homogenate (or concentration of dispersed solid tissue per mL of homogenate suspension) using the following equation:

$$\frac{\text{g of Liver x Average density* of homogenate (mg/mL)}}{\text{(g of Liver + g of Water)}}$$

* refer to **13.1.1** for details.

13.1.3 Calculate actual concentrations of PFOS and other fluorochemicals in calibration standards using the following equation:

$$\frac{\text{µL of Standard x Concentration (µg /mL)}}{\text{mg Liver / 1 mL homogenate*}} = \text{Final Concentration (µg/g or mg/kg) of PFOS in Liver}$$

*refer to **13.1.2** for details.

14.0 METHOD PERFORMANCE

14.1 The method detection limit (MDL) is analyte and matrix specific. Refer to MDL report for specific MDL and limit of quantitation (LOQ) values (refer to **Attachments B and C**).

14.2 The following quality control samples are extracted with each batch of samples to evaluate the quality of the extraction and analysis.

14.2.1 Method blanks and matrix blanks.

14.2.2 Matrix spike and matrix spike duplicate samples to determine accuracy and precision of the extraction.

14.2.3 Continuing calibration verification samples to determine the continued accuracy of the initial calibration curve.

14.3 Refer to section **14** of **ETS-8-7.0** for method performance criteria.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

16.0 RECORDS

16.1 Complete the extraction worksheet attached to this method, and tape in the study notebook or include in the 3-ring study binder, as appropriate.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 Attachment A, Extraction worksheet

17.2 Attachment B, MDL/LOQ values and summary

17.3 Attachment C, Calibration standard calculation and concentration worksheet

18.0 REFERENCES

18.1 The validation report associated with this method is **ETS-8-6.0 & 7.0-V-1**.

18.2 AMDT-EP-22, "Routine Maintenance of Ultra-Turrax T-25"

18.3 FACT-M-1.1, "Extraction of PFOS or Other Anionic Fluorochemical Surfactants from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry"

19.0 AFFECTED DOCUMENTS

19.1 ETS-8-7.0, "Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

Study #_____ Matrix_____ Box #_____ Wk/Day_____ Date Spiked/Analyst	Surrogate Std approx. ppm actual ppm #_____	FC Mix Std approx. 0.5 ppm actual ppm #_____	FC Mix Std approx. 5 ppm actual ppm #_____	FC Mix Std approx. 50 ppm actual ppm #_____	Comments
CCV					
MS					
MSD					
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

Blank Liver Homogenate: Std # Liver amount = g

Liver Extraction Method :	**Date & Initials**
Spike surrogate and Standard mix, Vortex 15 sec.	
Pipette 1 mL of Liver Solution	
Pipette 1 mL of †0.5 M TBA, pH 10. pH = _____	Std. #_____
Pipette 2 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ Buffer	Std. #_____
Dispense 5ml of Methyl-t-Butyl Ether	TN-A-_____
Shake 20 min.	Shaker Speed
Centrifuge 20-25 min.	Centrifuge Speed
Remove a 4 mL aliquot of organic layer	
Put on Nitrogen Evaporator to dryness	Evaporator Temperature
Add 1.0 mL of Methanol	TN-A-_____
Vortex 30 sec.	
Filter using a 3cc B-D syringe with a 0.2µm SRI filter into autosample vial	

Cont. Cal. Verifications used the same matrix as for the standard curve.

MDL/LOQ values for rabbit liver

Compound	MDL (ppb)	LOQ (ppb)	Linear Calibration Range (LCR) Approximate concentrations to be used for preparing the Standard Calibration Curve
PFOS	8.45	26.9	30 ppb – 1200 ppb
PFOSA	3.50	11.1	12 ppb – 1200 ppb
PFOSAA	24.6	78.3	30 ppb – 1200 ppb
EtFOSE-OH	108	345	60 ppb – 900 ppb*
M556	82.3	262	60 ppb – 1200 ppb
PFOSEA	33.9	108	30 ppb- 1200 ppb

MDL/LOQ values in rat, bovine, and monkey liver were not statistically determined. Two curves in each of these matrices were extracted and analyzed with the rabbit liver curves to determine equivalence. Responses in the rat, bovine, and monkey liver curves were equivalent to the rabbit responses, therefore, their MDL and LOQ will be assumed to be equivalent to those values as determined for the rabbit liver.

Refer to LOQ Summary and MDL study in ETS-8-6.0 & 7.0-V-1 for further information
* EtFOSE-OH estimates only for MDL and LOQ. Did not meet criteria for validation.

Compound: PFOS

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.19 - 1237	12 - 1200	12 - 1200	6 - 300	12 - 300	60 - 1200	60 - 1200

Compound: PFOSA

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.19 - 1237	12 - 1200	12 - 1200	12 - 300	12 - 300	60 - 1200	60 - 1200

Compound: PFOSAA

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.16 - 1232	12 - 1200	30 - 1200	30 - 900	60 - 900	N/A	N/A

Compound: EtFOSE-OH

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.17 - 1235	31 - 900	31 - 900	N/A	N/A	N/A	N/A

Compound: PFOSEA

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.17 - 1235	31 - 1200	31 - 1200	N/A	N/A	N/A	N/A

Compound: M556

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.17 - 1235	31 - 1200	60 - 1200	N/A	N/A	N/A	N/A

Ion Pair Standard Curves – Tissue

Prep date(s):
Analyte(s):
Sample matrix:

Standard number:
Equipment number:
Final solvent and TN:
Blank liver/identifier:

Method/revision:
Target analyte(s):
FC mix std approx. 0.500 ppm:
FC mix std approx. 5.00 ppm:
FC mix std approx. 50.0 ppm:
Surrogate std approx. 100 ppm:

Actual concentrations of standards in the FC mix

PFOS Std conc ug/mL	PFOSA Std conc ug/mL	PFOSAA Std conc ug/mL	EtFOSE Std conc ug/mL	PFOSEA Std conc ug/mL	M556 Std conc ug/mL	Std conc ug/mL	All Am't spiked mL	All Density g
0.500	0.500	0.500	0.500	0.500	0.500		0.002	0.167
0.500	0.500	0.500	0.500	0.500	0.500		0.004	0.167
0.500	0.500	0.500	0.500	0.500	0.500		0.010	0.167
0.500	0.500	0.500	0.500	0.500	0.500		0.020	0.167
0.500	0.500	0.500	0.500	0.500	0.500		0.040	0.167
5.00	5.00	5.00	5.00	5.00	5.00		0.010	0.167
5.00	5.00	5.00	5.00	5.00	5.00		0.020	0.167
5.00	5.00	5.00	5.00	5.00	5.00		0.030	0.167
50.0	50.0	50.0	50.0	50.0	50.0		0.004	0.167

Calculated concentrations of standards in the sample matrix

PFOS Final conc ng/g	PFOSA Final conc ng/g	PFOSAA Final conc ng/g	EtFOSE Final conc ng/g	PFOSEA Final conc ng/g	M556 Final conc ng/g	Std conc ng/g	Surrogate Std conc ng/mL	All Am't spiked mL
5.99	5.99	5.99	5.99	5.99	5.99		100	0.005
12.0	12.0	12.0	12.0	12.0	12.0			
29.9	29.9	29.9	29.9	29.9	29.9		**Surrogate**	
59.9	59.9	59.9	59.9	59.9	59.9		Final conc	
120	120	120	120	120	120		ng/mL	
299	299	299	299	299	299		0.500	
599	599	599	599	599	599			
898	898	898	898	898	898			
1198	1198	1198	1198	1198	1198			

Validated ranges – approximate concentrations

Liver	PFOS	PFOSA	PFOSAA	EtFOSE-OH	POAA	PFOSEA
Rabbit	5-1000 ppb	5-1000 ppb	5-1000 ppb	5-1000 ppb	5-1000 ppb	5-1000 ppb
Bovine	Estimates only, use rabbit values.					
Rat	Estimates only, use rabbit values.					
Monkey	Estimates only, use rabbit values.					

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF FLUOROCHEMICALS IN LIVER EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: FACT-M-2.0 **Adoption Date:** 5/26/98

Revision Date: N/A

Author: Lisa Clemen

Approved By:

Laboratory Manager — Date: 5/26/98

Group Leader — Date: 5/26/98

Technical Reviewer — Date: 5/27/98

Initial RSV Date 4/5/01

Exact Copy of Original

1.0 SCOPE AND APPLICATION

1.1 **Scope:** This method is for the analysis of extracts of liver or other tissues for fluorochemical surfactants using HPLC-electrospray/mass spectrometry.

1.2 **Applicable Compounds:** Potassium perfluorooctanesulfonate, anionic fluorochemical surfactants, or other ionizable compounds.

1.3 **Matrices:** Rabbit, rat, bovine, and monkey livers or other livers as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the analysis of fluorochemical surfactants extracted from liver using HPLC-electrospray/mass spectrometry. The analysis is performed by monitoring a single ion characteristic of a particular fluorochemical, such as the potassium perfluorooctanesulfonate (PFOS) anion, M/Z= 499. Samples may also be screened to verify compound identification.

3.0 Definitions

3.1 None.

4.0 Warnings and Cautions

4.1 Health and Safety Warnings:

4.1.1 Use caution with the voltage cable for the probe. When the voltage cable is plugged into the probe DO NOT TOUCH THE PROBE, there is risk of electrical shock.

4.2 Cautions:

4.2.1 Do not run solvent pumps above capacity of 400 bar (5800 psi). If pressure goes over 400 bar, the HP1100 will initiate automatic shutdown.

4.2.2 Do not run solvent pumps to dryness.

5.0 Interferences

5.1 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 changed in order to optimize the system.

6.1.1 Micromass Electrospray Mass Spectrometer

6.1.2 HP1100 low pulse solvent pumping system and autosampler.

7.0 Supplies and Materials

7.1 Supplies

7.1.1 Nitrogen gas, refrigerated liquid, regulated to approximately 100 psi.

7.1.2 HPLC column, specifics to be determined by the analyst.

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, all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus system.

8.1.3 Ammonium acetate, HPLC grade or equivalent.

8.2 Standards

8.2.1 Typically one H_2O blank, one liver blank, and seven liver standards are prepared during the extraction procedure. See FACT-M-1.

9.0 SAMPLE HANDLING

9.1 Fresh liver standards are prepared with each analysis. Extracted standards and samples are stored in capped autovials or capped 15 mL centrifuge tubes until analysis.

9.2 If analysis will be delayed, extracted standards and samples may be refrigerated until analysis can be performed.

10.0 QUALITY CONTROL

10.1 Matrix Blanks and Method Blanks

10.1.1 Analyze a method blank and matrix blank prior to each calibration curve.

10.2 Matrix Spikes

10.2.1 Analyze a matrix spike and matrix spike duplicate with each analysis.

10.2.2 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spike concentrations may fall in the low-range of the initial calibration curve.

10.2.3 See section 13 to calculate percent recovery.

10.3 Continuing Calibration Checks

10.3.1 Analyze a mid-range calibration standard after every tenth sample. If a significant change (± 30%) in peak area occurs, relative to the initial standard curve, stop the run. Only those samples analyzed before the last acceptable calibration standard will be used. The remaining samples must be reanalyzed.

10.3.2 See section 13 to calculate percent difference.

10.4 System Suitability

10.4.1 System suitability (e.g. peak area, retention time and peak shape, etc.) will be assessed for each run.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Analyze the extracted liver standards prior to and following each set of extracts. The mean of two standard values, at each standard concentration, will be plotted by linear regression for the calibration curve using MassLynx or other suitable software.

11.2 The r^2 value for the data should be 0.98 or greater. Lower values may be acceptable at the discretion of the analyst.

11.3 If the curve does not meet requirements, perform routine maintenance or reextract the standard curve (if necessary) and reanalyze.

12.0 PROCEDURES

12.1 Acquisition Set up

12.1.1 Click on start button in the Acquisition Control Panel. Set up a sample list. Assign a filename using letter-MO-DAY-last digit of year-sample number, assign a method (MS) for acquiring, and type in sample descriptions.

12.1.2 To create a method click on scan button in the Acquisition control panel and select SIR. Set Ionization Mode as appropriate and mass to 499 or other appropriate masses. A scan is usually collected along with the SIRs. Save method.

12.1.3 Typically the sample list begins with the first set of liver standards and ends with the second set of standards.

12.1.4 Samples are analyzed with a continuing calibration check injected after every tenth sample. Solvent blanks should be analyzed periodically to monitor possible analyte carryover and are not considered samples but may be included as such.

12.2 Using the Autosampler

12.2.1 Set up sample tray according to the sample list prepared in section 12.1.1.

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

12.2.2.1 Sample size = 10 µL injection with a sample wash

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 15 minutes

12.2.2.4 Solvent ramp =

Time	MeOH	2.0 mM Ammonium acetate
0.00 min.	45%	55%
7.5 min.	90%	10%
11.0 min.	90%	10%
11.5 min.	45%	55%

Note: In this instrument configuration, the run must be set up on the electrospray software with a "Waiting for inlet start" message before the "Start" button is pressed on the HP Workstation.

12.2.2.5 Press the "Start" button.

12.3 Instrument Sep-up

12.3.1 Refer to AMDT-EP-31 for more details.

12.3.2 Check the solvent level in reservoirs and refill if necessary.

12.3.3 Check the stainless steel capillary at the end of the probe. Use an eye piece to check the tip. 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.

12.3.4 Set HPLC pump to "On". Set the flow to 10 - 500 uL/min or as appropriate. Observe droplets coming out of the tip of the probe. Allow to equilibrate for approximately 10 minutes.

12.3.5 Turn on the nitrogen. A fine mist should be expelled with no nitrogen leaking around the tip of the probe.

12.3.6 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:

12.3.6.1 Drying gas 250-400 liters/hour

12.3.6.2 ESI nebulizing gas 10-15 liters/hour

12.3.6.3 LC constant flow mode flow rate 10 – 500 uL/min

12.3.6.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the instrument is operating correctly.)

12.3.7 Carefully guide the probe into the opening. Insert probe until it will not go any further. Connect the voltage cables to the probe.

12.3.8 Record tune parameters in the instrument log.

12.3.9 Using the cross-flow counter electrode in the ES/MS source is recommended for the analysis of biological matrices.

12.3.10 Click on start button in the Acquisition Control Panel. Press the start button at top of sample list. Ensure start and end sample number includes 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 PFOS anion in total liver (mg):

$$\frac{\left(\dfrac{\text{ug PFOS anion calc. from std curve}}{\text{g of liver used for analysis}}\right)}{1000\ \text{ug} / 1\ \text{mg}} \times \text{Total mass of liver (g)}$$

14.0 METHOD PERFORMANCE

14.1 The method detection limit is equal to at least three times the baseline noise in the matrix blank.

14.2 The practical quantitation limit is equal to the lowest standard in the calibration curve.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

16.0 RECORDS

16.1 Store chromatograms in the study folder. Each chromatogram should have the following information included either in the header or hand written on the chromatogram: study number, sample name, extraction date, and dilution factor (if applicable).

16.2 Plot calibration curve by linear regression and store in the study folder.

16.3 Print sample list from MassLynx and tape into the instrument runlog.

16.4 Print data integration summary from MassLynx and tape into the instrument runlog.

16.5 Copy instrument runlog pages, including instrument parameters and sample results, and tape into appropriate study notebook.

16.6 Summarize data using suitable software and store in the study folder.

16.7 Back up electronic data to appropriate media. Record in study notebook the file name and location of backup electronic data.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 Attachment A: FACT-M-2 Data reporting spreadsheet

17.2 The validation report associated with this method is FACT-M-1.0 & 2.0-V-1.

18.0 REFERENCES

18.1 AMDT-EP-31, "Operation of VG Platform Electrospray Mass Spectrometer"

19.0 AFFECTED DOCUMENTS

19.1 FACT-M-1.0, "Extraction of Potassium Perfluorooctanesulfonate from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry"

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

Laboratory Study

Study:
Test Material:
Matrix/Final Solvent:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y Intercept:
Date of Extraction/Analyst:
Date of Analysis/Analyst:

Group Dose	Sample#	Concentration ug/mL	Initial Vol. mL	Dilution Factor	Final Conc. ug/mL

Slope: Taken from linear regression equation.
Group/Dose: Taken from the study folder.
Sample#: Taken from the study folder.
Concentration (ug/mL): Taken from the MassLynx integration summary.
Initial Volume (mL): Taken from the study folder.
Dilution Factor: Taken from the study folder.
Final Conc. (ug/mL): Calculated by dividing the initial volume from the concentration

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF FLUOROCHEMICALS IN SERUM EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: FACT-M-4.0 **Adoption Date:** 4/22/98

Revision Date: N/A

Author: Lisa Clemen

Approved By:

Laboratory Manager 4/22/98 Date

Group Leader 4/22/98 Date

Lisa Clemen
Technical Reviewer 4/14/98 Date

Exact Copy of Original
Initial RSD
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the analysis of extracts of serum or tissue for fluorochemical surfactants using HPLC-electrospray/mass spectrometry.

1.2 Applicable Compounds: Potassium perfluorooctanesulfonate, anionic fluorochemical surfactants, or other ionizable compounds.

1.3 Matrices: Rabbit, rat, and bovine serum or other sera as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the analysis of fluorochemical surfactants extracted from serum using HPLC-electrospray/mass spectrometry. The analysis is performed by monitoring a single ion characteristic of a particular fluorochemical, such as the potassium perfluorooctanesulfonate (PFOS) anion, M/Z= 499. Samples may also be screened to verify compound identification.

3.0 Definitions

3.1 None.

4.0 Warnings and Cautions

4.1 Health and Safety Warnings:

4.1.1 Use caution with the voltage cable for the probe. When the voltage cable is plugged into the probe DO NOT TOUCH THE PROBE, there is risk of electrical shock.

4.2 Cautions:

4.2.1 Do not run solvent pumps above capacity of 400 bar (5800 psi). If pressure goes over 400 bar, the HP1100 will initiate automatic shutdown.

4.2.2 Do not run solvent pumps to dryness.

5.0 Interferences

5.1 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 changed in order to optimize the system.

6.1.1 Micromass Electrospray Mass Spectrometer

6.1.2 HP1100 low pulse solvent pumping system and autosampler.

7.0 Supplies and Materials

7.1 Supplies

7.1.1 Nitrogen gas, refrigerated liquid, regulated to approximately 100 psi.

7.1.2 HPLC column, specifics to be determined by the analyst.

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, all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus system.

8.1.3 Ammonium acetate, HPLC grade or equivalent.

8.2 Standards

8.2.1 Typically one H_2O blank, one serum blank, and seven serum standards are prepared during the extraction procedure. See FACT-M-3.

9.0 Sample Handling

9.1 Fresh serum standards are prepared with each analysis. Extracted standards and samples are stored in capped autovials or capped 15 mL centrifuge tubes until analysis.

9.2 If analysis will be delayed, extracted standards and samples may be refrigerated at 4° C until analysis can be performed.

10.0 Quality Control

10.1 Matrix Blanks and Method Blanks

10.1.1 Analyze a method blank and a matrix blank prior to each calibration curve.

10.2 Matrix Spikes

10.2.1 Analyze a matrix spike and matrix spike duplicate with each analysis.

10.2.2 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spike concentrations may fall in the low-range of the initial calibration curve.

10.2.3 See section 13 to calculate percent recovery.

10.3 Continuing Calibration Checks

10.3.1 Analyze a mid-range calibration standard after every tenth sample. If a significant change (± 30%) in peak area occurs, relative to the initial standard curve, stop the run. Only those samples analyzed before the last acceptable calibration standard will be used. The remaining samples must be reanalyzed.

10.3.2 See section 13 to calculate percent difference.

10.4 System Suitability

10.4.1 System suitability (e.g., peak area, retention time, peak shape, etc.) will be assessed for each run.

11.0 Calibration and Standardization

11.1 Analyze the extracted serum standards prior to and following each set of extracts. The mean of two standard values, at each standard concentration, will be plotted by linear regression for the calibration curve using MassLynx or other suitable software.

11.2 The r^2 value for the data should be 0.98 or greater. Lower values may be acceptable at the discretion of the analyst.

11.3 If the curve does not meet requirements, perform routine maintenance or reextract the standard curve (if necessary) and reanalyze.

12.0 PROCEDURES

12.1 Acquisition Set up

12.1.1 Click on start button in the Acquisition Control Panel. Set up a sample list. Assign a filename using letter-MO-DAY-last digit of year-sample number, assign a method (MS) for acquiring, and type in sample descriptions.

12.1.2 To create a method click on scan button in the Acquisition control panel and select SIR (Single Ion Recording). Set Ionization Mode as appropriate and mass to 499 or other appropriate masses. A scan is usually collected along with the SIRs. Save method.

12.1.3 Typically the sample list begins with the first set of serum standards and ends with the second set of standards.

12.1.4 Samples are analyzed with a continuing calibration check injected after every tenth sample. Solvent blanks should be analyzed periodically to monitor possible analyte carryover and are not considered samples but may be included as such.

12.2 Using the Autosampler

12.2.1 Set up sample tray according to the sample list prepared in section 12.1.1.

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

12.2.2.1 Sample size = 10 µL injection with a sample wash

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 15 minutes

12.2.2.4 Solvent ramp =

Time	MeOH	2.0 mM Ammonium acetate
0.00 min.	45%	55%
7.5 min.	90%	10%
11.0 min.	90%	10%
11.5 min.	45%	55%

Note: In this instrument configuration, the run must be set up on the electrospray software with a "Waiting for inlet start" message before the "Start" button is pressed on the HP Workstation.

12.2.2.5 Press the "Start" button.

12.3 Instrument Set-up

12.3.1 Refer to AMDT-EP-31 for more details.

12.3.2 Check the solvent level in reservoirs and refill if necessary.

12.3.3 Check the stainless steel capillary at the end of the probe. Use an eye piece to check the tip. 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.

12.3.4 Set HPLC pump to "On". Set the flow to 10 - 500 uL/min or as appropriate. Observe droplets coming out of the tip of the probe. Allow to equilibrate for approximately 10 minutes.

12.3.5 Turn on the nitrogen. A fine mist should be expelled with no nitrogen leaking around the tip of the probe.

12.3.6 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:

12.3.6.1 Drying gas 250-400 liters/hour

12.3.6.2 ESI nebulizing gas 10-15 liters/hour

12.3.6.3 HPLC constant flow mode flow rate 10 – 500 µL/min

12.3.6.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the HPLC is operating correctly.)

12.3.7 Carefully guide the probe into the opening. Insert probe until it will not go any further. Connect the voltage cables to the probe.

12.3.8 Record tune parameters in the instrument log.

12.3.9 Using the cross-flow counter electrode in the ES/MS source is recommended for the analysis of biological matrices.

12.3.10 Click on start button in the Acquisition Control Panel. Press the start button at top of sample list. Ensure start and end sample number includes all samples to be analyzed.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.4 Calculate matrix spike percent recoveries using the following equation:

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

13.1.5 Calculate percent difference using the following equation:

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

13.1.6 Calculate actual concentration of PFOS, or other fluorochemical, anion in serum (µg/mL):

$$\frac{\text{µg of PFO calc. from std. Curve}}{\text{Initial Volume of serum (mL)}} \times \text{Dilution Factor} \times \text{Final Volume (mL)}$$

14.0 METHOD PERFORMANCE

14.1 The method detection limit is equal to half the lowest standard in the calibration curve.

14.2 The practical quantitation limit is equal to the lowest standard in the calibration curve.

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 Store chromatograms in the study folder. Each chromatogram must have the following information included either in the header or hand written on the chromatogram: study number, sample name, extraction date, and dilution factor (if applicable).

16.2 Plot calibration curve by linear regression and store in the study folder.

16.3 Print sample list from MassLynx and tape into the instrument runlog.

16.4 Print data integration summary from MassLynx and tape into the instrument runlog.

16.5 Copy instrument runlog pages, including instrument parameters and sample results, and tape into appropriate study notebook.

16.6 Summarize data using suitable software and store in the study folder.

16.7 Back up electronic data to appropriate medium. Record in study notebook the file name and location of backup electronic data.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 Attachment A: FACT-M-4 Data reporting spreadsheet

17.2 The validation report associated with this method is FACT-M-3.0 & 4.0-V-1.

18.0 REFERENCES

18.1 AMDT-EP-31, "Operation of VG Platform Electrospray Mass Spectrometer"

19.0 AFFECTED DOCUMENTS

19.1 FACT-M-3.0, "Extraction of Fluorochemical Anions from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry"

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

Laboratory Study

Study:
Test Material:
Matrix/Final Solvent:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y Intercept:
Date of Extraction/Analyst:
Date of Analysis/Analyst:

Group Dose	Sample#	Concentration ug/mL	Initial Vol. mL	Dilution Factor	Final Conc. ug/mL

Slope: Taken from linear regression equation.
Group/Dose: Taken from the study folder.
Sample#: Taken from the study folder.
Concentration (ug/mL): Taken from the MassLynx integration summary.
Initial Volume (mL): Taken from the study folder.
Dilution Factor: Taken from the study folder.
Final Conc. (ug/mL): Calculated by dividing the initial volume from the concentration

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICALS IN SERUM EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-5.1

Adoption Date: 03/01/99

Revision Date: 4/26/99

Author: Lisa Clemen, Robert Wynne

Approved By:

Laboratory Manager	Date 4/26/
Group Leader	Date 4/26/99
Technical Reviewer	Date 04/26/99

Exact Copy of Original
Initial RSD
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 **Scope:** This method describes the analysis of serum extracts for fluorochemical surfactants using HPLC-electrospray/mass spectrometry.

1.2 **Applicable Compounds:** Fluorochemical surfactants or other fluorinated compounds, or other ionizable compounds.

1.3 **Matrices:** Rabbit, rat, bovine, monkey, and human serum, or other fluids as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the analysis of fluorochemical surfactants extracted from serum or other fluids, using HPLC-electrospray/mass spectrometry, or similar system as appropriate. The analysis is performed by monitoring a single ion characteristic of a particular fluorochemical, such as the perfluorooctanesulfonate (PFOS) anion, m/z= 499. Additionally, samples may be analyzed using a tandem mass spectrometer to further verify the identity of a compound by detecting daughter ions of the parent ion.

3.0 Definitions

3.1 Atmospheric Pressure Ionization (API): The Micromass Quattro II triple quadrupole systems allow for various methods of ionization by utilizing various sources, probes, and interfaces. These include but are not limited to: Electrospray Ionization (ESI), Atmospheric Pressure chemical Ionization (APcI), Thermospray, etc. The ionization process in these techniques 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 charged droplets are produced by the application of a strong electrical field.

3.3 Mass Spectrometry, Mass Spectrometer (MS), Tandem Mass Spectrometer (MS/MS): The API Quattro II triple quadrupole systems are equipped with quadrupole mass selective detectors. Ions are selectively discriminated by mass to charge ratio (m/z) and subsequently detected. A single MS may be employed for ion detection or a series (MS/MS) for more specific fragmentation information.

3.4 Conventional vs. Z-spray probe interface: The latest models of Micromass Quattro II triple quadrupole systems (post 1998) utilize a "Z-spray" conformation. The spray emitted from a 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.)

3.5 Mass Lynx Software: System software designed for the specific operation of these Quattro II triple quadrupole systems. Currently MassLynx has Windows 95 and WindowsNT 4.0 versions. All versions are similar. For more details see the manual specific to the instrument (Micromass Quattro II triple quadrupole MassLynx or MassLynx NT 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 gloves, eyewear, and clothing.

4.2 Cautions:

4.2.1 Do not operate solvent pumps above capacity of 400 bar (5800 psi) back pressure. If the back pressure 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 Quattro II triple quadrupole 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 (House air system)

7.1.2 HPLC analytical column, specifics to be determined by the analyst and documented in the raw data.

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, 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

8.2 Standards

8.2.1 Typically two method blanks, two matrix blanks, and eighteen matrix standards are prepared during the extraction procedure. See **ETS-8-4.1**.

9.0 SAMPLE HANDLING

9.1 Fresh matrix standards are prepared with each analysis. Extracted standards and samples are stored in capped autovials or capped 15 mL centrifuge tubes until analysis.

9.2 If analysis will be delayed, extracted standards and samples can be refrigerated at approximately 4° C, or at room temperature, until analysis 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 batch 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 and analyzed to determine the matrix effect on the recovery efficiency.

10.2.2 Matrix spike duplicates are prepared and analyzed to measure the precision and the recovery for each analyte.

10.2.3 Analyze a matrix spike and matrix spike duplicate per forty samples, with a minimum of 2 spikes per batch.

10.2.4 Matrix spike and matrix spike duplicate concentrations will fall in the mid-range of the initial calibration curve. Additional spike concentrations may fall in the low-range of the initial calibration curve.

10.3 Continuing Calibration Verifications

10.3.1 Continuing calibration verifications 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 batch.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Analyze the extracted matrix standards prior to and following each set of extracts. The average of two standard curves will be plotted by linear regression ($y = my + b$), weighted $1/x$, not forced through zero, using MassLynx or other suitable software.

11.2 If the curve does not meet requirements, perform routine maintenance or reextract the 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 of the standard curve. 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 Click on start button in the Acquisition Control Panel. Set up a sample list. Assign a filename using MO-DAY-last digit of year-sample number, assign a method (MS) for acquiring, and type in sample descriptions.

12.1.2 To create a method click on scan button in the Acquisition control panel and select SIR (Single Ion Recording) or MRM. Set Ionization Mode as appropriate and mass to 499 or other appropriate masses. A full scan is usually collected along with the SIRs. Save acquisition method. If MS/MS instruments are employed, additional product ion fragmentation information may be collected. See Micromass MassLynx GUIDE TO DATA ACQUISITION for additional information and MRM (Multiple Reaction Monitoring).

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

12.1.4 Samples are analyzed with a continuing calibration check injected after every tenth sample. Solvent blanks should be analyzed periodically to monitor possible analyte carryover and are not considered samples but may be included as such.

12.2 Using the Autosampler

12.2.1 Set up sample tray according to the sample list prepared in Section **12.1.1**.

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

12.2.2.1 Sample size = 10 µL injection

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 13.5 minutes

12.2.2.4 Solvent ramp =

Time	MeOH	2.0 mM Ammonium acetate
0.00 min.	40%	60%
8.50 min.	90%	10%
11.0 min.	90%	10%
12.0 min.	40%	60%

12.2.2.5 Press the "Start" button.

12.3 Instrument Set-up

12.3.1 Refer to **ETS-9-24.0** for more details.

12.3.2 Check the solvent level in reservoirs and refill if necessary.

12.3.3 Check the stainless steel capillary at the end of the probe. Use an eyepiece to check the tip. 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.

12.3.4 Set HPLC pump to "On". Set the flow to 10 - 500 uL/min or as appropriate. Observe droplets coming out of the tip of the probe. Allow to equilibrate for approximately 10 minutes.

12.3.5 Turn on the nitrogen. A fine mist should be expelled with no nitrogen leaking around the tip of the probe. Readjust the tip of the probe if no mist is observed.

12.3.6 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:

12.3.6.1 Drying gas 250-400 liters/hour

12.3.6.2 ESI nebulizing gas 10-15 liters/hour

12.3.6.3 HPLC constant flow mode, flow rate 10 – 500 µL/min

12.3.6.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the HPLC is operating correctly.)

12.3.7 Carefully guide the probe into the opening. Insert probe until it will not go any further. Connect the voltage cables to the probe.

12.3.8 Print the tune page, with its parameters, and store it in the study binder with a copy taped into the instrument log.

12.3.9 Using the cross-flow counter electrode in the ES/MS source is recommended for the analysis of biological matrices.

12.3.10 Click on start button in the Acquisition Control Panel (this may vary among MassLynx versions, see appropriate MassLynx USER'S GUIDE). Press the start button. Ensure start and end sample number includes all samples to be analyzed.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.4 Calculate matrix spike percent recoveries using the following equation:

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

13.1.5 Calculate percent difference using the following equation:

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

13.1.6 Calculate actual concentration of PFOS, or other fluorochemical, in matrix (µg/mL):

$$\frac{\text{(ng of PFOS calc. from std. Curve x Dilution Factor)}}{\text{(Initial Volume of matrix (mL) + mL of Surrogate Standard)}} \times \frac{1\ \mu g}{1000\ ng}$$

Final Volume (mL)

14.0 METHOD PERFORMANCE

14.1 Method Detection Limit (MDL) and Limit of Quantitation (LOQ) are method, analyte, and matrix specific. Please see **ETS-8-4.1, Attachment B,** for a listing of current validated MDL and LOQ values.

14.2 Solvent Blanks, Method Blanks, and Matrix Blanks

14.2.1 Solvent blanks, method blanks, and matrix blanks values are must be below the lowest standard in the calibration curve

14.3 Calibration Curves

14.3.1 The r^2 value for the calibration curve must be 0.980 or better.

14.4 Matrix Spikes

14.4.1 Matrix spike percent recoveries are must be within ± 30% of the spiked concentration.

14.5 Continuing Calibration Verifications

14.5.1 Continuing calibration verification percent recoveries must be ± 30% of the spiked concentration.

14.6 If criteria listed in this method performance section isn't met, maintenance may be performed on the system and samples reanalyzed or other actions as determined by the analyst. Document all actions in the appropriate logbook.

14.7 If data are 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 to include in the appropriate study folder. Copy these pages and tape into the instrument runlog.

16.3 Plot the calibration curve by linear regression, weighted 1/x, 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 (Excel 5.0) and store in the study folder, see **Attachment A** for an example of a summary spreadsheet.

16.6 Back up electronic data to appropriate medium. Record in study notebook the file name and location of backup electronic data.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 Attachment A: ETS-8-5.1 Data summary spreadsheet.

18.0 References

18.1 FACT-M-4.1, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry

18.2 ETS-9-24.0, "Operation and Maintenance of the Micromass Atmospheric Pressure Ionization/Mass Spectrometer Quattro II triple quadrupole Systems"

18.3 The validation report associated with this method is **ETS-8-4.0 & 5.0-V-1**.

19.0 Affected Documents

19.1 ETS-8-4.1, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry"

20.0 Revisions

Revision Number.	Reason For Revision	Revision Date
1	Section 6.1.2 Clarification of HP1100 system components. Section 11.1 Average of two curves, not standard values, are used for plotting linear regression and added the 1/x weighting of the curve. Section 12.2.2.4 Clarification of solvent ramp. Section 17.1 Changed from attachment B to A.	04/02/99

Laboratory Study

Study:
Test Material:
Matrix/Final Solvent:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y Intercept:
Date of Extraction/Analyst:
Date of Analysis/Analyst:

Group Dose	Sample#	Concentration ug/mL	Initial Vol. mL	Dilution Factor	Final Conc. ug/mL

Slope: Taken from linear regression equation.
Group/Dose: Taken from the study folder.
Sample#: Taken from the study folder.
Concentration (ug/mL): Taken from the MassLynx integration summary.
Initial Volume (mL): Taken from the study folder.
Dilution Factor: Taken from the study folder.
Final Conc. (ug/mL): Calculated by dividing the initial volume from the concentration

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICALS IN LIVER EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-7.0 **Adoption Date:** 07/22/99

Revision Date: NA

Author: Lisa Clemen, Glenn Langenburg

Approved By:

Laboratory Manager 7/22/99 Date

Group Leader 7/14/99 Date

Technical Reviewer 07/14/99 Date

Exact Copy of Original
Initial RSD
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the analysis of liver extracts for fluorochemical surfactants using HPLC-electrospray/mass spectrometry.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds, or other ionizable compounds.

1.3 Matrices: Rabbit, rat, bovine, monkey liver, or other tissues as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the analysis of fluorochemical surfactants extracted from liver using HPLC-electrospray/mass spectrometry, or similar system as appropriate. The analysis is performed by monitoring a single ion characteristic of a particular fluorochemical, such as the perfluorooctanesulfonate (PFOS) anion, m/z = 499. Additionally, samples may be analyzed using a tandem mass spectrometer to further verify the identity of a compound by detecting daughter ions of the selected parent ion.

3.0 Definitions

3.1 **Atmospheric Pressure Ionization (API):** The Micromass Quattro II triple quadrupole systems allow for various methods of ionization by utilizing various sources, probes, and interfaces. These include but are not limited to: Electrospray Ionization (ESI), Atmospheric Pressure chemical Ionization (APcI), Thermospray, etc. The ionization process in these techniques 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 charged droplets are produced by the application of a strong electrical field.

3.3 **Mass Spectrometry, Mass Spectrometer (MS), Tandem Mass Spectrometer (MS/MS):** The API Quattro II triple quadrupole mass spectrometer is equipped with two quadrupole mass selective detectors and a collision cell. Ions are selectively discriminated by mass to charge ratio (m/z) and subsequently detected. A single MS may be employed for ion detection or an ion may be selected in the first quadrupole, fragmented in the collision cell, and these fragments may be analyzed in the second quadrupole.

3.4 **Conventional vs. Z-spray probe interface:** The latest models of Micromass Quattro II triple quadrupole (post 1998) utilize a "Z-spray" conformation. The spray emitted from a 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 other Z-spray systems, etc.)

3.5 **Mass Lynx Software:** System software designed for the specific operation of these Quattro II triple quadrupole systems. Currently MassLynx has Windows 95 and WindowsNT 4.0 versions. All versions are similar. For more details refer to the manual specific to the instrument (Micromass Quattro II triple quadrupole MassLynx or MassLynx NT 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 gloves, eyewear, and clothing.

4.2 Cautions:

4.2.1 Operate the solvent pumps below a back pressure of 400 bar (5800 psi). If the back pressure 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 shall 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 Quattro II triple quadrupole 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 air regulated to approximately 100 psi (house air system)

7.1.2 HPLC analytical column, specifics to be determined by the analyst and documented in the raw data

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 ATSM type I, or equivalent, and 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 mL volumetric container containing 2000 mL Milli-Q™ water, mix until all solids are dissolved. Store at room temperature.

8.2 Standards

8.2.1 Typically two method blanks, two matrix blanks, and eighteen matrix standards are prepared during the extraction procedure. Refer to **ETS-8-6.0**.

9.0 Sample Handling

9.1 Fresh matrix standards are prepared with each analysis. Extracted standards and samples are stored in capped autovials or capped 15 ml centrifuge tubes until analysis.

9.2 If analysis will be delayed, extracted standards and samples may be stored at room temperature, or refrigerated at approximately 4° C, until analysis can be performed.

10.0 Quality Control

10.1 Method Blanks and Matrix Blanks

10.1.1 Solvent blanks, method blanks, and matrix blanks are prepared and analyzed with each batch 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 and analyzed to determine the matrix effect on the recovery efficiency.

10.2.2 Matrix spike duplicates are prepared and analyzed to measure the precision and the recovery for each analyte.

10.2.3 Analyze a matrix spike and matrix spike duplicate per forty samples. With a minimum of 2 spikes per batch.

10.2.4 Matrix spike and matrix spike duplicate concentrations will fall in the mid-range of the initial calibration curve. Additional spike concentrations may fall in the low-range of the initial calibration curve.

10.3 Continuing Calibration Checks

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

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

11.0 Calibration and Standardization

11.1 Analyze the extracted matrix standards prior to and following each set of sample extracts. The average of two standard curves will be plotted by linear regression ($y = mx + b$), weighted 1/x, not forced through the origin, using MassLynx or other suitable software.

11.2 If the curve does not meet requirements perform routine maintenance or reextract the 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 of the standard curve. 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 MO-DAY-last digit of year-increasing letter of the alphabet starting with a

12.1.1.2 Assign a method (MS file) for acquiring

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 (Single Ion Recording) or MRM (Multiple Reaction Monitoring). Set Ionization Mode as appropriate and mass to 499 or other appropriate masses. A full scan is usually collected along with the SIRs. Save acquisition method. If MS/MS instruments are employed, additional product ion fragmentation information may be collected. Refer to Micromass MassLynx GUIDE TO DATA ACQUISITION for additional information and MRM.

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

12.1.4 Samples are analyzed with a continuing calibration verification injected standard after every tenth sample. Solvent blanks should be analyzed periodically to monitor possible analyte carryover and are not considered samples but may be included as such.

12.2 Using the Autosampler

12.2.1 Set up sample tray according to the sample list prepared in Section **12.1.1**.

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

12.2.2.1 Sample size = 10 µL injection

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 9 minutes

12.2.2.4 Solvent ramp conditions

Time	MeOH	2.0 mM Ammonium acetate
0.00 min.	40%	60%
1.0 min.	40%	60%
4.5 min.	95%	5%
6.5 min.	95%	5%
7.0 min.	40%	60%
9.0 mi.	40%	60%

12.2.2.5 Press the "Start" button.

12.3 Instrument Set-up

12.3.1 Refer to **ETS-9-24.0,** "Operation and Maintenance of the Micromass Quattro II Triple Quadrupole Mass Spectrometer Fitted with an Atmospheric Pressure Ionization Source," for more details.

12.3.2 Check the solvent level in reservoirs and refill if necessary.

12.3.3 Check the stainless steel capillary at the end of the probe. Use an eyepiece to check the tip. 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.

12.3.4 Turn on the nitrogen.

12.3.5 Open the tune page. Clicks on operate to initiate source block and desolvation heaters.

12.3.6 Open the Inlet Editor.

12.3.6.1 Set HPLC pump to "On"

12.3.6.2 Set the flow to 10 - 500 uL/min or as appropriate

12.3.6.3 Observe droplets coming out of the tip of the probe. A fine mist should be expelled with no nitrogen leaking around the tip of the probe. Readjust the tip of the probe if no mist is observed

12.3.6.4 Allow to equilibrate for approximately 10 minutes.

12.3.7 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:

12.3.7.1 Drying gas 250-400 liters/hour

12.3.7.2 ESI nebulizing gas 10-15 liters/hour

12.3.7.3 HPLC constant flow mode flow rate 10 – 500 µL/min

12.3.7.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the HPLC is operating correctly.)

12.3.7.5 Source block temperature 150°

12.3.7.6 Desolvation temperature 250°

12.3.8 Print the tune page, with its parameters, and store it in the study binder with a copy taped into the instrument log.

12.3.9 Click on start button in the Acquisition Control Panel (this may vary among MassLynx versions, refer to appropriate MassLynx User's Guide). Ensure start and end sample number includes all samples to be analyzed.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.4 Calculate matrix spike percent recoveries using the following equation:

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

13.1.5 Calculate percent difference using the following equation:

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

13.1.6 Calculate actual concentrations in matrix (µg/g):

$$\frac{\text{(ng of PFOS calc. from std. Curve x Dilution Factor)}}{\frac{\text{(Initial Weight of Liver (g)}}{\text{Final Volume (mL)}}} \times \frac{1\ \mu g}{1000\ ng}$$

14.0 METHOD PERFORMANCE

14.1 Method Detection Limit (MDL) and Limit of Quantitation (LOQ) are method, analyte, and matrix specific. Refer to **ETS-8-6.0, Attachment B** for a listing of current validated MDL and LOQ values.

14.2 Solvent Blanks, Method Blanks and Matrix Blanks

14.2.1 Solvent blanks, method blanks, and matrix blanks must be below the lowest standard in the calibration curve.

14.3 Calibration Curves

14.3.1 The r^2 value for the calibration must be 0.980 or better.

14.4 Matrix Spikes

14.4.1 Matrix spike percent recoveries must be within ± 30% of the spiked concentration.

14.5 Continuing Calibration Verification

14.5.1 Continuing calibration verification percent recoveries must be within ± 30% of the spiked concentration.

14.6 If criteria listed in the 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 appropriate logbook.

14.7 If data are 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 to include in the appropriate study folder. Copy these pages and tape into the instrument runlog.

16.3 Plot the calibration curve by linear regression, weighted 1/x, 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 (Excel 5.0+) and store in the study folder, refer to **Attachment A** for an example of a summary spreadsheet.

16.6 Back up electronic data to appropriate medium. Record in study notebook the file name and location of backup electronic data.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 Attachment A: ETS-8-7.0 Data summary spreadsheet

18.0 References

18.1 FACT-M-2.1, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry"

18.2 ETS-9-24.0, "Operation and Maintenance of the Micromass Atmospheric Pressure Ionization/Mass Spectrometer Quattro II triple quadrupole Systems"

18.3 The validation report associated with this method is **ETS-8-6.0 & 7.0-V-1**

19.0 Affected Documents

19.1 ETS-8-6.0, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Liver or Fluid for Analysis Using HPLC-Electrospray/Mass Spectrometry"

20.0 REVISIONS

Revision Number	Reason For Revision	Revision Date

Laboratory Study

Study:
Test Material:
Matrix/Final Solvent:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y Intercept:
Date of Extraction/Analyst:
Date of Analysis/Analyst:

Group Dose	Sample#	Concentration ng/g	Initial Wt. g	Dilution Factor	Final Conc. ug/g

Slope: Taken from linear regression equation.
Group/Dose: Taken from the study folder.
Sample#: Taken from the study folder.
Concentration (ng/g): Taken from the MassLynx integration summary.
Initial Wt. (g): Taken from the study folder.
Dilution Factor: Taken from the study folder.
Final Conc. (ug/g): Calculated by dividing the initial volume from the concentration

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Appendix D: Data Summary Tables

Table 10. FACT TOX-001 Data Summary of PFOS Concentration—Serum (µg/mL)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male	0.0405 ± 0.0215 (n = 5)	1.86 ± 0.252 (n = 5)	21.6 ± 1.99 (n = 5)	60.4 ± 8.76 (n = 5)	229 ± 42.5 (n = 5)	
	Female	0.117 ± 0.00828 (n = 5)	3.26 ± 0.415 (n = 5)	29.9 ± 3.84 (n = 5)	70.7 ± 16.8 (n = 5)	357 ± 65.2 (n = 5)	
Week 8[a]	Male	0.0518 ± 0.0174 (n = 5)				348 ± 41.5 (n = 5)	
	Female	0.181 ± 0.0174 (n = 5)				576 ± 40.1 (n = 5)	
Week 14[a]	Male	0.0779 ± 0.00791 (n = 5)	6.14 ± 0.688 (n = 5)	59.1 ± 11.3 (n = 5)	192 ± 24.6 (n = 5)		
	Female	0.196 ± 0.0361 (n = 5)	12.2 ± 1.83 (n = 5)	104 ± 25.4 (n = 5)	268 ± 34.0 (n = 5)		
Week 27	Male	0.0415 ± 0.00951 (n = 10)	2.75 ± 0.906 (n =10)	20.3 ± 2.85 (n = 10)	77.2 ± 15.2 (n = 10)		
	Female	0.0793 ± 0.0172 (n = 10)	4.85 ± 0.954 (n = 10)	39.7 ± 7.39 (n = 10)	170 ± 38.3 (n = 10)		
Week 53	Male	0.0191 ± 0.0134 (n = 15)	1.86 ± 1.128 (n = 10)	18.0 ± 7.18 (n = 10)	82.4 ± 66.0 (n = 15)		
	Female	0.0755 ± 0.0376 (n = 15)	4.67 ± 1.63 (n = 10)	33.6 ± 11.4 (n = 10)	163 ± 87.2 (n = 15)		
Week 105	Male	0.00936 ± 0.0123 (n = 14)	1.18 ± 1.41 (n = 22)	22.0 ± 17.6 (n = 22)	84.6 ± 45.4 (n = 17)		0.227 ± 0.377 (n = 10)
	Female	0.0271 ± 0.0237 (n = 13)	3.75 ± 2.65 (n = 11)*	55.2 ± 27.4 (n = 15)	122 ± 70.5 (n = 19)		2.41 ± 2.32 (n = 12)

[a] Not corrected for purity of the standard material.
*Animal C91216F sample lost during extraction.
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. The average fortified sample recovery was 96% with a standard deviation of 25%.

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Table 11. FACT TOX-001 Data Summary of PFOSA Concentration—Serum (µg/mL)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male	<LOQ[b] (n = 5)	0.0310 ± 0.00549 (n = 5)	0.698 ± 0.0818 (n = 5)	1.81 ± 0.520 (n = 5)	1.19 ± 0.147 (n = 5)	
	Female	<LOQ[b] (n = 5)	0.0622 ± 0.00874 (n = 5)	1.27 ± 0.404 (n = 5)	2.48 ± 0.344 (n = 5)	3.04 ± 2.00 (n = 5)	
Week 8[a]	Male	<LOQ[c] (n = 5)				1.10 ± 0.288 (n = 5)	
	Female	<LOQ[c] (n = 5)				1.08 ± 0.381 (n = 5)	
Week 14[a]	Male	<LOQ[e] (n = 5)	0.0598 ± 0.0110 (n = 5)	0.494 ± 0.141 (n = 5)	0.778 ± 0.0605 (n = 5)		
	Female	<LOQ[e] (n = 5)	0.110 ± 0.0280 (n = 5)	0.769 ± 0.141 (n = 5)	1.88 ± 0.503 (n = 5)		
Week 27[a]	Male	<LOQ[d] (n = 10)	0.0177 ± 0.0116 (n = 10)	0.226 ± 0.0368 (n = 10)	0.418 ± 0.0903 (n = 10)		
	Female	<LOQ[d] (n = 10)	0.0337 ± 0.00608 (n = 10)	0.508 ± 0.101 (n = 10)	0.875 ± 0.336 (n = 10)		
Week 53[a]	Male	<LOQ[d] (n = 15)	<LOQ[d] (n = 10)	0.237 ± 0.0751 (n = 10)	0.357 ± 0.138 (n = 15)		
	Female	<LOQ[d] (n = 15)	<LOQ[d] (n = 10)	0.366 ± 0.0824 (n = 10)	1.10 ± 0.536 (n = 15)		
Week 105[a]	Male	<LOQ[d] (n = 14)	0.0187 ± 0.0119 (n = 22)	0.306 ± 0.116 -(n = 22)	0.701 ± 0.363 (n = 17)		<LOQ[d] (n = 10)
	Female	<LOQ[d] (n = 13)	0.0846 ± 0.104 (n = 11)*	0.429 ± 0.133 (n = 15)	1.30 ± 0.512 (n = 19)		<LOQ[d] (n = 12)

[a] *Not corrected for purity of the standard material.*
[b] LOQ—Limit of Quantitation = 0.00249µg/mL
[c] LOQ—Limit of Quantitation = 0.00978µg/mL
[d] LOQ—Limit of Quantitation = 0.00493 µg/mL
[e] LOQ—Limit of Quantitation=0.0978 µg/mL
*Animal C91216F sample lost during extraction.

NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.

NOTE: The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. The average fortified sample recovery was 100% with a standard deviation of 16%.

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Table 12. FACT TOX-001 Data Summary of PFOSAA Concentration—Serum (µg/mL)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male	0.0511 ± 0.0291 (n = 5)	2.60 ± 1.63 (n = 5)	13.2 ± 2.30 (n = 5)	26.9 ± 9.28 (n = 5)	61.2 ± 14.2 (n = 5)	
	Female	0.0604 ± 0.0152 (n = 5)	3.07 ± 1.45 (n = 5)	23.1 ± 8.95 (n = 5)	33.8 ± 13.6 (n = 5)	92.5 ± 28.3 (n = 5)	
Week 8[a]	Male	0.0169 ± 0.00974 (n = 5)				67.0 ± 12.0 (n = 5)	
	Female	0.0433 ± 0.00973 (n = 5)				80.9 ± 15.3 (n = 5)	
Week 14[a]	Male	0.0360 ± 0.0145 (n = 5)	1.23 ± 0.506 (n = 5)	13.8 ± 6.63 (n = 5)	20.8 ± 4.38 (n = 5)		
	Female	0.0363 ± 0.00761 (n = 5)	1.87 ± 0.659 (n = 5)	16.4 ± 8.26 (n = 5)	33.8 ± 5.71 (n = 5)		
Week 27[a]	Male	<LOQ[b] (n = 10)	0.577 ± 0.455 (n = 10)	2.27 ± 0.848 (n = 10)	6.58 ± 2.06 (n = 10)		
	Female	<LOQ[b] (n = 10)	0.772 ± 0.311 (n = 10)	3.46 ± 1.03 (n = 10)	8.04 ± 2.13 (n = 10)		
Week 53[a]	Male	<LOQ[d] (n = 15)	0.597 ± 0.497 (n = 10)	4.80 ± 1.72 (n = 10)	9.0 ± 6.23 (n = 15)		
	Female	<LOQ[d] (n = 15)	0.556 ± 0.199 (n = 10)	4.22 ± 1.14 (n = 10)	12.8 ± 6.59 (n = 15)		
Week 105[a]	Male	<LOQ[c] (n = 14)	0.553 ± 0.345 (n = 22)	4.14 ± 1.92 (n = 22)	10.7 ± 7.93 (n = 17)		<LOQ[b] (n = 10)
	Female	<LOQ[c] (n = 13)	1.51 ± 1.42 (n = 11)*	6.14 ± 2.10 (n = 15)	11.9 ± 3.02 (n = 19)		<LOQ[b] (n = 12)

[a] Not corrected for purity of the standard material.
[b] LOQ—Limit of Quantitation = 0.0248 µg/mL
[c] LOQ—Limit of Quantitation = 0.00493 µg/mL
[d] LOQ—Limit of Quantitation = 0.0097
*Animal C91216F sample was lost during extraction.
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. The average fortified sample recovery was 109% with a standard deviation of 23%.

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Table 13. FACT TOX-001 Data Summary of EtFOSE-OH Concentration—Serum (µg/mL)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male						
	Female						
Week 8[a]	Male						
	Female						
Week 14[a]	Male						
	Female						
Week 27[a]	Male	<LOQ[b] (n = 10)	<LOQ[c] (n = 10)	<LOQ[c] (n = 10)	<LOQ[c] (n = 10)		
	Female	<LOQ[b] (n = 10)	<LOQ[c] (n = 10)	<LOQ[c] (n = 10)	<LOQ[c] (n = 10)		
Week 53[a]	Male	<LOQ[b] (n = 15)	<LOQ[d] (n = 10)	<LOQ[b] (n = 10)	0.0215 ± 0.00992 (n = 15)		
	Female	<LOQ[d] (n = 15)	<LOQ[b] (n = 10)	0.0258 ± 0.00252 (n = 10)	0.0231± 0.00963 (n = 15)		
Week 105[a]	Male	<LOQ[b] (n = 14)	<LOQ[b] (n = 22)	0.00912 ± 0.00430 (n = 22)	0.0182 ± 0.0170 (n = 17)		<LOQ[b] (n = 10)
	Female	<LOQ[b] (n = 13)	<LOQ[b] (n = 11)*	0.0108 ± 0.00522 (n = 15)	0.0364 ± 0.0145 (n = 19)		<LOQ[b] (n = 12)

[a] Not corrected for purity of the standard material.
[b]LOQ—Limit of Quantitation = 0.00977 µg/mL
[c]LOQ—Limit of Quantitation = 0.0248 µg/mL
[d]LOQ—Limit of Quantitation = 0.00493 ug/mL
*Animal C91216F sample was lost during extraction.
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The results of quality control analyses (curve fit, CCVs, and MS/MSDs) for EtFOSE-OH were inconsistent and indicate that data presented should be considered to be qualitative only. Values are presented in this data table in the spirit of full disclosure, but should not be used in any quantitative assessment of the data.

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Table 14. FACT TOX-001 Data Summary of M556 Concentration—Serum (µg/mL)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male						
	Female						
Week 8[a]	Male						
	Female						
Week 14[a]	Male						
	Female						
Week 27[a]	Male	<LOQ[b] (n = 10)	0.267 ± 0.0634 (n = 10)	2.03 ± 0.449 (n = 10)	6.61 ± 1.30 (n = 10)		
	Female	<LOQ[b] (n = 10)	0.303 ± 0.0744 (n = 10)	3.96 ± 0.796 (n = 10)	6.36 ± 2.11 (n = 10)		
Week 53[a]	Male	<LOQ[c] (n = 15)	0.371 ± 0.110 (n = 10)	4.00 ± 0.970 (n = 10)	10.4 ± 5.48 (n = 15)		
	Female	<LOQ[c] (n = 15)	0.439 ± 0.134 (n = 10)	4.10 ± 0.771 (n = 10)	12.6 ± 6.98 (n = 15)		
Week 105[a]	Male	<LOQ[c] (n = 14)	0.145 ± 0.115 (n = 22)	2.86 ± 1.191 (n = 22)	6.58 ± 2.71 (n = 17)		<LOQ[c] (n = 10)
	Female	<LOQ[c] (n = 13)	0.680 ± 0.667 (n = 11)*	3.02 ± 1.08 (n = 15)	5.06 ± 1.39 (n = 19)		<LOQ[c] (n = 12)

[a] Not corrected for purity of the standard material.
[b] LOQ—Limit of Quantitation = 0.0248 µg/mL
[c] LOQ—Limit of Quantitation = 0.00494 µg/mL
*Animal C91216F sample lost during extraction.
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. The average fortified sample recovery was 123% with a standard deviation of 20%.

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Table 15. FACT TOX-001 Data Summary of PFOSEA Concentration—Serum (µg/mL)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male						
	Female						
Week 8[a]	Male						
	Female						
Week 14[a]	Male						
	Female						
Week 27[a]	Male	<LOQ[b] (n = 10)	<LOQ[b,c] (n = 10)	<LOQ[d] (n = 10)	<LOQ[d] (n = 10)		
	Female	<LOQ[b] (n = 10)	<LOQ[c] (n = 10)	<LOQ[d] (n = 10)	<LOQ[d] (n = 10)		
Week 53[a]	Male	<LOQ[c] (n = 15)	<LOQ[c] (n = 10)	<LOQ[e] (n = 10)	<LOQ[b,c] (n = 15)		
	Female	<LOQ[c] (n = 15)	<LOQ[e] (n = 10)	<LOQ[b] (n = 10)	<LOQ[b,c] (n = 15)		
Week 105[a]	Male	<LOQ[c] (n = 14)	<LOQ[c] (n= 22)	<LOQ[c] (n = 22)	<LOQ[c] (n = 17)		<LOQ[c] (n = 10)
	Female	<LOQ[c] (n = 13)	<LOQ[c] (n = 11)*	<LOQ[c] (n = 15)	<LOQ[c] (n = 19)		<LOQ[c] (n = 12)

[a] Not corrected for purity of the standard material.
[b]LOQ—Limit of Quantitation = 0.0247 µg/mL
[c]LOQ—Limit of Quantitation = 0.00492 µg/mL
[d]LOQ—Limit of Quantitation = 0.00975 µg/mL
[e]LOQ—Limit of Quantitation = 0.0492 µg/mL
*Animal C91216F sample lost during extraction.
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The results of quality control analyses (curve fit, CCVs, and MS/MSDs) for PFOSEA were inconsistent and indicate that data presented for this analyte should be considered to be qualitative only. Values are presented here in the spirit of full disclosure, but should not be used in any quantitative assessment of the data.

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Table 16. FACT TOX-001 Data Summary of PFOS Concentration—Liver (µg/g)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male	0.774 ± 0.138 (n = 5)	12.4 ± 1.67 (n = 5)	96.1 ± 20.4 (n = 5)	199 ± 53.2 (n = 5)	499 ± 208 (n = 5)	
	Female	0.368 ± 0.144 (n = 5)	11.3 ± 1.94 (n = 5)	120 ± 25.3 (n = 5)	229 ± 121 (n = 5)	597 ± 180 (n = 5)	
Week 8[a]	Male	2.13 ± 0.668 (n = 5)				1067 ± 197 (n = 5)	
	Female	0.549 ± 0.328 (n = 5)				1065 ± 213 (n = 5)	
Week 14	Male	1.23 ± 0.262 (n = 5)	23.4 ± 2.14 (n = 5)	80.8 ± 50.9 (n = 5)	392 ± 94.1 (n = 5)		
	Female	0.402 ± 0.129 (n = 5)	23.6 ± 3.48 (n = 5)	141 ± 56.0 (n = 5)	335 ± 178 (n = 5)		
Week 27	Male						
	Female						
Week 53	Male	0.571 ± 0.175 (n = 5)			695 ± 189 (n = 5)		
	Female	0.390 ± 0.0993 (n = 5)			694 ± 123 (n = 5)		
Week 105	Male	0.143 ± 0.133 (n = 18)	7.56 ± 7.10 (n = 22)	90.1 ± 70.3 (n = 22)	313 ± 220 (n = 17)		2.05 ± 3.36 (n = 10)
	Female	0.178 ± 0.111 (n = 14)	38.5 ± 21.9 (n = 13)	127 ± 68.5 (n = 15)	297 ± 171 (n = 19)		7.69 ± 7.62 (n = 12)

[a] Not corrected for purity of the standard material.

NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.

NOTE: The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. The average fortified sample recovery was 110% with a standard deviation of 29% (revised results).

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Table 17. FACT TOX-001 Data Summary of PFOSA Concentration—Liver (µg/g)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male	0.0150 ± 0.00746 (n = 5)	0.562 ± 0.212 (n =5)	6.82 ± 1.08 (n = 5)	9.57 ± 2.33 (n = 5)	8.94 ± 5.05 (n = 5)	
	Female	0.0184 ± 0.00804 (n = 5)	0.681 ± 0.0572 (n = 5)	6.14 ± 1.08 (n = 5)	8.25 ± 2.19 (n = 5)	15.3 ± 2.31 (n = 5)	
Week 8[a]	Male	0.0313 ± 0.00163 (n = 5)				19.2 ± 0.987[+] (n = 5)	
	Female	0.0310 ± 0.00189 (n = 5)				15.5 ± 1.65[+] (n = 5)	
Week 14[a]	Male	0.0284 ± 0.0202 (n = 4)*	1.11 ± 0.194 (n = 5)	8.25 ± 2.00 (n = 5)	16.5 ± 1.20 (n = 5)		
	Female	0.0156 ± 0.00784 (n = 5)	1.80 ± 0.0831 (n = 5)	7.98 ± 1.03 (n = 5)	14.6 ± 1.52 (n = 5)		
Week 27[a]	Male						
	Female						
Week 53[a]	Male	0.0182 ± 0.0100 (n = 5)			28.2 ± 8.44 (n = 5)		
	Female	0.0139 ± 0.00677 (n = 5)			25.3 ± 4.37 (n = 5)		
Week 105[a]	Male	<LOQ[b] (n = 18)	0.744 ± 0.294 (n = 21)	7.44 ± 2.17 (n = 22)	13.5 ± 4.23 (n = 17)		<LOQ[c] (n = 10)
	Female	<LOQ[b] (n = 14)	1.82 ± 1.72 (n = 13)	6.77 ± 1.72 (n = 15)	15.6 ± 3.61 (n = 19)		<LOQ[c] (n = 12)

[a] Not corrected for purity of the standard material.
[b]LOQ—Limit of Quantitation = 0.0123 µg/g
[c]LOQ—Limit of Quantitation = 0.00614 µg/g
*Animal C90748M sample lost during extraction.
[+]CCVs failed, data entered as estimated.
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. The average fortified sample recovery was 102% with a standard deviation of 17% (revised results).

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Table 18. FACT TOX-001 Data Summary of PFOSAA Concentration—Liver (µg/g)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male	0.134 ± 0.0617 (n = 5)	3.63 ± 3.42 (n = 5)	59.6 ± 22.6 (n = 5)	53.1 ± 21.9 (n = 5)	85.7 ± 20.4 (n = 5)	
	Female	0.0995 ± 0.0390 (n = 5)	4.09 ± 2.16 (n = 5)	61.8 ± 34.1 (n = 5)	52.5 ± 14.9 (n = 5)	80.1 ± 50.9 (n = 5)	
Week 8[a]	Male	0.0980 ± 0.0351 (n = 5)				483 ± 72.7 (n = 5)	
	Female	0.0920 ± 0.0561 (n = 5)				330 ± 67.2 (n = 5)	
Week 14[a]	Male	0.145 ± 0.0611 (n = 4)*	1.85 ± 0.351 (n = 5)	20.0 ± 14.6 (n = 5)	81.1 ± 24.1 (n = 5)		
	Female	0.106 ± 0.0699 (n = 5)	2.66 ± 0.462 (n = 5)	14.9 ± 8.95 (n = 5)	59.2 ± 14.6 (n = 5)		
Week 27[a]	Male						
	Female						
Week 53[a]	Male	<LOQ[b] (n = 5)			55.7 ± 21.3 (n = 5)		
	Female	<LOQ[b] (n = 5)			47.6 ± 5.67 (n = 5)		
Week 105[a]	Male	0.0461 ± 0.0174 (n = 18)	1.86 ± 2.00 (n = 21)	11.4 ± 6.95 (n = 22)	23.6 ± 13.3 (n = 17)		0.0522 ± 0.0614 (n = 10)
	Female	0.0354 ± 0.0162 (n = 14)	3.49 ± 4.47 (n = 13)	7.98 ± 3.48 (n = 15)	26.9 ± 7.08 (n = 19)		<LOQ[c] (n = 12)

[a] Not corrected for purity of the standard material.
[b]LOQ—Limit of Quantitation = 0.0307 µg/g
[c]LOQ—Limit of Quantitation = 0.0123 µg/g
*Animal C90748M lost during extraction.
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. The average fortified sample recovery was 105% with a standard deviation of 19% (revised results).

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Table 19. FACT TOX-001 Data Summary of EtFOSE-OH Concentration—Liver (µg/g)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4[a]	Male	<LOQ[b] (n = 5)	<LOQ[c] (n = 5)	0.0870 ± 0.0351 (n = 5)	0.464 ± 0.154 (n = 5)	0.704 ± 0.335 (n = 5)	
	Female	<LOQ[b] (n = 5)	<LOQ[c] (n = 5)	0.0442 ± 0.00682 (n = 5)	0.375 ± 0.213 (n = 5)	0.762 ± 0.443 (n = 5)	
Week 8[a]	Male	0.0316 ± 0.00402 (n = 5)				0.338 ± 0.463 (n = 5)	
	Female	<LOQ[d] (n = 5)				0.352 ± 0.359 (n = 5)	
Week 14[a]	Male	<LOQ[b] (n = 5)	NA	0.206 ± 0.0677 (n = 5)	NA		
	Female	<LOQ[b] (n = 5)	NA	0.148 ± 0.0224 (n = 5)	NA		
Week 27[a]	Male						
	Female						
Week 53[a]	Male	NR			NR		
	Female	NR			NR		
Week 105[a]	Male	0.0796 ± 0.0287 (n = 18)	0.0649 ± 0.0130 (n = 22)	0.0883 ± 0.0415 (n = 22)	0.395 ± 0.229 (n = 17)		<LOQ[e] (n = 10)
	Female	0.0906 ± 0.0394 (n = 14)	0.0766 ± 0.0210 (n = 13)	0.103 ± 0.0276 (n = 15)	0.994 ± 1.30 (n = 19)		<LOQ[e] (n = 12)

[a] Not corrected for purity of the standard material.
[b] LOQ—Limit of Quantitation = 0.0596 µg/g
[c] LOQ—Limit of Quantitation = 0.119 µg/g
[d] LOQ—Limit of Quantitation = 0.0298 µg/g
[e] LOQ—Limit of Quantitation = 0.0614 µg/g
NA—Not applicable
NR—Not reported
*Animal C91216F sample lost during extraction.
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The results of quality control analyses (curve fit, CCVs, and MS/MSDs) for EtFOSE-OH were inconsistent and indicate that data presented for this analyte should be considered to be qualitative only. Values are presented here in the spirit of full disclosure, but should not be used in any quantitative assessment of the data.

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Table 20. FACT TOX-001 Data Summary of M556 Concentration—Liver (µg/g)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4	Male						
	Female						
Week 8	Male						
	Female						
Week 14	Male						
	Female						
Week 27	Male						
	Female						
Week 53[a]	Male	<LOQ[b] (n = 5)			58.9 ± 17.0 (n = 5)		
	Female	<LOQ[b] (n = 5)			36.3 ± 9.51 (n = 5)		
Week 105[a]	Male	<LOQ[b,e] (n = 18)	0.450 ± 0.378 (n = 22)	9.51 ± 3.29 (n = 22)	27.1 ± 9.41 (n = 17)		<LOQ[c,d] (n = 10)
	Female	<LOQ[b,f] (n = 14)	2.03 ± 2.74 (n = 13)	8.57 ± 3.33 (n = 15)	28.5 ± 8.86 (n = 19)		<LOQ[c,d] (n = 12)

[a] Not corrected for purity of the standard material.
[b] LOQ—Limit of Quantitation = 0.0308 µg/g
[c] LOQ—Limit of Quantitation = 0.00615 µg/g
[d] LOQ—Limit of Quantitation = 0.123 µg/g
[e] LOQ—Limit of Quantitation = 0.0123 µg/g
[f] LOQ—Limit of Quantitation = 0.0615 µg/g

NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.

NOTE: The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered accurate to within one standard deviation of the average fortified samples recovery. The average fortified sample recovery was 102% with a standard deviation of 15%.

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Table 21. FACT TOX-001 Data Summary of PFOSEA Concentration—Liver (µg/g)

Timepoint	Sex	Group 1 Control Average ±SD	Group 2 Low Average ±SD	Group 3 Mid Average ±SD	Group 4 Mid-High Average ±SD	Group 5 High Average ±SD	Group 6 Mid-High Recovery Average ± SD
Week 4	Male						
	Female						
Week 8	Male						
	Female						
Week 14	Male						
	Female						
Week 27	Male						
	Female						
Week 53	Male	NR			NR		
	Female	NR			NR		
Week 105[a]	Male	<LOQ[b,c] (n = 18)	<LOQ[b,c] (n = 22)	<LOQ[b] (n = 22)	0.0408 ± 0.0566 (n = 17)		<LOQ[b] (n = 10)
	Female	<LOQ[b,c] (n = 14)	<LOQ[b,c] (n = 13)	<LOQ[b] (n = 15)	0.0241 ± 0.0139 (n = 19)		<LOQ[b] (n = 12)

[a] Not corrected for purity of the standard material
[b] LOQ—Limit of Quantitation = 0.0307 µg/g
[c] LOQ—Limit of Quantitation = 0.0613 µg/g
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: The results of quality control analyses (curve fit, CCVs, and MS/MSDs) for PFOSEA were inconsistent and indicate that data presented for this analyte should be considered to be qualitative only. Values are presented here in the spirit of full disclosure, but should not be used in any quantitative assessment of the data.

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Appendix E: Data Spreadsheets

			PFOS	PFOSA	PFOSAA
Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		04/27/98 Lot 215	04/27/98 Lot L-2353	04/27/98 Lot 617
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		04/28/98 Lot 215	04/30/98 Lot L-2353	05/05/98 Lot 617
Matrix:	Rat Serum		05/05/98 Lot 215		
Method/Revision:	FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1	Filenames			
Analytical Equipment System Number:	Chick 080697 and Madeline 041098		**PFOS**	**PFOSA**	**PFOSAA**	**Dilutions**
Instrument Software/Version:	MassLynx 2.3 & 3.1	**Blks**	R0427802-03 & 59-60	R0427802-03, R0430803-04	R0427802-03 & 59-60	1/1, 11, 1/1
Filename:	See listing to the right	**Grp 1**	R0427813-24	R0427813-24	R0427813-24	1/1, 1/1, 1/1
R-Squared Value:	See Attachments	**Grp 2**	R0505812-23	R0430814-25	R0505812-23	1/50, 1/10, 1/50
Slope:	See Attachments	**Grp 3**	R0505826-37	R0430828-39	R0505826-37	1/250, 1/50, 1/250
Y-Intercept:	See Attachments	**Grp 4**	R0505840-51	R0430842-53	R0505840-51	1/500, 1/100, 1/500
Dates of Extraction/Analyst:	04/23/98, 04/28/98 RWW	**Grp 5**	R0428813-24	R0427841-47, 52-53	R0427827-38	1/1000, 1/5, 1/500
Dates of Analysis/Analyst:	04/27/98, 04/28/98, 04/30/98, 05/05/98 KJH/HOJ			R0427815O-51		1/500
Date of Data Reduction/Analyst:	09/01/00, 12/01/00, 12/04/00, 12/05/00 MMH/KJH	4/23/1998 **MS, MSD**	R0427856-57	R0427856-57	R0427856-57	1/1, 1/1, 1/1
Sample Data		4/28/1998 **MS, MSD**	R0505852-53	R0430856-57	R0505852-53	1/1, 1/1, 1/1

WEEK 4 RAT SERA REWORK

Group Dose	Sample #	Extraction Vol. mL	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
				Lot 215							Lot L-2353						
Method Blk	H2O Blk-1	1	0.9275	Unknown	1	0.00	R0427802	<LOQ (0.0231 ug/mL)			Unknown	1	0.00	R0427802	<LOQ (0.00249 ug/mL)		
	H2O Blk-2	1	0.9275	Unknown	1	0.680	R0427859	<LOQ (0.0231 ug/mL)	<LOQ	NA	Unknown	1	0.00	R0430803	<LOQ	<LOQ	NA
Matrix Blk	Rat Serum Blk-1	1	0.9275	Unknown	1	0.00	R0427803	<LOQ (0.0231 ug/mL)			Unknown	1	0.00	R0427803	<LOQ (0.00249 ug/mL)		
	Rat Serum Blk-2	1	0.9275	Unknown	1	2.48	R0427860	<LOQ (0.0231 ug/mL)	<LOQ	NA	Unknown	1	0.00	R0430804	<LOQ	<LOQ	NA
QC-250 ppb	MS 4/23/98	1	NA	NA	1	240	R0427856	96%			NA	1	243	R0427856	97%		
	MSD 4/23/98	1	NA	NA	1	233	R0427857	93%	95%	3%	NA	1	228	R0427857	91%	94%	6%
	MS 4/28/98	1	NA	NA	1	161	R0505852	65%			NA	1	295	R0430856	118%		
	MSD 4/28/98	1	NA	NA	1	457	R0505853	183% *	124%	96%	NA	1	283	R0430857	113%	116%	4%
Group 1 Control 0.0 mg/kg	C90719M	1	0.9275	Unknown	1	26.3	R0427813	0.0244			Unknown	1	0.00	R0427813	<LOQ (0.00249 ug/mL)		
	C90732M	1	0.9275	Unknown	1	32.5	R0427814	0.0302			Unknown	1	0.06	R0427814	<LOQ (0.00249 ug/mL)		
	C90725M	1	0.9275	Unknown	1	26.3	R0427815	0.0244			Unknown	1	0.00	R0427815	<LOQ (0.00249 ug/mL)		
	C90753M	1	0.9275	Unknown	1	80.1	R0427816	0.0743		53.0	Unknown	1	0.59	R0427816	<LOQ (0.00249 ug/mL)		NA
	C90775M	1	0.9275	Unknown	1	53.4	R0427817	0.0495	0.0405	0.0215	Unknown	1	0.31	R0427817	<LOQ (0.00249 ug/mL)	<LOQ	NA
	C91135F	1	0.9275	Unknown	1	120	R0427820	0.111			Unknown	1	0.35	R0427820	<LOQ (0.00249 ug/mL)		
	C91143F	1	0.9275	Unknown	1	114	R0427821	0.106			Unknown	1	0.83	R0427821	<LOQ (0.00249 ug/mL)		
	C91165F	1	0.9275	Unknown	1	135	R0427822	0.126			Unknown	1	0.64	R0427822	<LOQ (0.00249 ug/mL)		
	C91169F	1	0.9275	Unknown	1	133	R0427823	0.123		7.10	Unknown	1	0.37	R0427823	<LOQ (0.00249 ug/mL)		NA
	C91171F	1	0.9275	Unknown	1	127	R0427824	0.117	0.117	0.00828	Unknown	1	0.21	R0427824	<LOQ (0.00249 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90789M	1	0.9275	Unknown	50	32.5	R0505812	1.51			Unknown	10	2.55	R0430814	0.0255		
	C90814M	1	0.9275	Unknown	50	38.5	R0505813	1.78			Unknown	10	2.68	R0430815	0.0268		
	C90817M	1	0.9275	Unknown	50	41.3	R0505814	1.91			Unknown	10	2.97	R0430816	0.0297		
	C90833M	1	0.9275	Unknown	50	47.6	R0505815	2.21		13.6	Unknown	10	3.89	R0430817	0.0389		17.7
	C90834M	1	0.9275	Unknown	50	40.2	R0505816	1.87	1.86	0.252	Unknown	10	3.39	R0430818	0.03390	0.0310	0.00549
	C91198F	1	0.9275	Unknown	50	64.9	R0505819	3.01			Unknown	10	5.39	R0430821	0.0539		
	C91205F	1	0.9275	Unknown	50	78.2	R0505820	3.63			Unknown	10	6.56	R0430822	0.0656		
	C91222F	1	0.9275	Unknown	50	57.2	R0505821	2.65			Unknown	10	7.32	R0430823	0.0732		
	C91230F	1	0.9275	Unknown	50	74.7	R0505822	3.47		12.7	Unknown	10	5.26	R0430824	0.0526		14.1
	C91237F	1	0.9275	Unknown	50	76.4	R0505823	3.54	3.26	0.415	Unknown	10	6.57	R0430825	0.0657	0.0622	0.00874
Group 3 Mid Dose 30.0 mg/kg	C90846M	1	0.9275	Unknown	250	84.0	R0505826	19.5			Unknown	50	14.0	R0430828	0.701		
	C90853M	1	0.9275	Unknown	250	102	R0505827	23.6			Unknown	50	13.4	R0430829	0.668		
	C90866M	1	0.9275	Unknown	250	99.5	R0505828	23.1			Unknown	50	12.2	R0430830	0.611		
	C90890M	1	0.9275	Unknown	250	84.2	R0505829	19.5		9.19	Unknown	50	13.6	R0430831	0.680		11.7
	C90891M	1	0.9275	Unknown	250	96.8	R0505830	22.4	21.6	1.99	Unknown	50	16.6	R0430832	0.832	0.698	0.0818
	C91251F	1	0.9275	Unknown	250	119	R0505833	27.6			Unknown	50	25.9	R0430835	1.29		
	C91272F	1	0.9275	Unknown	250	145	R0505834	33.7			Unknown	50	34.4	R0430836	1.72		
	C91285F	1	0.9275	Unknown	250	105	R0505835	24.3			Unknown	50	12.3	R0430837	0.614		
	C91287F	1	0.9275	Unknown	250	138	R0505836	31.9		12.8	Unknown	50	27.0	R0430838	1.35		31.7
	C91291F	1	0.9275	Unknown	250	138	R0505837	32.0	29.9	3.84	Unknown	50	27.8	R0430839	1.39	1.27	0.404
Group 4 Mid-High Dose 100 mg/kg	C90935M	1	0.9275	Unknown	500	111	R0505840	51.5			Unknown	100	19.2	R0430842	1.92		
	C90939M	1	0.9275	Unknown	500	141	R0505841	65.4			Unknown	100	12.6	R0430843	1.26		
	C90942M	1	0.9275	Unknown	500	116	R0505842	53.6			Unknown	100	12.9	R0430844	1.29		
	C90961M	1	0.9275	Unknown	500	157	R0505843	72.8		14.5	Unknown	100	24.2	R0430845	2.42		28.7
	C90966M	1	0.9275	Unknown	500	126	R0505844	58.4	60.4	8.76	Unknown	100	21.7	R0430846	2.17	1.81	0.520
	C91316F	1	0.9275	Unknown	500	126	R0505847	58.6			Unknown	100	23.4	R0430849	2.34		
	C91317F	1	0.9275	Unknown	500	155	R0505848	71.9			Unknown	100	30.0	R0430850	3.00		
	C91333F	1	0.9275	Unknown	500	172	R0505849	79.7			Unknown	100	26.6	R0430851	2.66		
	C91345F	1	0.9275	Unknown	500	200	R0505850	92.8		23.7	Unknown	100	22.3	R0430852	2.23		13.8
	C91350F	1	0.9275	Unknown	500	109	R0505851	50.5	70.7	16.8	Unknown	100	21.8	R0430853	2.18	2.48	0.344
Group 5 High Dose 300 mg/kg	C90984M	1	0.9275	Unknown	500	404	R0427827	188			Unknown	5	208	R0427841	1.04		
	C91005M	1	0.9275	Unknown	1000	294	R0428814	273			Unknown	5	266	R0427842	1.33		
	C91024M	1	0.9275	Unknown	1000	293	R0428815	272			Unknown	5	208	R0427843	1.04		
	C91033M	1	0.9275	Unknown	500	481	R0427830	223		18.6	Unknown	5	267	R0427844	1.34		12.3
	C91037M	1	0.9275	Unknown	500	406	R0427831	188	229	42.5	Unknown	5	239	R0427845	1.20	1.19	0.147
	C91423F	1	0.9275	Unknown	1000	330	R0428820	306			Unknown	5	476	R0427849	2.38		
	C91427F	1	0.9275	Unknown	1000	291	R0428821	270			Unknown	500	6.96	R0427835	3.48		
	C91430F	1	0.9275	Unknown	1000	417	R0428822	387			Unknown	500	12.6	R0427836	6.29		
	C91443F	1	0.9275	Unknown	1000	451	R0428823	419		18.3	Unknown	5	382	R0427852	1.91		65.6
	C91448F	1	0.9275	Unknown	1000	435	R0428824	403	357	65.2	Unknown	5	233	R0427853	1.17	3.04	2.00

* Sample quantitated out of linear range of curve.

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate

Date Entered/By: 12/06/00 KJH
Date Verified/ By: 12/06/00 HOJ / 04/30/01 LAC
Purity Entered/Verified: 12/27/00 LAC

Corrected PFOS LOQ (0.0249 ug/mL) to include std correction factors
new LOQ is 0.0231 ug/mL. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1
Analytical Equipment System Number:	Chick 080697 and Madeline 041098
Instrument Software/Version:	MassLynx 2.3 & 3.1
Filename:	See listing to the right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	04/23/98, 04/28/98 RWW
Dates of Analysis/Analyst:	04/27/98, 04/28/98, 04/30/98, 05/05/98 KJH/HOJ
Date of Data Reduction/Analyst:	09/01/00, 12/01/00, 12/04/00, 12/05/00 MMH/KJH

Sample Data

WEEK 4 RAT SERA REWORK Lot 617

Group Dose	Sample #	PFOSAA Purity Correction Factor	PFOSAA Dilution Factor	PFOSAA Conc. ng/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	Unknown	1	0.00	R0427802	<LOQ (0.00256 ug/mL)		
	H2O Blk-2	Unknown	1	0.00	R0427859	<LOQ (0.00256 ug/mL)	<LOQ	NA
Matrix Blk	Rat Serum Blk-1	Unknown	1	0.00	R0427803	<LOQ (0.00256 ug/mL)		
	Rat Serum Blk-2	Unknown	1	0.00	R0427860	<LOQ (0.00256 ug/mL)	<LOQ	NA
QC-250 ppb	MS 4/23/98	NA	1	271	R0427856	108%		
	MSD 4/23/98	NA	1	252	R0427857	101%	104%	7%
	MS 4/28/98	NA	1	143	R0505852	57%		
	MSD 4/28/98	NA	1	466	R0505853	186% *	122%	106%
Group 1	C90719M	Unknown	1	13.7	R0427813	0.0137		
Control	C90732M	Unknown	1	43.2	R0427814	0.0432		
0.0 mg/kg	C90725M	Unknown	1	37.7	R0427815	0.0377		
	C90753M	Unknown	1	82.4	R0427816	0.0824		56.8
	C90775M	Unknown	1	78.7	R0427817	0.0787	0.0511	0.0291
	C91135F	Unknown	1	44.9	R04279820	0.0449		
	C91143F	Unknown	1	85.7	R04279821	0.0857		
	C91165F	Unknown	1	54.4	R04279822	0.0544		
	C91169F	Unknown	1	57.2	R04279823	0.0572		25.2
	C91171F	Unknown	1	59.7	R04279824	0.0597	0.0604	0.0152
Group 2	C90789M	Unknown	50	31.1	R0505812	1.55		
Low Dose	C90814M	Unknown	50	42.7	R0505813	2.13		
3.0 mg/kg	C90817M	Unknown	50	32.1	R0505814	1.60		
	C90833M	Unknown	50	109	R0505815	5.46		62.7
	C90834M	Unknown	50	45.0	R0505816	2.25	2.60	1.63
	C91198F	Unknown	50	112	R0505819	5.61		
	C91205F	Unknown	50	49.3	R0505820	2.47		
	C91222F	Unknown	50	54.2	R0505821	2.71		
	C91230F	Unknown	50	37.9	R0505822	1.90		47.3
	C91237F	Unknown	50	53.9	R0505823	2.70	3.07	1.45
Group 3	C90846M	Unknown	250	56.4	R0505826	14.1		
Mid Dose	C90853M	Unknown	250	45.8	R0505827	11.4		
30.0 mg/kg	C90866M	Unknown	250	45.3	R0505828	11.3		
	C90890M	Unknown	250	49.5	R0505829	12.4		17.4
	C90891M	Unknown	250	67.2	R0505830	16.8	13.2	2.30
	C91251F	Unknown	250	73.9	R0505833	18.5		
	C91272F	Unknown	250	139	R0505834	34.9		
	C91285F	Unknown	250	54.4	R0505835	13.6		
	C91287F	Unknown	250	74.3	R0505836	18.6		38.7
	C91291F	Unknown	250	121	R0505837	30.2	23.1	8.95
Group 4	C90935M	Unknown	500	44.6	R0505840	22.3		
Mid-High Dose	C90939M	Unknown	500	44.8	R0505841	22.4		
100 mg/kg	C90942M	Unknown	500	39.0	R0505842	19.5		
	C90961M	Unknown	500	85.3	R0505843	42.6		34.5
	C90966M	Unknown	500	55.5	R0505844	27.8	26.9	9.28
	C91316F	Unknown	500	47.5	R0505847	23.8		
	C91317F	Unknown	500	60.4	R0505848	30.2		
	C91333F	Unknown	500	64.0	R0505849	32.0		
	C91345F	Unknown	500	115	R0505850	57.3		40.1
	C91350F	Unknown	500	51.5	R0505851	25.8	33.8	13.6
Group 5	C90984M	Unknown	500	123	R0427827	61.3		
High Dose	C91005M	Unknown	500	137	R0427828	68.4		
300 mg/kg	C91024M	Unknown	500	151	R0427829	75.6		
	C91033M	Unknown	500	126	R0427830	63.1		23.2
	C91037M	Unknown	500	75.8	R0427831	37.9	61.2	14.2
	C91423F	Unknown	500	175	R0427834	87.5		
	C91427F	Unknown	500	168	R0427835	83.9		
	C91430F	Unknown	500	196	R0427836	97.8		
	C91443F	Unknown	500	271	R0427837	136		30.6
	C91448F	Unknown	500	115	R0427838	57.6	92.5	28.3

* Sample quantitated out of linear range of curve.

Date Entered/By:	12/06/00 KJH
Date Verified/ By:	12/06/00 HOJ / 04/30/01 LAC
Purity Entered/Verified:	12/27/00 LAC

Corrected PFOS LOQ (0.0249 ug/mL) to include std correction factors
new LOQ is 0.0231 ug/mL. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1
Analytical Equipment System Number:	Chick 080697 and Madeline 041098
Instrument Software/Version:	MassLynx 2.3 & 3.1
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	04/23/98, 04/28/98 RWW
Dates of Analysis/Analyst:	04/27/98, 04/28/98, 04/30/98, 05/05/98 KJH/HOJ
Date of Data Reduction/Analyst:	09/01/00, 12/01/00, 12/04/00, 12/05/00 MMH/KJH

Sample Data

WEEK 4 RAT SERA REWORK

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	<LOQ (0.0231 ug/mL)			<LOQ (0.00249 ug/mL)			<LOQ (0.00256 ug/mL)		
	H2O Blk-2	<LOQ (0.0231 ug/mL)	<LOQ	NA	<LOQ	<LOQ	NA	<LOQ (0.00256 ug/mL)	<LOQ	NA
Matrix Blk	Rat Serum Blk-1	<LOQ (0.0231 ug/mL)			<LOQ (0.00249 ug/mL)			<LOQ (0.00256 ug/mL)		
	Rat Serum Blk-2	<LOQ (0.0231 ug/mL)	<LOQ	NA	<LOQ	<LOQ	NA	<LOQ (0.00256 ug/mL)	<LOQ	NA
QC-250 ppb	MS 4/23/98	96%			97%			108%		
	MSD 4/23/98	93%	95%	3%	91%	94%	6%	101%	104%	7%
	MS 4/28/98	65%			118%			57%		
	MSD 4/28/98	183% *	124%	96%	113%	116%	4%	186% *	122%	106%
Group 1	C90719M	0.0244			<LOQ (0.00249 ug/mL)			0.01372		
Control	C90732M	0.0302			<LOQ (0.00249 ug/mL)			0.0432		
0.0 mg/kg	C90725M	0.0244			<LOQ (0.00249 ug/mL)			0.0377		
	C90753M	0.0743		53.0	<LOQ (0.00249 ug/mL)		NA	0.0824		56.8
	C90775M	0.0495	0.0405	0.0215	<LOQ (0.00249 ug/mL)	<LOQ	NA	0.0787	0.0511	0.0291
	C91135F	0.111			<LOQ (0.00249 ug/mL)			0.0449		
	C91143F	0.106			<LOQ (0.00249 ug/mL)			0.0857		
	C91165F	0.126			<LOQ (0.00249 ug/mL)			0.0544		
	C91169F	0.123		7.10	<LOQ (0.00249 ug/mL)		NA	0.0572		25.2
	C91171F	0.117	0.117	0.0083	<LOQ (0.00249 ug/mL)	<LOQ	NA	0.0597	0.0604	0.0152
Group 2	C90789M	1.51			0.0255			1.55		
Low Dose	C90814M	1.78			0.0268			2.13		
3.0 mg/kg	C90817M	1.91			0.0297			1.60		
	C90833M	2.21		13.6	0.0389		17.7	5.46		62.7
	C90834M	1.87	1.86	0.252	0.03390	0.0310	0.00549	2.25	2.60	1.63
	C91198F	3.01			0.0539			5.61		
	C91205F	3.63			0.0656			2.47		
	C91222F	2.65			0.0732			2.71		
	C91230F	3.47		12.7	0.0526		14.1	1.90		47.3
	C91237F	3.54	3.26	0.415	0.0657	0.0622	0.00874	2.70	3.07	1.45
Group 3	C90846M	19.5			0.701			14.1		
Mid Dose	C90853M	23.6			0.668			11.4		
30.0 mg/kg	C90866M	23.1			0.611			11.3		
	C90890M	19.5		9.19	0.680		11.7	12.4		17.4
	C90891M	22.4	21.6	1.99	0.832	0.698	0.0918	16.8	13.2	2.30
	C91251F	27.6			1.29			18.5		
	C91272F	33.7			1.72			34.9		
	C91285F	24.3			0.614			13.6		
	C91287F	31.9		12.8	1.35		31.7	18.6		38.7
	C91291F	32.0	29.9	3.84	1.39	1.27	0.404	30.2	23.1	9.0
Group 4	C90935M	51.5			1.92			22.3		
Mid-High Dose	C90939M	65.4			1.26			22.4		
100 mg/kg	C90942M	53.6			1.29			19.5		
	C90961M	72.8		14.5	2.42		28.7	42.6		34.5
	C90966M	58.4	60.4	8.8	2.17	1.81	0.520	27.8	26.9	9.3
	C91316F	58.6			2.34			23.8		
	C91317F	71.9			3.00			30.2		
	C91333F	79.7			2.66			32.0		
	C91345F	92.8		23.7	2.23		13.8	57.3		40.1
	C91350F	50.5	70.7	16.8	2.18	2.48	0.344	25.8	33.8	13.6
Group 5	C90984M	188			1.04			61.3		
High Dose	C91005M	273			1.33			68.4		
300 mg/kg	C91024M	272			1.04			75.6		
	C91033M	223		18.6	1.34		12.3	63.1		23.2
	C91037M	188	229	42.5	1.20	1.19	0.147	37.9	61.2	14.2
	C91423F	306			2.38			87.5		
	C91427F	270			3.48			83.9		
	C91430F	387			6.29			97.8		
	C91443F	419		18.3	1.91		65.6	136		30.6
	C91448F	403	357	65.2	1.17	3.04	2.00	57.6	92.5	28.3

* Sample quantitated out of linear range of curve

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate

Date Entered/By:	12/06/00 KJH
Date Verified/ By:	12/06/00 HOJ / 04/30/01- LAC
Purity Entered/Verified:	12/27/00 LAC

Corrected PFOS LOQ (0.0249 ug/mL) to include std correction factors
new LOQ is 0.0231 ug/mL. LAC 02/19/01

		PFOS	PFOSA	PFOSAA
		05/14/98 Lot 215	05/14/98 Lot L-2353	05/14/98 Lot 617

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1
Analytical Equipment System Number:	Madeline 041098
Instrument Software/Version:	MassLynx 2.3 & 3.1
Filename:	See listing to the right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Date of Extraction/Analyst:	05/11/98, 05/14/98 RWW/OK
Date of Analysis/Analyst:	05/13/98, 05/14/98 HOJ/KJH/LAD/JJ
Date of Data Reduction/Analyst:	04/10/00, 01/03/01 CSH/MMH

Filenames

	PFOS	PFOSA	PFOSAA	Dilutions
Blks	R05149802-03 & 86-87	R05149802-03 & 86-87	R05149802-03 & 86-87	
Grp 1	R05149812-23	R05149812-23	R05149812-23	1/1, 1/1, 1/1
Grp 5	R05149826-37	R05149854-65	R05149840-51	1/5000, 1/15, 1/500
MS, MSD	R05149883-84	R05149883-84	R05149883-84	1/1, 1/1, 1/1

Sample Data

WEEK 8 RAT SERA REWORK

Group Dose	Sample #	Extraction Vol. mL	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec (Lot 215)	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec (Lot L-2353)	Mean PFOSA ug/mL
Method Blk	H2O Blk-1	1	0.9275	Unknown	1	0.00	R05149802	<LOQ (0.00905 ug/ml)			Unknown	1	0.00	R05149802	<LOQ (0.00978 ug/mL)	
	H2O Blk-2	1	0.9275	Unknown	1	8.35	R05149886	<LOQ (0.00905 ug/ml) **	<LOQ	NA	Unknown	1	0.00	R05149886	<LOQ (0.00978 ug/mL)	<LOQ
Matrix Blk	Rat Serum Blk-1	1	0.9275	Unknown	1	6.13	R05149803	<LOQ (0.00905 ug/ml)			Unknown	1	6.32	R05149803	<LOQ (0.00978 ug/mL)	
	Rat Serum Blk-2	1	0.9275	Unknown	1	0.00	R05149887	<LOQ (0.00905 ug/ml) **	<LOQ	NA	Unknown	1	0.00	R08149887	<LOQ (0.00978 ug/mL)	<LOQ
QC-75 ppb	MS 5/11/98	1	NA	NA	1	76.3	R05149883	104% **			NA	1	75.4	R05149883	103%	
	MSD 5/11/98	1	NA	NA	1	91.6	R05149884	125% **	115%	18%	NA	1	84.6	R05149884	116%	110%
Group 1	C90717M	1	0.9275	Unknown	1	36.9	R05149812	0.0342			Unknown	1	5.90	R05149812	<LOQ (0.00978 ug/mL)	
Control	C90718M	1	0.9275	Unknown	1	52.0	R05149813	0.0482			Unknown	1	5.88	R05149813	<LOQ (0.00978 ug/mL)	
0.0 mg/kg	C90737M	1	0.9275	Unknown	1	53.4	R05149814	0.0495			Unknown	1	6.12	R05149814	<LOQ (0.00978 ug/mL)	
	C90756M	1	0.9275	Unknown	1	87.2	R05149815	0.0809		33.5	Unknown	1	5.91	R05149815	<LOQ (0.00978 ug/mL)	
	C90769M	1	0.9275	Unknown	1	49.5	R05149816	0.0459	0.0518	0.0174	Unknown	1	0.00	R05149816	<LOQ (0.00978 ug/mL)	<LOQ
	C91126F	1	0.9275	Unknown	1	196	R05149819	0.182			Unknown	1	6.49	R05149819	<LOQ (0.00978 ug/mL)	
	C91140F	1	0.9275	Unknown	1	183	R05149820	0.170			Unknown	1	6.47	R05149820	<LOQ (0.00978 ug/mL)	
	C91145F	1	0.9275	Unknown	1	176	R05149821	0.163			Unknown	1	5.92	R05149821	<LOQ (0.00978 ug/mL)	
	C91162F	1	0.9275	Unknown	1	225	R05149822	0.209		9.60	Unknown	1	6.00	R05149822	<LOQ (0.00978 ug/mL)	
	C91163F	1	0.9275	Unknown	1	194	R05149823	0.180	0.181	0.0174	Unknown	1	6.12	R05149823	<LOQ (0.00978 ug/mL)	<LOQ
Group 5	C90971M	1	0.9275	Unknown	5000	73.6	R05149826	341			Unknown	15	76.7	R05149854	1.15	
High Dose	C90972M	1	0.9275	Unknown	5000	67.1	R05149827	311			Unknown	15	70.4	R05149855	1.06	
300 mg/kg	C90988M	1	0.9275	Unknown	5000	73.6	R05149828	341			Unknown	15	41.8	R05149856	0.627	
	C90996M	1	0.9275	Unknown	5000	70.5	R05149829	327		11.9	Unknown	15	88.5	R05149857	1.33	
	C91017M	1	0.9275	Unknown	5000	90.3	R05149830	419	348	41.5	Unknown	15	88.4	R05149858	1.33	1.10
	C91393F	1	0.9275	Unknown	5000	120	R05149833	557			Unknown	15	51.5	R05149861	0.773	
	C91394F	1	0.9275	Unknown	5000	121	R05149834	559			Unknown	15	113	R05149862	1.69	
	C91404F	1	0.9275	Unknown	5000	114	R05149835	530			Unknown	15	58.8	R05149863	0.882	
	C91415F	1	0.9275	Unknown	5000	130	R05149836	603		6.96	Unknown	15	55.4	R05149864	0.830	
	C91438F	1	0.9275	Unknown	5000	136	R05149837	631	576	40.1	Unknown	15	80.2	R05149865	1.20	1.08

** Tentative results, CCVs did not meet criteria. LAC 01/08/01

Date Entered/By: 05/24/00, 01/08/01 CSH/LAC

Date Verified/ By: 12/28/00 HOJ / 01/08/01 KJH / 04/30/01 LAC

Purity Entered/Verified: 12/27/00 LAC

PFOS = Perfluorooctanesulfonate

PFOSA = Perfluorooctanesulfonamide

PFOSAA = Perfluorooctanesulfonamidoacetate

Corrected PFOS LOQ (0.00976 ug/mL) to include std correction factors
new LOQ is 0.00905 ug/mL. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1
Analytical Equipment System Number:	Madeline 041098
Instrument Software/Version:	MassLynx 2.3 & 3.1
Filename:	See listing to the right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Date of Extraction/Analyst:	05/11/98, 05/14/98 RWW/OK
Date of Analysis/Analyst:	05/13/98, 05/14/98 HOJ/KJH/LAD/JJ
Date of Data Reduction/Analyst:	04/10/00, 01/03/01 CSH/MMH

Sample Data

WEEK 8 RAT SERA REWORK Lot 617

RSD Std. Dev. MS/MSD RPD	Group Dose	Sample #	PFOSAA Purity Correction Factor	PFOSAA Dilution Factor	PFOSAA Conc. ng/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
	Method Blk	H2O Blk-1	Unknown	1	0.00	R05149802	<LOQ (0.0100 ug/mL)		
NA		H2O Blk-2	Unknown	1	0.00	R05149886	<LOQ (0.0100 ug/mL) **	<LOQ	NA
	Matrix Blk	Rat Serum Blk-1	Unknown	1	0.00	R05149803	<LOQ (0.0100 ug/mL)		
NA		Rat Serum Blk-2	Unknown	1	0.00	R05149887	<LOQ (0.0100 ug/mL) **	<LOQ	NA
	QC-75 ppb	MS 5/11/98	NA	1	76.9	R05149883	103% **		
12%		MSD 5/11/98	NA	1	78.3	R05149884	104% **	103%	2%
	Group 1	C90717M	Unknown	1	18.6	R05149812	<LOQ (0.0100 ug/mL)		
	Control	C90718M	Unknown	1	24.3	R05149813	0.0243		
	0.0 mg/kg	C90737M	Unknown	1	17.9	R05149814	<LOQ (0.0100 ug/mL)		
NA		C90756M	Unknown	1	30.4	R05149815	0.0304		57.5
NA		C90769M	Unknown	1	24.2	R05149816	<LOQ (0.0100 ug/mL)	0.0169	0.00974
		C91126F	Unknown	1	30.2	R05149819	0.0302		
		C91140F	Unknown	1	55.2	R05149820	0.0552		
		C91145F	Unknown	1	38.1	R05149821	0.0381		
NA		C91162F	Unknown	1	43.4	R05149822	0.0434		22.5
NA		C91163F	Unknown	1	49.5	R05149823	0.0495	0.0433	0.00973
	Group 5	C90971M	Unknown	500	151	R05149840	75.3		
	High Dose	C90972M	Unknown	500	165	R05149841	82.3		
	300 mg/kg	C90988M	Unknown	500	103	R05149842	51.6		
26.2		C90996M	Unknown	500	126	R05149843	63.2		17.9
0.288		C91017M	Unknown	500	125	R05149844	62.3	67.0	12.0
		C91393F	Unknown	500	212	R05149847	106		
		C91394F	Unknown	500	130	R05149848	64.8		
		C91404F	Unknown	500	157	R05149849	78.7		
35.4		C91415F	Unknown	500	158	R05149850	79.1		18.8
0.381		C91438F	Unknown	500	152	R05149851	75.9	80.9	15.3

** Tentative results, CCVs did not meet criteria. LAC 01/08/01

Date Entered/By:	05/24/00, 01/08/01 CSH/LAC
Date Verified/ By:	12/28/00 HOJ / 01/08/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	12/27/00 LAC

Corrected PFOS LOQ (0.00976 ug/mL) to include std correction factors
new LOQ is 0.00905 ug/mL. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1
Analytical Equipment System Number:	Madeline 041098
Instrument Software/Version:	MassLynx 2.3 & 3.1
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Date of Extraction/Analyst:	05/11/98, 05/14/98 RWW/OK
Date of Analysis/Analyst:	05/13/98, 05/14/98 HOJ/KJH/LAD/JJ
Date of Data Reduction/Analyst:	04/10/00, 01/03/01 CSH/MMH

Sample Data

WEEK 8 RAT SERA REWORK

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	<LOQ (0.00905 ug/ml)			<LOQ (0.00978 ug/mL)			<LOQ (0.0100 ug/mL)		
	H2O Blk-2	<LOQ (0.00905 ug/ml) **	<LOQ	NA	<LOQ (0.00978 ug/mL)	<LOQ	NA	<LOQ (0.0100 ug/mL) **	<LOQ	NA
Matrix Blk	Rat Serum Blk-1	<LOQ (0.00905 ug/ml)			<LOQ (0.00978 ug/mL)			<LOQ (0.0100 ug/mL)		
	Rat Serum Blk-2	<LOQ (0.00905 ug/ml) **	<LOQ	NA	<LOQ (0.00978 ug/mL)	<LOQ	NA	<LOQ (0.0100 ug/mL) **	<LOQ	NA
QC-75 ppb	MS 5/11/98	104% **			103%			103% **		
	MSD 5/11/98	125% **	115%	18%	116%	110%	12%	104% **	103%	2%
Group 1	C90717M	0.0342			<LOQ (0.00978 ug/mL)			<LOQ (0.0100 ug/mL)		
Control	C90718M	0.0482			<LOQ (0.00978 ug/mL)			0.0243		
0.0 mg/kg	C90737M	0.0495			<LOQ (0.00978 ug/mL)			<LOQ (0.0100 ug/mL)		
	C90756M	0.0809		33.5	<LOQ (0.00978 ug/mL)		NA	0.0304		57.53
	C90769M	0.0459	0.0518	0.0174	<LOQ (0.00978 ug/mL)	<LOQ	NA	<LOQ (0.0100 ug/mL)	0.0169	0.00974
	C91126F	0.182			<LOQ (0.00978 ug/mL)			0.0302		
	C91140F	0.170			<LOQ (0.00978 ug/mL)			0.0552		
	C91145F	0.163			<LOQ (0.00978 ug/mL)			0.0381		
	C91162F	0.209		9.60	<LOQ (0.00978 ug/mL)		NA	0.0434		22.5
	C91163F	0.180	0.181	0.0174	<LOQ (0.00978 ug/mL)	<LOQ	NA	0.0495	0.0433	0.00973
Group 5	C90971M	341			1.15			75.3		
High Dose	C90972M	311			1.06			82.3		
300 mg/kg	C90988M	341			0.63			51.6		
	C90996M	327		11.9	1.33		26.2	63.2		17.9
	C91017M	419	348	41.5	1.33	1.10	0.288	62.3	67.0	12.0
	C91393F	557			0.773			106		
	C91394F	559			1.69			64.8		
	C91404F	530			0.88			78.7		
	C91415F	603		6.96	0.83		35.4	79.1		18.8
	C91438F	631	576	40.1	1.20	1.08	0.381	75.9	80.9	15.3

** Tentative results, CCVs did not meet criteria. LAC 01/08/01

Date Entered/By:	05/24/00, 01/08/01 CSH/LAC
Date Verified/ By:	12/28/00 HOJ / 01/08/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	12/27/00 LAC

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate

Corrected PFOS LOQ (0.00976 ug/mL) to include std correction factors
new LOQ is 0.00905 ug/mL. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1
Analytical Equipment System Number:	Madeline 041098
Instrument Software/Version:	MassLynx 2.3, 3.1, 3.2, 3.3
Filename:	See listing to the right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Date of Extraction/Analyst:	05/11/98 RWW/OK
Date of Analysis/Analyst:	05/14/98, 05/15/98, 05/17/98, 6/05/98 HOJ/KJH
Date of Data Reduction/Analyst:	12/01/00, 12/29/00, 01/02/01 MMH/CSH

PFOS	PFOSA	PFOSAA
05/14/98 Lot 215	05/14/98 Lot L-2353	05/14/98 Lot 617
05/15/98 Lot 215	05/17/98 Lot L-2353	05/15/98 Lot 617
06/05/98 Lot 215	06/05/98 Lot L-2353	06/05/98 Lot 617

Filenames

	PFOS	PFOSA	PFOSAA	Dilutions
Blks	51598004-5, 123-124	51798004-5, 97-98	51598004-5, 123-124	1/1, 1/1, 1/1
Grp 1	60598020-31	60598020-31	60598020-31	1/1, 1/1, 1/1
Grp 2	51598046-58	51798016-38	51598103-116	1/100, 1/2, 1/25
Grp 3	51598030-42	51798043-64	51598084-97	1/500, 1/5, 1/100
Grp 4	51598014-26	51798069-90	51598065-78	1/1000, 1/5, 1/250
MS, MSD	R05149883-84	R05149883-84	R05149883-84	1/1, 1/1, 1/1

Sample Data

WEEK 14 RAT SERA REWORK

Group Dose	Sample #	Extraction Vol. Ratio	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename (Lot 215)	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename (Lot L-2353)	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	1	0.9275	Unknown	1	0.00	51598004	<LOQ (0.0230 ug/mL)			Unknown	1	6.62	51798004	<LOQ(0.0978 ug/mL)		
	H2O Blk-2	1	0.9275	Unknown	1	0.00	51598123	<LOQ (0.0230 ug/mL)	<LOQ	NA	Unknown	1	6.71	51798097	<LOQ(0.0978 ug/mL)	<LOQ	NA
Matrix Blk	Rat Serum Blk-1	1	0.9275	Unknown	1	9.31	51598005	<LOQ (0.0230 ug/mL)			Unknown	1	6.59	51798005	<LOQ(0.0978 ug/mL)		
	Rat Serum Blk-2	1	0.9275	Unknown	1	6.03	51598124	<LOQ (0.0230 ug/mL)	<LOQ	NA	Unknown	1	6.83	51798098	<LOQ(0.0978 ug/mL)	<LOQ	NA
QC-75 ppb	MS 5/11/98	1	NA	NA	1	76.3	R05149883	104% **			NA	1	75.4	R05149883	103%		
	MSD 5/11/98	1	NA	NA	1	91.6	R05149884	125% **	115%	18%	NA	1	84.6	R05149884	116%	110%	12%
Group 1	C90731M	1	0.9275	Unknown	1	90.7	60598020	0.0842			Unknown	1	7.65	60598020	<LOQ(0.0978 ug/mL)		
Control	C90746M	1	0.9275	Unknown	1	95.2	60598021	0.0883			Unknown	1	0.00	60598021	<LOQ(0.0978 ug/mL)		
0.0 mg/kg	C90748M	1	0.9275	Unknown	1	81.2	60598022	0.0753			Unknown	1	0.00	60598022	<LOQ(0.0978 ug/mL)		
	C90768M	1	0.9275	Unknown	1	77.6	60598023	0.0720		10.1	Unknown	1	7.44	60598023	<LOQ(0.0978 ug/mL)		NA
	C90780M	1	0.9275	Unknown	1	75.5	60598024	0.0700	0.0779	0.00791	Unknown	1	7.30	60598024	<LOQ(0.0978 ug/mL)	<LOQ	NA
	C91129F	1	0.9275	Unknown	1	159	60598027	0.148			Unknown	1	0.00	60598027	<LOQ(0.0978 ug/mL)		
	C91155F	1	0.9275	Unknown	1	246	60598028	0.228			Unknown	1	0.00	60598028	<LOQ(0.0978 ug/mL)		
	C91160F	1	0.9275	Unknown	1	232	60598029	0.215			Unknown	1	8.00	60598029	<LOQ(0.0978 ug/mL)		
	C91174F	1	0.9275	Unknown	1	240	60598030	0.223		18.4	Unknown	1	0.00	60598030	<LOQ(0.0978 ug/mL)		NA
	C91181F	1	0.9275	Unknown	1	181	60598031	0.168	0.196	0.0361	Unknown	1	7.13	60598031	<LOQ(0.0978 ug/mL)	<LOQ	NA
Group 2	C90797M	1	0.9275	Unknown	100	59.0	51598046	5.48			Unknown	2	37.0	51798016	0.0740		
Low Dose	C90807M	1	0.9275	Unknown	100	61.3	51598047	5.69			Unknown	2	25.8	51798018	0.0516		
3.0 mg/kg	C90818M	1	0.9275	Unknown	100	77.0	51598048	7.14			Unknown	2	29.8	51798020	0.0597		
	C90831M	1	0.9275	Unknown	100	70.4	51598049	6.53		11.2	Unknown	2	23.4	51798022	0.0468		18.4
	C90836M	1	0.9275	Unknown	100	63.0	51598050	5.85	6.14	0.688	Unknown	2	33.4	51798025	0.0667	0.0598	0.0110
	C91192F	1	0.9275	Unknown	100	124	51598054	11.5			Unknown	2	46.5	51798030	0.0930		
	C91223F	1	0.9275	Unknown	100	130	51598055	12.0			Unknown	2	54.0	51798032	0.108		
	C91233F	1	0.9275	Unknown	100	165	51598056	15.3			Unknown	2	78.5	51798034	0.157		
	C91241F	1	0.9275	Unknown	100	128	51598057	11.8		14.9	Unknown	2	53.0	51798036	0.1059		25.5
	C91248F	1	0.9275	Unknown	100	113	51598058	10.5	12.2	1.83	Unknown	2	42.6	51798038	0.0852	0.110	0.0280
Group 3	C90843M	1	0.9275	Unknown	500	101	51598030	47.0			Unknown	5	66.0	51798043	0.330		
Mid Dose	C90852M	1	0.9275	Unknown	500	137	51598031	63.3			Unknown	5	124	51798045	0.621		
30.0 mg/kg	C90863M	1	0.9275	Unknown	500	101	51598032	47.0			Unknown	5	74.5	51798047	0.372		
	C90877M	1	0.9275	Unknown	500	153	51598033	71.0		19.2	Unknown	5	128	51798049	0.641		28.5
	C90880M	1	0.9275	Unknown	500	145	51598034	67.1	59.1	11.3	Unknown	5	101	57198051	0.505	0.494	0.141
	C91281F	1	0.9275	Unknown	500	260	51598038	120			Unknown	5	135	51798056	0.677		
	C91288F	1	0.9275	Unknown	500	158	51598039	73.1			Unknown	5	168	51798058	0.838		
	C91293F	1	0.9275	Unknown	500	248	51598040	115			Unknown	5	117	51798060	0.584		
	C91299F	1	0.9275	Unknown	500	281	51598041	130		24.5	Unknown	5	189	51798062	0.945		18.3
	C91304F	1	0.9275	Unknown	500	174	51598042	80.5	104	25.4	Unknown	5	160	51798064	0.799	0.769	0.141
Group 4	C90905M	1	0.9275	Unknown	1000	237	51598014	220			Unknown	5	142	51798069	0.708		
Mid-High Dose	C90907M	1	0.9275	Unknown	1000	191	51598015	177			Unknown	5	151	51798071	0.755		
100 mg/kg	C90917M	1	0.9275	Unknown	1000	233	51598016	216			Unknown	5	162	51798073	0.808		
	C90921M	1	0.9275	Unknown	1000	177	51598017	164		12.8	Unknown	5	173	51798075	0.866		7.8
	C90960M	1	0.9275	Unknown	1000	197	51598018	183	192	24.6	Unknown	5	151	57198077	0.753	0.778	0.0605
	C91329F	1	0.9275	Unknown	1000	242	51598022	225			Unknown	5	274	51798082	1.37		
	C91337F	1	0.9275	Unknown	1000	304	51598023	282			Unknown	5	408	51798084	2.04		
	C91355F	1	0.9275	Unknown	1000	289	51598024	268			Unknown	5	397	51798086	1.99		
	C91370F	1	0.9275	Unknown	1000	339	51598025	315		12.7	Unknown	5	285	51798088	1.42		26.7
	C91379F	1	0.9275	Unknown	1000	268	51598026	248	268	34.0	Unknown	5	518	57198090	2.59	1.88	0.503

Extraction Volume Ratio = Initial volume/final volume. For all samples and standards the initial volume is equal to the final volume for an extraction volume ratio of 1.

Date Entered/By: 6/19/98 LAC, 12/06/00 KJH, 01/05/01 LAC

Date Verified/ By: 7/16/98 GML / 12/07/00 HOJ / 01/08/01 KJH / 04/30/01 LAC

Purity Entered/Verified: 12/27/00 LAC

PFOS = Perfluorooctanesulfonate

PFOSA = Perfluorooctanesulfonamide

PFOSAA = Perfluorooctanesulfonamidoacetate

Corrected PFOS LOQ (0.0248 ug/mL) to include std correction factors
new LOQ is 0.0230 ug/mL. LAC 02/19/01

** Tentative results, CCVs did not meet criteria. LAC 01/08/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1
Analytical Equipment System Number:	Madeline 041098
Instrument Software/Version:	MassLynx 2.3, 3.1, 3.2, 3.3
Filename:	See listing to the right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Date of Extraction/Analyst:	05/11/98 RWW/OK
Date of Analysis/Analyst:	05/14/98, 05/15/98, 05/17/98, 6/05/98 HOJ/KJH
Date of Data Reduction/Analyst:	12/01/00, 12/29/00, 01/02/01 MMH/CSH

Sample Data

WEEK 14 RAT SERA REWORK Lot 617

Group Dose	Sample #	PFOSAA Purity Correction Factor	PFOSAA Dilution Factor	PFOSAA Conc. ug/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	Unknown	1	0.00	51598004	<LOQ (0.0255 ug/mL)		
	H2O Blk-2	Unknown	1	0.00	51598123	<LOQ (0.0255 ug/mL)	<LOQ	NA
Matrix Blk	Rat Serum Blk-1	Unknown	1	0.00	51598005	<LOQ (0.0255 ug/mL)		
	Rat Serum Blk-2	Unknown	1	0.00	51598124	<LOQ (0.0255 ug/mL)	<LOQ	NA
QC-75 ppb	MS 5/11/98	NA	1	76.9	R05149883	103% **		
	MSD 5/11/98	NA	1	78.3	R05149884	104% **	103%	2%
Group 1	C90731M	Unknown	1	53.9	60598020	0.0539		
Control	C90746M	Unknown	1	49.6	60598021	0.0496		
0.0 mg/kg	C90748M	Unknown	1	18.5	60598022	<LOQ (0.0255 ug/mL)		
	C90768M	Unknown	1	23.6	60598023	<LOQ (0.0255 ug/mL)		40.2
	C90780M	Unknown	1	22.9	60598024	<LOQ (0.0255 ug/mL)	0.0360	0.0145
	C91129F	Unknown	1	35.5	60598027	0.0355		
	C91155F	Unknown	1	38.0	60598028	0.0380		
	C91160F	Unknown	1	35.9	60598029	0.0359		
	C91174F	Unknown	1	46.9	60598030	0.0469		20.9
	C91181F	Unknown	1	18.4	60598031	<LOQ (0.0255 ug/mL)	0.0363	0.00761
Group 2	C90797M	Unknown	25	65.4	51598103	1.63		
Low Dose	C90807M	Unknown	25	28.5	51598104	0.711		
3.0 mg/kg	C90818M	Unknown	25	69.5	51598105	1.74		
	C90831M	Unknown	25	55.9	51598106	1.40		41.1
	C90836M	Unknown	25	27.1	51598107	0.68	1.23	0.506
	C91192F	Unknown	25	56.6	51598112	1.41		
	C91223F	Unknown	25	108	51598113	2.70		
	C91233F	Unknown	25	99.0	51598114	2.47		
	C91241F	Unknown	25	53.3	51598115	1.33		35.2
	C91248F	Unknown	25	57.6	51598116	1.44	1.87	0.659
Group 3	C90843M	Unknown	100	80.5	51598084	8.05		
Mid Dose	C90852M	Unknown	100	149	51598085	14.9		
30.0 mg/kg	C90863M	Unknown	100	88.4	51598086	8.84		
	C90877M	Unknown	100	246	51598087	24.6		48.1
	C90880M	Unknown	100	126	51598088	12.6	13.8	6.63
	C91281F	Unknown	100	106	51598093	10.6		
	C91288F	Unknown	100	142	51598094	14.2		
	C91293F	Unknown	100	141	51598095	14.1		
	C91299F	Unknown	100	309	51598096	30.9		50.4
	C91304F	Unknown	100	121	51598097	12.1	16.4	8.26
Group 4	C90905M	Unknown	250	57.7	51598065	14.4		
Mid-High Dose	C90907M	Unknown	250	99.1	51598066	24.8		
100 mg/kg	C90917M	Unknown	250	87.7	51598067	21.9		
	C90921M	Unknown	250	97.8	51598068	24.4		21.1
	C90960M	Unknown	250	73.5	51598069	18.4	20.8	4.38
	C91329F	Unknown	250	102	51598074	25.4		
	C91337F	Unknown	250	153	51598075	38.1		
	C91355F	Unknown	250	124	51598076	31.0		
	C91370F	Unknown	250	158	51598077	39.4		16.9
	C91379F	Unknown	250	141	51598078	35.3	33.8	5.71

Extraction Volume Ratio = Initial volume/final volume. For all samples and standards the initial volume is equal to the final volume for an extraction volume ratio of 1.

Date Entered/By:	6/19/98 LAC, 12/06/00 KJH, 01/05/01 LAC
Date Verified/ By:	7/16/98 GML / 12/07/00 HOJ / 01/08/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	12/27/00 LAC

Corrected PFOS LOQ (0.0248 ug/mL) to include std correction factors
new LOQ is 0.0230 ug/mL. LAC 02/19/01

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: FACT-M-3.0 & FACT-M-4.0 reworked using ETS-8-5.1
Analytical Equipment System Number: Madeline 041098
Instrument Software/Version: MassLynx 2.3, 3.1, 3.2, 3.3
Filename: See Attachments
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Date of Extraction/Analyst: 05/11/98 RWW/OK
Date of Analysis/Analyst: 05/14/98, 05/15/98, 05/17/98, 6/05/98 HOJ/KJH
Date of Data Reduction/Analyst: 12/01/00, 12/29/00, 01/02/01 MMH/CSH

Sample Data

WEEK 14 RAT SERA REWORK

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	<LOQ (0.0230 ug/mL)			<LOQ(0.0978 ug/mL)			<LOQ (0.0255 ug/mL)		
	H2O Blk-2	<LOQ (0.0230 ug/mL)	<LOQ	NA	<LOQ(0.0978 ug/mL)	<LOQ	NA	<LOQ (0.0255 ug/mL)	<LOQ	NA
Matrix Blk	Rat Serum Blk-1	<LOQ (0.0230 ug/mL)			<LOQ(0.0978 ug/mL)			<LOQ (0.0255 ug/mL)		
	Rat Serum Blk-2	<LOQ (0.0230 ug/mL)	<LOQ	NA	<LOQ(0.0978 ug/mL)	<LOQ	NA	<LOQ (0.0255 ug/mL)	<LOQ	NA
QC-75 ppb	MS 5/11/98	104% **			103%			103% **		
	MSD 5/11/98	125% **	115%	18%	116%	110%	12%	104% **	103%	2%
Group 1	C90731M	0.0842			<LOQ(0.0978 ug/mL)			0.0539		
Control	C90746M	0.0883			<LOQ(0.0978 ug/mL)			0.0496		
0.0 mg/kg	C90748M	0.0753			<LOQ(0.0978 ug/mL)			<LOQ (0.0255 ug/mL)		
	C90768M	0.0720		10.1	<LOQ(0.0978 ug/mL)		NA	<LOQ (0.0255 ug/mL)		40.2
	C90780M	0.0700	0.0779	0.00791	<LOQ(0.0978 ug/mL)	<LOQ	NA	<LOQ (0.0255 ug/mL)	0.0360	0.0145
	C91129F	0.148			<LOQ(0.0978 ug/mL)			0.0355		
	C91155F	0.228			<LOQ(0.0978 ug/mL)			0.0380		
	C91160F	0.215			<LOQ(0.0978 ug/mL)			0.0359		
	C91174F	0.223		18.4	<LOQ(0.0978 ug/mL)		NA	0.0469		20.9
	C91181F	0.168	0.196	0.0361	<LOQ(0.0978 ug/mL)	<LOQ	NA	<LOQ (0.0255 ug/mL)	0.0363	0.00761
Group 2	C90797M	5.48			0.0740			1.63		
Low Dose	C90807M	5.69			0.0516			0.711		
3.0 mg/kg	C90818M	7.14			0.0597			1.74		
	C90831M	6.53		11.2	0.0468		18.4	1.40		41.1
	C90836M	5.85	6.14	0.688	0.0667	0.0598	0.0110	0.68	1.23	0.506
	C91192F	11.5			0.0930			1.41		
	C91223F	12.0			0.108			2.70		
	C91233F	15.3			0.157			2.47		
	C91241F	11.8		14.9	0.106		25.5	1.33		35.2
	C91248F	10.5	12.24	1.83	0.0852	0.110	0.0280	1.44	1.87	0.659
Group 3	C90843M	47.0			0.330			8.05		
Mid Dose	C90852M	63.3			0.621			14.9		
30.0 mg/kg	C90863M	47.0			0.372			8.84		
	C90877M	71.0		19.2	0.641		28.5	24.6		48.1
	C90880M	67.1	59.1	11.3	0.505	0.494	0.141	12.6	13.8	6.63
	C91281F	120			0.677			10.6		
	C91288F	73.1			0.838			14.2		
	C91293F	115			0.584			14.1		
	C91299F	130		24.5	0.945		18.3	30.9		50.4
	C91304F	80.5	104	25.4	0.799	0.769	0.141	12.1	16.4	8.26
Group 4	C90905M	220			0.708			14.4		
Mid-High Dose	C90907M	177			0.755			24.8		
100 mg/kg	C90917M	216			0.808			21.9		
	C90921M	164		12.8	0.866		7.78	24.4		21.1
	C90960M	183	192	24.6	0.753	0.778	0.0605	18.4	20.8	4.38
	C91329F	225			1.37			25.4		
	C91337F	282			2.04			38.1		
	C91355F	268			1.99			31.0		
	C91370F	315		12.7	1.42		26.7	39.4		16.9
	C91379F	248	268	34.0	2.59	1.88	0.503	35.3	33.8	5.71

Extraction Volume Ratio = Initial volume/final volume. For all samples and standards the initial volume is equal to the final volume for an extraction volume ratio of 1.

Date Entered/By: 6/19/98 LAC, 12/06/00 KJH, 01/05/01 LAC
Date Verified/ By: 7/16/98 GML / 12/07/00 HOJ / 01/08/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 12/27/00 LAC

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate

Corrected PFOS LOQ (0.0248 ug/mL) to include std correction factors
new LOQ is 0.0230 ug/mL. LAC 02/19/01
** Tentative results. CCVs did not meet criteria. LAC 01/08/01

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number: Davey 070799 & Ruby 100699
Instrument Software/Version: Masslynx 3.3 & 3.4
Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Dates of Extraction/Analyst: 02/23/00, 03/28/00 SAL
Dates of Analysis/Analyst: 03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 5/17/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst: 03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 5/18/00, 07/25/00, 08/18/00 IAS/HOJ/MMH

Sample Data

WEEK 27 RAT SERA

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor	Surrogate Verified	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
					lot i71							lot L-15709							
Method Blk	RBS02230-H2O Blk-5	1	NA	0.9275	0.8640	1	0.00	R032700006	<LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	R032700006	<LOQ (0.00493 ug/mL)		
	RBS02230-H2O Blk-6	1	NA	0.9275	0.8640	1	0.00	D040300006	<LOQ (0.00781 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D040300006	<LOQ (0.00493 ug/mL)	<LOQ	NA
Matrix Blk	RBS02230-Sera Blk-5	1	NA	0.9275	0.8640	1	1.11	R032700007	<LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	R032700007	<LOQ (0.00493 ug/mL)		
	RBS02230-Sera Blk-6	1	NA	0.9275	0.8640	1	0.00	D040300007	<LOQ (0.00781 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D040300007	<LOQ (0.00493 ug/mL)	<LOQ	NA
Matrix Blk	RTS02230-Sera Blk-5	1	NA	0.9275	0.8640	1	1.28	R032700008	<LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	R032700008	<LOQ (0.00493 ug/mL)		
	RTS02230-Sera Blk-6	1	NA	0.9275	0.8640	1	0.00	D040300008	<LOQ (0.00781 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D040300008	<LOQ (0.00493 ug/mL)	<LOQ	NA
QC-250 ng/mL	RTS02230-MS-5-1	1	NA	NA	NA	1	268	R032700020	109%			NA	NA	1	259	R032700020	104%		
	RTS02230-MS-5-1	1	NA	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA		
	RTS02230-MS-5-2	1	NA	NA	NA	1	216	R032700021	88%			NA	NA	1	242	R032700021	98%		
	RTS02230-MS-5-2	1	NA	NA	NA	NA	NA	NA	NA	98%	21%	NA	NA	NA	NA	NA	NA	101%	7%
	RTS02230-MS-5-3	1	NA	NA	NA	1	166	R032700022	67%			NA	NA	1	181	R032700022	73%		
	RTS02230-MS-5-3	1	NA	NA	NA	1	154	R032800020	62% 2nd			NA	NA	NA	NA	NA	NA		
	RTS02230-MS-5-4	1	NA	NA	NA	1	184	R032700023	74%			NA	NA	1	202	R032700023	81%		
	RTS02230-MS-5-4	1	NA	NA	NA	NA	NA	NA	NA	68%	10%	NA	NA	NA	NA	NA	NA	77%	11%
Group 1 Control 0.0 mg/kg	C90721M	1	NA	0.9275	0.8640	1	37.2	R032700027	0.0298			Unknown	NA	1	0.00	R032700027	<LOQ (0.00493 ug/mL)		
	C90735M	1	NA	0.9275	0.8640	1	49.2	R032700028	0.0394			Unknown	NA	1	0.00	R032700028	<LOQ (0.00493 ug/mL)		
	C90736M	1	NA	0.9275	0.8640	1	44.6	R032700029	0.0357			Unknown	NA	1	0.00	R032700029	<LOQ (0.00493 ug/mL)		
	C90738M	1	NA	0.9275	0.8640	1	40.0	R032700030	0.0320			Unknown	NA	1	0.00	R032700030	<LOQ (0.00493 ug/mL)		
	C90739M	1	NA	0.9275	0.8640	1	68.5	R032700031	0.0549			Unknown	NA	1	0.00	R032700031	<LOQ (0.00493 ug/mL)		
	C90747M	1	NA	0.9275	0.8640	1	72.6	R032700035	0.0582			Unknown	NA	1	0.00	R032700035	<LOQ (0.00493 ug/mL)		
	C90751M	1	NA	0.9275	0.8640	1	55.9	R032700036	0.0448			Unknown	NA	1	0.00	R032700036	<LOQ (0.00493 ug/mL)		
	C90752M	1	NA	0.9275	0.8640	1	57.6	R032700037	0.0462			Unknown	NA	1	0.00	R032700037	<LOQ (0.00493 ug/mL)		
	C90755M	1	NA	0.9275	0.8640	1	50.3	R032700038	0.0403		22.9	Unknown	NA	1	0.00	R032700038	<LOQ (0.00493 ug/mL)		NA
	C90766M	1	NA	0.9275	0.8640	1	42.2	R032700039	0.0338	0.0415	0.00951	Unknown	NA	1	0.00	R032700039	<LOQ (0.00493 ug/mL)	<LOQ	NA
	C91127F	1	NA	0.9275	0.8640	1	113	R032700043	0.0908			Unknown	NA	1	0.00	R032700043	<LOQ (0.00493 ug/mL)		
	C91128F	1	NA	0.9275	0.8640	1	142	R032700044	0.114			Unknown	NA	1	0.00	R032700044	<LOQ (0.00493 ug/mL)		
	C91133F	1	NA	0.9275	0.8640	1	120	R032700045	0.0966			Unknown	NA	1	0.00	R032700045	<LOQ (0.00493 ug/mL)		
	C91137F	1	NA	0.9275	0.8640	1	99.7	R032700046	0.0799			Unknown	NA	1	0.00	R032700046	<LOQ (0.00493 ug/mL)		
	C91144F	1	NA	0.9275	0.8640	1	79.4	R032700047	0.0636			Unknown	NA	1	0.00	R032700047	<LOQ (0.00493 ug/mL)		
	C91153F	1	NA	0.9275	0.8640	1	98.1	R032700051	0.0786			Unknown	NA	1	0.00	R032700051	<LOQ (0.00493 ug/mL)		
	C91154F	1	NA	0.9275	0.8640	1	88.1	R032700052	0.0706			Unknown	NA	1	0.00	R032700052	<LOQ (0.00493 ug/mL)		
	C91158F	1	NA	0.9275	0.8640	1	87.9	R032700053	0.0705			Unknown	NA	1	0.00	R032700053	<LOQ (0.00493 ug/mL)		
	C91161F	1	NA	0.9275	0.8640	1	68.3	R032700054	0.0547		21.7	Unknown	NA	1	0.00	R032700054	<LOQ (0.00493 ug/mL)		NA
	C91183F	1	NA	0.9275	0.8640	1	92.4	R032700055	0.0740	0.0793	0.0172	Unknown	NA	1	0.00	R032700055	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90795M	1	NA	0.9275	0.8640	10	274	R032800052	2.19			Unknown	NA	1	12.5	R032700059	0.0125		
	C90799M	1	NA	0.9275	0.8640	10	380	R032800053	3.05			Unknown	NA	1	17.4	R032700060	0.0174		
	C90802M	1	NA	0.9275	0.8640	10	165	R032800054	1.32			Unknown	NA	1	5.53	R032700061	0.00553		
	C90803M	1	NA	0.9275	0.8640	10	500	R032800055	4.01			Unknown	NA	1	20.4	R032700062	0.0204		
	C90805M	1	NA	0.9275	0.8640	10	211	R032800056	1.69			Unknown	NA	1	12.6	R032700063	0.0126		
	C90806M	1	NA	0.9275	0.8640	10	408	R032800059	3.27			Unknown	NA	1	14.5	R032700067	0.0145		
	C90824M	1	NA	0.9275	0.8640	10	279	R032800060	2.23			Unknown	NA	1	15.8	R032700068	0.0158		
	C90825M	1	NA	0.9275	0.8640	10	366	R032800061	2.93			Unknown	NA	1	17.6	R032700069	0.0176		
	C90839M	1	NA	0.9275	0.8640	10	343	R032800062	2.75		33.0	Unknown	NA	1	11.9	R032700070	0.0119		65.4
	C90840M	1	NA	0.9275	0.8640	10	505	R032800063	4.05	2.75	0.906	Unknown	NA	1	48.5	R032700071	0.0485	0.0177	0.0116
	C91196F	1	NA	0.9275	0.8640	10	442	R032800066	3.54			Unknown	NA	1	34.0	R032700075	0.0340		
	C91207F	1	NA	0.9275	0.8640	10	677	R032800067	5.42			Unknown	NA	1	35.1	R032700076	0.0351		
	C91210F	1	NA	0.9275	0.8640	10	528	R032800068	4.23			Unknown	NA	1	37.9	R032700077	0.0379		
	C91211F	1	NA	0.9275	0.8640	10	430	R032800069	3.44			Unknown	NA	1	23.4	R032700078	0.0234		
	C91214F	1	NA	0.9275	0.8640	10	554	R032800070	4.44			Unknown	NA	1	29.0	R032700079	0.0290		
	C91221F	1	NA	0.9275	0.8640	10	600	R032800073	4.81			Unknown	NA	1	29.8	R032700083	0.0298		
	C91224F	1	NA	0.9275	0.8640	10	704	R032800074	5.64			Unknown	NA	1	35.8	R032700084	0.0358		
	C91235F	1	NA	0.9275	0.8640	10	722	R032800075	5.78			Unknown	NA	1	28.4	R032700085	0.0284		
	C91243F	1	NA	0.9275	0.8640	10	603	R032800076	4.83		19.7	Unknown	NA	1	43.8	R032700086	0.0438		18.0
	C91244F	1	NA	0.9275	0.8640	10	787	R032800077	6.31	4.85	0.954	Unknown	NA	1	39.6	R032700087	0.0396	0.0337	0.00608

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
new LOQs are 0.00394 & 0.00781 ug/mL. LAC 02/19/01

Date Entered/By: 04/11/00, 04/12/00, 04/17/00, 5/4/00, 5/23/00, 08/15/00, 09/01/00 CSH/LAC 4/3/01 mmh
Date Verified/ By: 02/22/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799 & Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/23/00, 03/28/00 SAL
Dates of Analysis/Analyst:	03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 5/17/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst:	03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 5/18/00, 07/25/00, 08/18/00 IAS/HOJ/MMH
Sample Data	Box 00-021

WEEK 27 RAT SERA lot T-7121.1 lot Unknown/SD013

Group Dose	Sample #	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Dilution Factor	PFOSAA Conc. ng/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	EtFOSE-OH Purity Correction Factor	Surrogate Verified	EtFOSE-OH Dilution Factor	EtFOSE-OH Conc. ng/mL	Filename	Concentration of EtFOSE-OH ug/mL or % Rec	Mean EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	RBS02230-H2O Blk-5	Unknown	NA	1	0.00	R032700006	<LOQ (0.0493 ug/mL)			Unknown	NA	1	0.00	R032700006	<LOQ (0.00977 ug/mL)		
	RBS02230-H2O Blk-6	Unknown	NA	1	0.00	D040300006	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	A081700017	<LOQ (0.00977 ug/mL)	<LOQ	NA
Matrix Blk	RBS02230-Sera Blk-5	Unknown	NA	1	0.00	R032700007	<LOQ (0.0493 ug/mL)			Unknown	NA	1	0.00	R032700007	<LOQ (0.00977 ug/mL)		
	RBS02230-Sera Blk-6	Unknown	NA	1	0.00	D040300007	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	A081700018	<LOQ (0.00977 ug/mL)	<LOQ	NA
Matrix Blk	RTS02230-Sera Blk-5	Unknown	NA	1	0.00	R032700008	<LOQ (0.0493 ug/mL)			Unknown	NA	1	0.00	R032700008	<LOQ (0.00977 ug/mL)		
	RTS02230-Sera Blk-6	Unknown	NA	1	0.00	D040300008	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	A081700019	<LOQ (0.00977 ug/mL)	<LOQ	NA
QC-250 ng/mL	RTS02230-MS-5-1	NA	NA	1	321	R032700020	129%			NA	NA	1	43.9	R032700020	18%		
	RTS02230-MS-5-1	NA	NA	NA	NA	NA	NA			NA	NA	1	123	R032800018	50% 2nd		
	RTS02230-MS-5-2	NA	NA	1	304	R032700021	122%			NA	NA	1	82.4	R032700021	33%		
	RTS02230-MS-5-2	NA	NA	NA	NA	NA	NA	126%	6%	NA	NA	1	77.2	R032800019	31% 2nd	25%	46%
	RTS02230-MS-5-3	NA	NA	1	241	R032700022	97%			NA	NA	1	64.4	R032700022	26%		
	RTS02230-MS-5-3	NA	NA	NA	NA	NA	NA			NA	NA	1	80.4	R032800020	32% 2nd		
	RTS02230-MS-5-4	NA	NA	1	285	R032700023	115%			NA	NA	1	56.8	R032700023	23%		
	RTS02230-MS-5-4	NA	NA	NA	NA	NA	NA	106%	17%	NA	NA	1	71.7	R032800021	29% 2nd	27%	11%
Group 1 Control 0.0 mg/kg	C90721M	Unknown	NA	1	0.00	R051700019	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700027	<LOQ (0.00977 ug/mL)		
	C90735M	Unknown	NA	1	0.00	R051700020	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700028	<LOQ (0.00977 ug/mL)		
	C90736M	Unknown	NA	1	0.50	R051700021	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700029	<LOQ (0.00977 ug/mL)		
	C90738M	Unknown	NA	1	0.28	R051700022	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700030	<LOQ (0.00977 ug/mL)		
	C90739M	Unknown	NA	1	0.00	R051700023	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700031	<LOQ (0.00977 ug/mL)		
	C90747M	Unknown	NA	1	2.10	R051700028	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700035	<LOQ (0.00977 ug/mL)		
	C90751M	Unknown	NA	1	4.49	R051700029	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700036	<LOQ (0.00977 ug/mL)		
	C90752M	Unknown	NA	1	0.00	R051700030	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700037	<LOQ (0.00977 ug/mL)		
	C90755M	Unknown	NA	1	3.04	R051700031	<LOQ (0.0248 ug/mL)		NA	Unknown	NA	1	0.00	R032700038	<LOQ (0.00977 ug/mL)		NA
	C90766M	Unknown	NA	1	0.47	R051700032	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	R032700039	<LOQ (0.00977 ug/mL)	<LOQ	NA
	C91127F	Unknown	NA	1	0.00	R051700037	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700043	<LOQ (0.00977 ug/mL)		
	C91128F	Unknown	NA	1	8.07	R051700038	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700044	<LOQ (0.00977 ug/mL)		
	C91133F	Unknown	NA	1	0.00	R051700039	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700045	<LOQ (0.00977 ug/mL)		
	C91137F	Unknown	NA	1	0.05	R051700040	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700046	<LOQ (0.00977 ug/mL)		
	C91144F	Unknown	NA	1	0.00	R051700041	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700047	<LOQ (0.00977 ug/mL)		
	C91153F	Unknown	NA	1	0.00	R051700046	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700051	<LOQ (0.00977 ug/mL)		
	C91154F	Unknown	NA	1	0.00	R051700047	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700052	<LOQ (0.00977 ug/mL)		
	C91158F	Unknown	NA	1	0.00	R051700048	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700053	<LOQ (0.00977 ug/mL)		
	C91161F	Unknown	NA	1	0.00	R051700049	<LOQ (0.0248 ug/mL)		NA	Unknown	NA	1	0.00	R032700054	<LOQ (0.00977 ug/mL)		NA
	C91183F	Unknown	NA	1	0.00	R051700050	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	R032700055	<LOQ (0.00977 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90795M	Unknown	NA	1	270	R032700059	0.270			Unknown	NA	1	0.00	D040600043	<LOQ (0.0248 ug/mL)		
	C90799M	Unknown	NA	1	455	R032700060	0.455			Unknown	NA	1	0.00	D040600042	<LOQ (0.0248 ug/mL)		
	C90802M	Unknown	NA	1	237	R032700061	0.237			Unknown	NA	1	0.00	D040600044	<LOQ (0.0248 ug/mL)		
	C90803M	Unknown	NA	1	713	R032700062	0.713			Unknown	NA	1	0.00	D040600045	<LOQ (0.0248 ug/mL)		
	C90805M	Unknown	NA	1	356	R032700063	0.356			Unknown	NA	1	0.00	D040600046	<LOQ (0.0248 ug/mL)		
	C90806M	Unknown	NA	1	369	R032700067	0.369			Unknown	NA	1	0.00	D040600049	<LOQ (0.0248 ug/mL)		
	C90824M	Unknown	NA	1	410	R032700068	0.410			Unknown	NA	1	0.00	D040600050	<LOQ (0.0248 ug/mL)		
	C90825M	Unknown	NA	1	646	R032700069	0.646			Unknown	NA	1	0.00	D040600051	<LOQ (0.0248 ug/mL)		
	C90839M	Unknown	NA	1	512	R032700070	0.512		78.9	Unknown	NA	1	0.00	D040600052	<LOQ (0.0248 ug/mL)		NA
	C90840M	Unknown	NA	10	180	R032800063	1.80	0.577	0.455	Unknown	NA	1	0.00	D040600053	<LOQ (0.0248 ug/mL)	<LOQ	NA
	C91196F	Unknown	NA	1	578	R032700075	0.578			Unknown	NA	1	0.00	D040600056	<LOQ (0.0248 ug/mL)		
	C91207F	Unknown	NA	1	641	R032700076	0.641			Unknown	NA	1	0.00	D040600057	<LOQ (0.0248 ug/mL)		
	C91210F	Unknown	NA	1	663	R032700077	0.663			Unknown	NA	1	0.00	D040600058	<LOQ (0.0248 ug/mL)		
	C91211F	Unknown	NA	1	699	R032700078	0.699			Unknown	NA	1	0.00	D040600059	<LOQ (0.0248 ug/mL)		
	C91214F	Unknown	NA	10	165	R032800070	1.65			Unknown	NA	1	0.00	D040600060	<LOQ (0.0248 ug/mL)		
	C91221F	Unknown	NA	1	684	R032700083	0.684			Unknown	NA	1	0.00	D040600063	<LOQ (0.0248 ug/mL)		
	C91224F	Unknown	NA	1	685	D040600064	0.685			Unknown	NA	1	0.00	D040600064	<LOQ (0.0248 ug/mL)		
	C91235F	Unknown	NA	1	655	R032700085	0.655			Unknown	NA	1	0.00	D040600065	<LOQ (0.0248 ug/mL)		
	C91243F	Unknown	NA	1	702	R032700086	0.702		40.3	Unknown	NA	1	0.00	D040600066	<LOQ (0.0248 ug/mL)		NA
	C91244F	Unknown	NA	1	766	D040600067	0.766	0.772	0.311	Unknown	NA	1	0.00	D040600067	<LOQ (0.0248 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
now LOQs are 0.00394 & 0.00781 ug/mL LAC 02/19/01

Date Entered/By: 04/11/00, 04/12/00, 04/17/00, 5/4/00, 5/23/00, 08/15/00, 09/01/00 CSH/LAC 4/3/01 mmh
Date Verified/ By: 02/22/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799 & Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/23/00, 03/28/00 SAL
Dates of Analysis/Analyst:	03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 5/17/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst:	03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 5/18/00, 07/25/00, 08/18/00 IAS/HOJ/MMH
Sample Data	Box 00-021

WEEK 27 RAT SERA — lot NB113047-80 — lot 529

Group Dose	Sample #	M556 Purity Correction Factor	Surrogate Verified	M556 Dilution Factor	M556 Conc. ng/mL	Filename	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Dilution Factor	PFOSEA Conc. ng/mL	Filename	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	RBS02230-H2O Blk-5	Unknown	NA	1	0.00	R032700006	<LOQ (0.00492 ug/mL)			Unknown	NA	1	0.00	R032700006	<LOQ (0.0247 ug/mL)		
	RBS02230-H2O Blk-6	Unknown	NA	1	0.00	D040300006	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D040300006	<LOQ (0.0248 ug/mL)	<LOQ	NA
Matrix Blk	RBS02230-Sera Blk-5	Unknown	NA	1	0.00	R032700007	<LOQ (0.00492 ug/mL)			Unknown	NA	1	0.00	R032700007	<LOQ (0.0247 ug/mL)		
	RBS02230-Sera Blk-6	Unknown	NA	1	0.00	D040300007	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D040300007	<LOQ (0.0248 ug/mL)	<LOQ	NA
Matrix Blk	RTS02230-Sera Blk-5	Unknown	NA	1	0.00	R032700008	<LOQ (0.00492 ug/mL)			Unknown	NA	1	0.00	R032700008	<LOQ (0.0247 ug/mL)		
	RTS02230-Sera Blk-6	Unknown	NA	1	0.00	D040300008	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D040300008	<LOQ (0.0248 ug/mL)	<LOQ	NA
QC-250 ng/mL	RTS02230-MS-5-1	NA	NA	1	375	R032700020	151%			NA	NA	1	164	R032700020	67%		
	RTS02230-MS-5-1	NA	NA	1	272	R032800018	110% 2nd			NA	NA	1	265	R032800018	107% 2nd		
	RTS02230-MS-5-2	NA	NA	1	372	R032700021	150%			NA	NA	1	124	R032700021	50%		
	RTS02230-MS-5-2	NA	NA	1	262	R032800019	106% 2nd	150%	1%	NA	NA	1	204	R032800019	83% 2nd	58%	28%
	RTS02230-MS-5-3	NA	NA	1	323	R032700022	130%			NA	NA	1	91.0	R032700022	37%		
	RTS02230-MS-5-3	NA	NA	1	222	R032800020	89% 2nd			NA	NA	1	143	R032800020	58% 2nd		
	RTS02230-MS-5-4	NA	NA	1	350	R032700023	141%			NA	NA	1	116	R032700023	47%		
	RTS02230-MS-5-4	NA	NA	1	243	R032800021	98% 2nd	136%	8%	NA	NA	1	141	R032800021	57% 2nd	42%	24%
Group 1 Control 0.0 mg/kg	C90721M	Unknown	NA	1	15.9	D071900018	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700027	<LOQ (0.0247 ug/mL)		
	C90735M	Unknown	NA	1	0.00	D071900019	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700028	<LOQ (0.0247 ug/mL)		
	C90736M	Unknown	NA	1	0.00	D071900020	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700029	<LOQ (0.0247 ug/mL)		
	C90738M	Unknown	NA	1	0.00	D071900024	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700030	<LOQ (0.0247 ug/mL)		
	C90739M	Unknown	NA	1	0.00	R051700023	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700031	<LOQ (0.0247 ug/mL)		
	C90747M	Unknown	NA	1	7.34	R051700028	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700035	<LOQ (0.0247 ug/mL)		
	C90751M	Unknown	NA	1	0.330	R051700029	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700036	<LOQ (0.0247 ug/mL)		
	C90752M	Unknown	NA	1	0.00	R051700030	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700037	<LOQ (0.0247 ug/mL)		
	C90755M	Unknown	NA	1	0.00	R051700031	<LOQ (0.0248 ug/mL)		NA	Unknown	NA	1	0.00	R032700038	<LOQ (0.0247 ug/mL)		NA
	C90766M	Unknown	NA	1	0.00	R051700032	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	R032700039	<LOQ (0.0247 ug/mL)	<LOQ	NA
	C91127F	Unknown	NA	1	0.0900	R051700037	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700043	<LOQ (0.0247 ug/mL)		
	C91128F	Unknown	NA	1	0.00	R051700038	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700044	<LOQ (0.0247 ug/mL)		
	C91133F	Unknown	NA	1	0.00	R051700039	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700045	<LOQ (0.0247 ug/mL)		
	C91137F	Unknown	NA	1	0.00	R051700040	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700046	<LOQ (0.0247 ug/mL)		
	C91144F	Unknown	NA	1	0.00	R051700041	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700047	<LOQ (0.0247 ug/mL)		
	C91153F	Unknown	NA	1	2.95	R051700046	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700051	<LOQ (0.0247 ug/mL)		
	C91154F	Unknown	NA	1	0.00	R051700047	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700052	<LOQ (0.0247 ug/mL)		
	C91158F	Unknown	NA	1	0.00	R051700048	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	R032700053	<LOQ (0.0247 ug/mL)		
	C91161F	Unknown	NA	1	0.00	R051700049	<LOQ (0.0248 ug/mL)		NA	Unknown	NA	1	0.00	R032700054	<LOQ (0.0247 ug/mL)		NA
	C91183F	Unknown	NA	1	0.00	R051700050	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	R032700055	<LOQ (0.0247 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90795M	Unknown	NA	1	218	D040500043	0.218			Unknown	NA	1	0.00	R032700059	<LOQ (0.0247 ug/mL)		
	C90799M	Unknown	NA	1	229	D040500042	0.229			Unknown	NA	1	0.00	R032700060	<LOQ (0.0247 ug/mL)		
	C90802M	Unknown	NA	1	202	D040500044	0.202			Unknown	NA	1	0.00	R032700061	<LOQ (0.0247 ug/mL)		
	C90803M	Unknown	NA	1	273	D040500045	0.273			Unknown	NA	1	0.00	R032700062	<LOQ (0.0247 ug/mL)		
	C90805M	Unknown	NA	1	214	D040500046	0.214			Unknown	NA	1	0.00	R032700063	<LOQ (0.0247 ug/mL)		
	C90806M	Unknown	NA	1	213	D040500049	0.213			Unknown	NA	1	0.00	D040600049	<LOQ (0.00492 ug/mL)		
	C90824M	Unknown	NA	1	315	D040500050	0.315			Unknown	NA	1	0.00	D040600050	<LOQ (0.00492 ug/mL)		
	C90825M	Unknown	NA	1	299	D040500051	0.299			Unknown	NA	1	0.00	D040600051	<LOQ (0.00492 ug/mL)		
	C90839M	Unknown	NA	1	306	D040500052	0.306		23.8	Unknown	NA	1	0.00	D040600052	<LOQ (0.00492 ug/mL)		NA
	C90840M	Unknown	NA	1	399	D040500053	0.399	0.267	0.0634	Unknown	NA	1	0.00	D040600053	<LOQ (0.00492 ug/mL)	<LOQ	NA
	C91196F	Unknown	NA	1	245	D040500056	0.245			Unknown	NA	1	0.00	D040600056	<LOQ (0.00492 ug/mL)		
	C91207F	Unknown	NA	1	307	D040500057	0.307			Unknown	NA	1	0.00	D040600057	<LOQ (0.00492 ug/mL)		
	C91210F	Unknown	NA	1	323	D040500058	0.323			Unknown	NA	1	0.00	D040600058	<LOQ (0.00492 ug/mL)		
	C91211F	Unknown	NA	1	223	D040500059	0.223			Unknown	NA	1	0.00	D040600059	<LOQ (0.00492 ug/mL)		
	C91214F	Unknown	NA	1	399	D040500060	0.399			Unknown	NA	1	0.00	D040600060	<LOQ (0.00492 ug/mL)		
	C91221F	Unknown	NA	1	352	D040500063	0.352			Unknown	NA	1	0.00	D040600063	<LOQ (0.00492 ug/mL)		
	C91224F	Unknown	NA	1	243	D040500064	0.243			Unknown	NA	1	0.00	D040600064	<LOQ (0.00492 ug/mL)		
	C91235F	Unknown	NA	1	187	D040500065	0.187			Unknown	NA	1	0.00	D040600065	<LOQ (0.00492 ug/mL)		
	C91243F	Unknown	NA	1	389	D040500066	0.389		24.6	Unknown	NA	1	0.00	D040600066	<LOQ (0.00492 ug/mL)		NA
	C91244F	Unknown	NA	1	363	D040500067	0.363	0.303	0.0744	Unknown	NA	1	0.00	D040600067	<LOQ (0.00492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
new LOQs are 0.00394 & 0.00781 ug/mL. LAC 02/19/01

Date Entered/By:	04/11/00, 04/12/00, 04/17/00, 5/4/00, 5/23/00, 08/15/00, 09/01/00 CSH/LAC 4/3/01 mmh
Date Verified/ By:	02/22/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799 & Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/23/00, 03/28/00 SAL
Dates of Analysis/Analyst:	03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 5/17/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst:	03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 5/18/00, 07/25/00, 08/18/00 IAS/HOJ/MMH

Sample Data

WEEK 27 RAT SERA

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of EtFOSE-OH ug/mL or % Rec	Mean EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	RBS02230-H2O Blk-5	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0493 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.00492 ug/mL)			<LOQ (0.0247 ug/mL)		
	RBS02230-H2O Blk-6	<LOQ (0.00781 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA
Matrix Blk	RBS02230-Sera Blk-5	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0493 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.00492 ug/mL)			<LOQ (0.0247 ug/mL)		
	RBS02230-Sera Blk-6	<LOQ (0.00781 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA
Matrix Blk	RTS02230-Sera Blk-5	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0493 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.00492 ug/mL)			<LOQ (0.0247 ug/mL)		
	RTS02230-Sera Blk-6	<LOQ (0.00781 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA
QC-250 ng/mL	RTS02230-MS-5-1	109%			104%			129%			18%			151%			67%		
	RTS02230-MS-5-1	NA			NA			NA			50% 2nd			110% 2nd			107% 2nd		
	RTS02230-MS-5-2	88%			98%			122%			33%			150%			50%		
	RTS02230-MS-5-2	NA	98%	21%	NA	101%	7%	NA	126%	6%	31% 2nd	25%	46%	106% 2nd	150%	1%	83% 2nd	58%	28%
	RTS02230-MS-5-3	67%			73%			97%			26%			130%			37%		
	RTS02230-MS-5-3	62% 2nd			NA			NA			32% 2nd			89% 2nd			58% 2nd		
	RTS02230-MS-5-4	74%			81%			115% #			23%			141%			47%		
	RTS02230-MS-5-4	NA	68%	10%	NA	77%	11%	NA #	106%	17%	29% 2nd	27%	11%	98% 2nd	136%	8%	57% 2nd	42%	24%
Group 1	C90721M	0.0298			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
Control	C90735M	0.0394			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
0.0 mg/kg	C90736M	0.0357			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C90738M	0.0320			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C90739M	0.0549			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C90747M	0.0582			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C90751M	0.0448			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C90752M	0.0462			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C90755M	0.0403		22.9	<LOQ (0.00493 ug/mL)		NA	<LOQ (0.0248 ug/mL)		NA	<LOQ (0.00977 ug/mL)		NA	<LOQ (0.0248 ug/mL)		NA	<LOQ (0.0247 ug/mL)		NA
	C90766M	0.0338	0.0415	0.00951	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.0247 ug/mL)	<LOQ	NA
	C91127F	0.0908			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C91128F	0.114			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C91133F	0.097			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C91137F	0.0799			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C91144F	0.0636			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C91153F	0.0786			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C91154F	0.0706			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C91158F	0.0705			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.0247 ug/mL)		
	C91161F	0.0547		21.7	<LOQ (0.00493 ug/mL)		NA	<LOQ (0.0248 ug/mL)		NA	<LOQ (0.00977 ug/mL)		NA	<LOQ (0.0248 ug/mL)		NA	<LOQ (0.0247 ug/mL)		NA
	C91183F	0.0740	0.0793	0.0172	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.0247 ug/mL)	<LOQ	NA
Group 2	C90795M	2.19			0.0125			0.270			<LOQ (0.0248 ug/mL)			0.218			<LOQ (0.0247 ug/mL)		
Low Dose	C90799M	3.05			0.0174			0.455			<LOQ (0.0248 ug/mL)			0.229			<LOQ (0.0247 ug/mL)		
3.0 mg/kg	C90802M	1.32			0.00553			0.237			<LOQ (0.0248 ug/mL)			0.202			<LOQ (0.0247 ug/mL)		
	C90803M	4.01			0.0204			0.713			<LOQ (0.0248 ug/mL)			0.273			<LOQ (0.0247 ug/mL)		
	C90805M	1.69			0.0126			0.356			<LOQ (0.0248 ug/mL)			0.214			<LOQ (0.0247 ug/mL)		
	C90806M	3.27			0.0145			0.369			<LOQ (0.0248 ug/mL)			0.213			<LOQ (0.00492 ug/mL)		
	C90824M	2.23			0.0158			0.410			<LOQ (0.0248 ug/mL)			0.315			<LOQ (0.00492 ug/mL)		
	C90825M	2.93			0.0176			0.646			<LOQ (0.0248 ug/mL)			0.299			<LOQ (0.00492 ug/mL)		
	C90839M	2.75		33.0	0.0119		65.4	0.512		78.9	<LOQ (0.0248 ug/mL)		NA	0.306		23.8	<LOQ (0.00492 ug/mL)		NA
	C90840M	4.05	2.75	0.906	0.0485	0.0177	0.0116	1.80	0.577	0.455	<LOQ (0.0248 ug/mL)	<LOQ	NA	0.399	0.267	0.0634	<LOQ (0.00492 ug/mL)	<LOQ	NA
	C91196F	3.54			0.0340			0.578			<LOQ (0.0248 ug/mL)			0.245			<LOQ (0.00492 ug/mL)		
	C91207F	5.42			0.0351			0.641			<LOQ (0.0248 ug/mL)			0.307			<LOQ (0.00492 ug/mL)		
	C91210F	4.23			0.0379			0.663			<LOQ (0.0248 ug/mL)			0.323			<LOQ (0.00492 ug/mL)		
	C91211F	3.44			0.0234			0.699			<LOQ (0.0248 ug/mL)			0.223			<LOQ (0.00492 ug/mL)		
	C91214F	4.44			0.0290			1.65			<LOQ (0.0248 ug/mL)			0.399			<LOQ (0.00492 ug/mL)		
	C91221F	4.81			0.0298			0.684			<LOQ (0.0248 ug/mL)			0.352			<LOQ (0.00492 ug/mL)		
	C91224F	5.64			0.0358			0.685			<LOQ (0.0248 ug/mL)			0.243			<LOQ (0.00492 ug/mL)		
	C91235F	5.78			0.0284			0.655			<LOQ (0.0248 ug/mL)			0.187			<LOQ (0.00492 ug/mL)		
	C91243F	4.83		19.7	0.0438		18.0	0.702		40.3	<LOQ (0.0248 ug/mL)		NA	0.389		24.6	<LOQ (0.00492 ug/mL)		NA
	C91244F	6.31	4.85	0.954	0.0396	0.0337	0.00608	0.766	0.772	0.311	<LOQ (0.0248 ug/mL)	<LOQ	NA	0.363	0.303	0.0744	<LOQ (0.00492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
new LOQs are 0.00394 & 0.00781 ug/mL. LAC 02/19/01

Date Entered/By:	04/11 00, 04/12 00, 04/17 00, 5/4/00, 5 23 00, 08 15 00, 09/01/00 CSH/LAC 4/3/01 mmh
Date Verified/ By:	02 22 01 KJH / 04/30/01 LAC
Purity Entered/Verified:	02 19 01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799 & Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/23/00, 03/28/00 SAL
Dates of Analysis/Analyst:	03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 05/19/00, 06/26/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst:	03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 05/23/00, 06/27/00, 07/25/00, 08/18/00 IAS/HOJ/MMH
Sample Data	**Box** 00-021

WEEK 27 RAT SERA

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Std Correction Factor	PFOS Purity Correction Factor (lot 171)	PFOS Dilution Factor	PFOS Conc. ug/mL	Filename	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor (lot L-15709)	Surrogate Verified	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90847M	1	NA	0.9275	0.8640	100	183	R051900019	14.7			Unknown	NA	1	215	A062600016	0.215		
Mid Dose	C90855M	1	NA	0.9275	0.8640	100	269	R051900020	21.6			Unknown	NA	1	302	A062600017	0.302		
30.0 mg/kg	C90858M	1	NA	0.9275	0.8640	100	268	R051900021	21.4			Unknown	NA	1	193	A062600018	0.193		
	C90859M	1	NA	0.9275	0.8640	100	305	R051900022	24.4			Unknown	NA	1	239	A062600019	0.239		
	C90864M	1	NA	0.9275	0.8640	100	268	R051900023	21.5			Unknown	NA	1	236	A062600020	0.236		
	C90865M	1	NA	0.9275	0.8640	100	271	R051900028	21.7			Unknown	NA	1	228	A062600024	0.228		
	C90871M	1	NA	0.9275	0.8640	100	281	R051900029	22.5			Unknown	NA	1	204	A062600025	0.204		
	C90872M	1	NA	0.9275	0.8640	100	235	R051900030	18.9			Unknown	NA	1	247	A062600026	0.247		
	C90882M	1	NA	0.9275	0.8640	100	243	R051900031	19.5		14.0	Unknown	NA	1	233	A062600027	0.233		16.3
	C90886M	1	NA	0.9275	0.8640	100	213	R051900032	17.0	20.3	2.85	Unknown	NA	1	163	A062600028	0.163	0.226	0.0368
	C91252F	1	NA	0.9275	0.8640	100	629	R051900037	50.4			Unknown	NA	1	441	A062600032	0.441		
	C91254F	1	NA	0.9275	0.8640	100	536	R051900038	42.9			Unknown	NA	1	446	A062600033	0.446		
	C91264F	1	NA	0.9275	0.8640	100	391	R051900039	31.4			Unknown	NA	1	433	A062600034	0.433		
	C91267F	1	NA	0.9275	0.8640	100	506	R051900040	40.5			Unknown	NA	1	516	A062600035	0.516		
	C91283F	1	NA	0.9275	0.8640	100	419	R051900041	33.5			Unknown	NA	1	631	A062600036	0.631		
	C91286F	1	NA	0.9275	0.8640	100	587	R051900046	47.0			Unknown	NA	1	490	A062600040	0.490		
	C91292F	1	NA	0.9275	0.8640	100	481	R051900047	38.5			Unknown	NA	1	557	A062600041	0.557		
	C91295F	1	NA	0.9275	0.8640	100	552	R051900048	44.2			Unknown	NA	1	706	A062600042	0.706		
	C91301F	1	NA	0.9275	0.8640	100	520	R051900049	41.7		18.6	Unknown	NA	1	491	A062600043	0.491		19.9
	C91303F	1	NA	0.9275	0.8640	100	329	R051900050	26.3	39.7	7.39	Unknown	NA	1	365	A062600044	0.365	0.508	0.101
Group 4	C90916M	1	NA	0.9275	0.8640	2000	60.7	R051900055	97.3			Unknown	NA	1	424	A062600048	0.424		
Mid-High Dose	C90931M	1	NA	0.9275	0.8640	2000	42.9	R051900056	68.7			Unknown	NA	1	524	A062600049	0.524		
100 mg/kg	C90937M	1	NA	0.9275	0.8640	2000	50.9	R051900057	81.6			Unknown	NA	1	382	A062600050	0.382		
	C90940M	1	NA	0.9275	0.8640	100	836	R032800027	67.0			Unknown	NA	1	476	A062600051	0.476		
	C90944M	1	NA	0.9275	0.8640	2000	52.3	R051900058	83.9			Unknown	NA	1	410	A062600052	0.410		
	C90945M	1	NA	0.9275	0.8640	2000	50.9	R051900059	81.5			Unknown	NA	1	269	A062600056	0.269		
	C90954M	1	NA	0.9275	0.8640	100	874	R032800032	70.0			Unknown	NA	1	442	A062600057	0.442		
	C90956M	1	NA	0.9275	0.8640	100	624	R032800033	50.0			Unknown	NA	1	292	A062600058	0.292		
	C90957M	1	NA	0.9275	0.8640	2000	63.3	A081700023	101		19.7	Unknown	NA	1	554	A062600059	0.554		21.6
	C90963M	1	NA	0.9275	0.8640	100	875	R032800035	70.1	77.2	15.2	Unknown	NA	1	402	A062600060	0.402	0.418	0.0903
	C91312F	1	NA	0.9275	0.8640	2000	122	D040300020	196			Unknown	NA	1	779	A062600064	0.779		
	C91323F	1	NA	0.9275	0.8640	2000	145	D040300021	232			Unknown	NA	1	863	A062600065	0.863		
	C91328F	1	NA	0.9275	0.8640	2000	84.8	D040300022	136			Unknown	NA	1	606	A062600066	0.606		
	C91339F	1	NA	0.9275	0.8640	2000	88.5	D040300023	142			Unknown	NA	1	954	A062600067	0.954		
	C91340F	1	NA	0.9275	0.8640	2000	73.6	D040300024	118			Unknown	NA	1	758	A062600068	0.758		
	C91351F	1	NA	0.9275	0.8640	2000	95.8	D040300027	154			Unknown	NA	1	963	A062600072	0.963		
	C91354F	1	NA	0.9275	0.8640	2000	138	D040300028	221			Unknown	NA	1	896	A062600073	0.896		
	C91357F	1	NA	0.9275	0.8640	2000	119	D040300029	190			Unknown	NA	1	558	A062600074	0.558		
	C91369F	1	NA	0.9275	0.8640	2000	99.8	D040300030	160		22.6	Unknown	NA	1	627	A062600075	0.627		38.6
	C91371F	1	NA	0.9275	0.8640	2000	91.1	D040300031	146	170	38.3	Unknown	NA	10	174	D071900017	1.74	0.875	0.338

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = $C_8F_{17}SO_2N(H)CH_2COO^-$
PFOSEA = Perfuorooctane sulfonyl ethylamide

Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
new LOQs are 0.00394 & 0.00781 ug/mL. LAC 02/19/01

Date Entered/By:	04/11/00, 04/12/00, 04/17/00, 06/02/00, 06/07/00, 07/11/00, 08/15/00, 09/01/00 CSH/LAC
Date Verified/ By:	02/22/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number: Davey 070799 & Ruby 100699
Instrument Software/Version: Masslynx 3 3 & 3.4
Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Dates of Extraction/Analyst: 02/23/00, 03/28/00 SAI.
Dates of Analysis/Analyst: 03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 05/19/00, 06/26/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst: 03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 05/23/00, 06/27/00, 07/25/00, 08/18/00 IAS/HOJ/MMH

Sample Data

WEEK 27 RAT SERA lot T-7121.1 lot Unknown/SD013

Group Dose	Sample #	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Dilution Factor	PFOSAA Conc. ug/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	EtFOSE-OH Purity Correction Factor	Surrogate Verified	EtFOSE-OH Dilution Factor	EtFOSE-OH Conc. ug/mL	Filename	Concentration of EtFOSE-OH ug/mL or % Rec	Mean EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90847M	Unknown	NA	10	230	R032800080	2.30			Unknown	NA	1	0.490	A062600016	<LOQ (0.0248 ug/mL)		
Mid Dose	C90855M	Unknown	NA	10	444	R032800081	4.44			Unknown	NA	1	1.00	A062600017	<LOQ (0.0248 ug/mL)		
30.0 mg/kg	C90858M	Unknown	NA	10	204	R032800082	2.04			Unknown	NA	1	0.00	A062600018	<LOQ (0.0248 ug/mL)		
	C90859M	Unknown	NA	10	256	R032800083	2.56			Unknown	NA	1	0.00	A062600019	<LOQ (0.0248 ug/mL)		
	C90864M	Unknown	NA	10	247	R032800084	2.47			Unknown	NA	1	0.00	A062600020	<LOQ (0.0248 ug/mL)		
	C90865M	Unknown	NA	10	212	R032800087	2.12			Unknown	NA	1	0.490	A062600024	<LOQ (0.0248 ug/mL)		
	C90871M	Unknown	NA	10	194	R032800088	1.94			Unknown	NA	1	0.400	A062600025	<LOQ (0.0248 ug/mL)		
	C90872M	Unknown	NA	10	153	R032800089	1.53			Unknown	NA	1	1.46	A062600026	<LOQ (0.0248 ug/mL)		
	C90882M	Unknown	NA	10	188	R032800090	1.88		37.4	Unknown	NA	1	2.41	A062600027	<LOQ (0.0248 ug/mL)		NA
	C90886M	Unknown	NA	10	139	R032800091	1.39	2.27	0.848	Unknown	NA	1	0.560	A062600028	<LOQ (0.0248 ug/mL)	<LOQ	NA
	C91252F	Unknown	NA	10	378	R032800094	3.78			Unknown	NA	1	0.00	A062600032	<LOQ (0.0248 ug/mL)		
	C91254F	Unknown	NA	10	305	R032800095	3.05			Unknown	NA	1	0.00	A062600033	<LOQ (0.0248 ug/mL)		
	C91264F	Unknown	NA	10	284	R032800096	2.84			Unknown	NA	1	0.00	A062600034	<LOQ (0.0248 ug/mL)		
	C91267F	Unknown	NA	10	326	R032800097	3.26			Unknown	NA	1	0.00	A062600035	<LOQ (0.0248 ug/mL)		
	C91283F	Unknown	NA	10	278	R032800098	2.78			Unknown	NA	1	4.10	A062600036	<LOQ (0.0248 ug/mL)		
	C91286F	Unknown	NA	10	606	R032800101	6.06			Unknown	NA	1	0.00	A062600040	<LOQ (0.0248 ug/mL)		
	C91292F	Unknown	NA	10	293	R032800102	2.93			Unknown	NA	1	5.02	A062600041	<LOQ (0.0248 ug/mL)		
	C91295F	Unknown	NA	10	424	R032800103	4.24			Unknown	NA	1	0.930	A062600042	<LOQ (0.0248 ug/mL)		
	C91301F	Unknown	NA	10	280	R032800104	2.80		29.9	Unknown	NA	1	0.330	A062600043	<LOQ (0.0248 ug/mL)		NA
	C91303F	Unknown	NA	10	286	R032800105	2.86	3.46	1.03	Unknown	NA	1	0.00	A062600044	<LOQ (0.0248 ug/mL)	<LOQ	NA
Group 4	C90916M	Unknown	NA	100	99.1	R032800024	9.91			Unknown	NA	1	6.69	A062600048	<LOQ (0.0248 ug/mL)		
Mid-High Dose	C90931M	Unknown	NA	100	70.5	R032800025	7.05			Unknown	NA	1	6.19	A062600049	<LOQ (0.0248 ug/mL)		
100 mg/kg	C90937M	Unknown	NA	100	75.0	R032800026	7.50			Unknown	NA	1	5.90	A062600050	<LOQ (0.0248 ug/mL)		
	C90940M	Unknown	NA	100	85.6	R032800027	8.56			Unknown	NA	1	6.38	A062600051	<LOQ (0.0248 ug/mL)		
	C90944M	Unknown	NA	100	67.1	R032800028	6.71			Unknown	NA	1	9.47	A062600052	<LOQ (0.0248 ug/mL)		
	C90945M	Unknown	NA	10	460	R032800045	4.60			Unknown	NA	1	4.20	A062600056	<LOQ (0.0248 ug/mL)		
	C90954M	Unknown	NA	10	503	R032800046	5.03			Unknown	NA	1	4.52	A062600057	<LOQ (0.0248 ug/mL)		
	C90956M	Unknown	NA	10	342	R032800047	3.42			Unknown	NA	1	6.76	A062600058	<LOQ (0.0248 ug/mL)		
	C90957M	Unknown	NA	10	480	R032800048	4.80		31.3	Unknown	NA	1	17.4	A062600059	<LOQ (0.0248 ug/mL)		NA
	C90963M	Unknown	NA	100	81.9	R032800035	8.19	6.58	2.06	Unknown	NA	1	8.79	A062600060	<LOQ (0.0248 ug/mL)	<LOQ	NA
	C91312F	Unknown	NA	100	96.4	D040300034	9.64			Unknown	NA	1	8.05	A062600064	<LOQ (0.0248 ug/mL)		
	C91323F	Unknown	NA	100	97.7	D040300035	9.77			Unknown	NA	1	14.7	A062600065	<LOQ (0.0248 ug/mL)		
	C91328F	Unknown	NA	100	63.0	D040300036	6.30			Unknown	NA	1	5.99	A062600066	<LOQ (0.0248 ug/mL)		
	C91339F	Unknown	NA	100	86.5	D040300037	8.65			Unknown	NA	1	13.4	A062600067	<LOQ (0.0248 ug/mL)		
	C91340F	Unknown	NA	100	53.5	D040300038	5.35			Unknown	NA	1	8.89	A062600068	<LOQ (0.0248 ug/mL)		
	C91351F	Unknown	NA	100	82.6	D040300041	8.26			Unknown	NA	1	25.6	A062600072	0.0256		
	C91354F	Unknown	NA	100	113	D040300042	11.3			Unknown	NA	1	11.6	A062600073	<LOQ (0.0248 ug/mL)		
	C91357F	Unknown	NA	100	55.5	D040300043	5.55			Unknown	NA	1	3.93	A062600074	<LOQ (0.0248 ug/mL)		
	C91369F	Unknown	NA	100	58.0	D040300044	5.80		26.5	Unknown	NA	1	9.06	A062600075	<LOQ (0.0248 ug/mL)		NA
	C91371F	Unknown	NA	100	98.0	D040300045	9.80	8.04	2.13	Unknown	NA	1	18.2	A062600076	<LOQ (0.0248 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N{(H)CH2COO}
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
new LOQs are 0.00394 & 0.00781 ug/mL. LAC 02/19/01

Date Entered/By: 04/11/00, 04/12/00, 04/17/00, 06/02/00, 06/07/00, 07/11/00, 08/15/00, 09/01/00 CSH/LAC
Date Verified/ By: 02/22/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799 & Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/23/00, 03/28/00 SAL
Dates of Analysis/Analyst:	03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 05/19/00, 06/26/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst:	03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 05/23/00, 06/27/00, 07/25/00, 08/18/00 IAS/HOJ/MMH

Sample Data

WEEK 27 RAT SERA lot NB113047-80 lot 529

Group Dose	Sample #	M556 Purity Correction Factor	Surrogate Verified	M556 Dilution Factor	M556 Conc. ng/mL	Filename	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Dilution Factor	PFOSEA Conc. ng/mL	Filename	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90847M	Unknown	NA	10	207	R051900083	2.07			Unknown	NA	1	0.00	A062600016	<LOQ (0.00975 ug/mL)		
Mid Dose	C90855M	Unknown	NA	10	205	R051900084	2.05			Unknown	NA	1	0.150	A062600017	<LOQ (0.00975 ug/mL)		
30.0 mg/kg	C90858M	Unknown	NA	10	211	R051900085	2.11			Unknown	NA	1	0.00	A062600018	<LOQ (0.00975 ug/mL)		
	C90859M	Unknown	NA	10	186	R051900086	1.86			Unknown	NA	1	0.140	A062600019	<LOQ (0.00975 ug/mL)		
	C90864M	Unknown	NA	10	182	R051900091	1.82			Unknown	NA	1	0.00	A062600020	<LOQ (0.00975 ug/mL)		
	C90865M	Unknown	NA	10	251	A081700024	2.51			Unknown	NA	1	0.00	A062600024	<LOQ (0.00975 ug/mL)		
	C90871M	Unknown	NA	10	167	R051900093	1.67			Unknown	NA	1	0.00	A062600025	<LOQ (0.00975 ug/mL)		
	C90872M	Unknown	NA	10	301	A081700025	3.01			Unknown	NA	1	0.00	A062600026	<LOQ (0.00975 ug/mL)		
	C90882M	Unknown	NA	10	149	R051900095	1.49		22.2	Unknown	NA	1	0.00	A062600027	<LOQ (0.00975 ug/mL)		NA
	C90886M	Unknown	NA	10	168	R051900100	1.68	2.03	0.449	Unknown	NA	1	0.00	A062600028	<LOQ (0.00975 ug/mL)	<LOQ	NA
	C91252F	Unknown	NA	10	385	A081700026	3.85			Unknown	NA	1	0.00	A062600032	<LOQ (0.00975 ug/mL)		
	C91254F	Unknown	NA	10	340	R051900102	3.40			Unknown	NA	1	0.00	A062600033	<LOQ (0.00975 ug/mL)		
	C91264F	Unknown	NA	10	331	R051900103	3.31			Unknown	NA	1	0.00	A062600034	<LOQ (0.00975 ug/mL)		
	C91267F	Unknown	NA	10	480	R051900104	4.80			Unknown	NA	1	0.00	A062600035	<LOQ (0.00975 ug/mL)		
	C91283F	Unknown	NA	10	398	R051900105	3.98			Unknown	NA	1	0.620	A062600036	<LOQ (0.00975 ug/mL)		
	C91286F	Unknown	NA	10	298	R051900110	2.98			Unknown	NA	1	0.00	A062600040	<LOQ (0.00975 ug/mL)		
	C91292F	Unknown	NA	10	550	R051900111	5.50			Unknown	NA	1	0.00	A062600041	<LOQ (0.00975 ug/mL)		
	C91295F	Unknown	NA	10	432	R051900112	4.32			Unknown	NA	1	0.00	A062600042	<LOQ (0.00975 ug/mL)		
	C91301F	Unknown	NA	10	428	R051900113	4.28		20.1	Unknown	NA	1	0.00	A062600043	<LOQ (0.00975 ug/mL)		NA
	C91303F	Unknown	NA	10	314	R051900118	3.14	3.96	0.796	Unknown	NA	1	0.00	A062600044	<LOQ (0.00975 ug/mL)	<LOQ	NA
Group 4	C90916M	Unknown	NA	100	71.8	R051900065	7.18			Unknown	NA	1	0.510	A062600048	<LOQ (0.00975 ug/mL)		
Mid-High Dose	C90931M	Unknown	NA	100	84.7	R051900066	8.47			Unknown	NA	1	0.00	A062600049	<LOQ (0.00975 ug/mL)		
100 mg/kg	C90937M	Unknown	NA	100	81.7	R051900067	8.17			Unknown	NA	1	0.00	A062600050	<LOQ (0.00975 ug/mL)		
	C90940M	Unknown	NA	100	61.8	R051900068	6.18			Unknown	NA	1	0.00	A062600051	<LOQ (0.00975 ug/mL)		
	C90944M	Unknown	NA	100	69.8	R051900073	6.98			Unknown	NA	1	0.170	A062600052	<LOQ (0.00975 ug/mL)		
	C90945M	Unknown	NA	100	56.6	R051900074	5.66			Unknown	NA	1	0.00	A062600056	<LOQ (0.00975 ug/mL)		
	C90954M	Unknown	NA	100	73.8	R051900075	7.38			Unknown	NA	1	0.00	A062600057	<LOQ (0.00975 ug/mL)		
	C90956M	Unknown	NA	100	58.4	R051900076	5.84			Unknown	NA	1	0.00	A062600058	<LOQ (0.00975 ug/mL)		
	C90957M	Unknown	NA	100	61.4	R051900077	6.14		19.7	Unknown	NA	1	0.00	A062600059	<LOQ (0.00975 ug/mL)		NA
	C90963M	Unknown	NA	100	40.9	R051900082	4.09	6.61	1.30	Unknown	NA	1	0.00	A062600060	<LOQ (0.00975 ug/mL)	<LOQ	NA
	C91312F	Unknown	NA	100	41.3	D040300034	4.13			Unknown	NA	1	0.00	A062600064	<LOQ (0.00975 ug/mL)		
	C91323F	Unknown	NA	100	64.0	D040300035	6.40			Unknown	NA	1	0.350	A062600065	<LOQ (0.00975 ug/mL)		
	C91328F	Unknown	NA	100	44.1	D040300036	4.41			Unknown	NA	1	0.00	A062600066	<LOQ (0.00975 ug/mL)		
	C91339F	Unknown	NA	100	88.4	D040300037	8.84			Unknown	NA	1	0.490	A062600067	<LOQ (0.00975 ug/mL)		
	C91340F	Unknown	NA	100	51.4	D040300038	5.14			Unknown	NA	1	0.00	A062600068	<LOQ (0.00975 ug/mL)		
	C91351F	Unknown	NA	100	74.0	D040300041	7.40			Unknown	NA	1	0.00	A062600072	<LOQ (0.00975 ug/mL)		
	C91354F	Unknown	NA	100	77.2	D040300042	7.72			Unknown	NA	1	1.65	A062600073	<LOQ (0.00975 ug/mL)		
	C91357F	Unknown	NA	100	43.6	D040300043	4.36			Unknown	NA	1	0.380	A062600074	<LOQ (0.00975 ug/mL)		
	C91369F	Unknown	NA	100	49.6	D040300044	4.96		33.2	Unknown	NA	1	0.0100	A062600075	<LOQ (0.00975 ug/mL)		NA
	C91371F	Unknown	NA	100	102	D040300045	10.2	6.36	2.11	Unknown	NA	1	0.00	A062600076	<LOQ (0.00975 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N{(H)CH2COO}
PFOSEA = Perfuorooctane sulfonyl ethylamide

Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
new LOQs are 0.00394 & 0.00781 ug/mL. LAC 02/19/01

Date Entered/By:	04/11/00, 04/12/00, 04/17/00, 06/02/00, 06/07/00, 07/11/00, 08/15/00, 09/01/00 CSH/LAC
Date Verified/ By:	02/22/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799 & Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/23/00, 03/28/00 SAL
Dates of Analysis/Analyst:	03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 05/19/00, 06/26/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst:	03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 05/23/00, 06/27/00, 07/25/00, 08/18/00 IAS/HOJ/MMH

Sample Data

WEEK 27 RAT SERA

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90847M	14.7			0.215			2.30		
Mid Dose	C90855M	21.6			0.302			4.44		
30.0 mg/kg	C90858M	21.4			0.193			2.04		
	C90859M	24.4			0.239			2.56		
	C90864M	21.5			0.236			2.47		
	C90865M	21.7			0.228			2.12		
	C90871M	22.5			0.204			1.94		
	C90872M	18.9			0.247			1.53		
	C90882M	19.5		14.0	0.233		16.3	1.88		37.4
	C90886M	17.0	20.3	2.85	0.163	0.226	0.0368	1.39	2.27	0.848
	C91252F	50.4			0.441			3.78		
	C91254F	42.9			0.446			3.05		
	C91264F	31.4			0.433			2.84		
	C91267F	40.5			0.516			3.26		
	C91283F	33.5			0.631			2.78		
	C91286F	47.0			0.490			6.06		
	C91292F	38.5			0.557			2.93		
	C91295F	44.2			0.706			4.24		
	C91301F	41.7		18.6	0.491		19.9	2.80		29.9
	C91303F	26.3	39.7	7.39	0.365	0.508	0.101	2.86	3.46	1.03
Group 4	C90916M	97			0.424			9.91		
Mid-High Dose	C90931M	68.7			0.524			7.05		
100 mg/kg	C90937M	81.6			0.382			7.50		
	C90940M	67.0			0.476			8.56		
	C90944M	83.9			0.410			6.71		
	C90945M	81.5			0.269			4.60		
	C90954M	70.0			0.442			5.03		
	C90956M	50.0			0.292			3.42		
	C90957M	101		19.7	0.554		21.6	4.80		31.3
	C90963M	70.1	77.2	15.2	0.402	0.418	0.0903	8.19	6.58	2.06
	C91312F	196			0.779			9.64		
	C91323F	232			0.863			9.77		
	C91328F	136			0.606			6.30		
	C91339F	142			0.954			8.65		
	C91340F	118			0.758			5.35		
	C91351F	154			0.963			8.26		
	C91354F	221			0.896			11.3		
	C91357F	190			0.558			5.55		
	C91369F	160		22.6	0.627		38.6	5.80		26.5
	C91371F	146	170	38.3	1.74	0.875	0.338	9.80	8.04	2.13

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Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
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Date Verified/ By:	02/22/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799 & Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/23/00, 03/28/00 SAL
Dates of Analysis/Analyst:	03/07/00, 03/27/00, 03/28/00, 04/03/00, 04/05/00, 04/06/00, 05/19/00, 06/26/00, 07/19/00, 08/17/00 IAS/HOJ/MMH/CSH
Date of Data Reduction/Analyst:	03/09/00, 04/03/00, 04/04/00, 04/05/00, 04/07/00, 04/10/00, 05/23/00, 06/27/00, 07/25/00, 08/18/00 IAS/HOJ/MMH

Sample Data

WEEK 27 RAT SERA

Group Dose	Sample #	Concentration of EtFOSE-OH ug/mL or % Rec	Mean EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90847M	<LOQ (0.0248 ug/mL)			2.07			<LOQ (0.00975 ug/mL)		
Mid Dose	C90855M	<LOQ (0.0248 ug/mL)			2.05			<LOQ (0.00975 ug/mL)		
30.0 mg/kg	C90858M	<LOQ (0.0248 ug/mL)			2.11			<LOQ (0.00975 ug/mL)		
	C90859M	<LOQ (0.0248 ug/mL)			1.86			<LOQ (0.00975 ug/mL)		
	C90864M	<LOQ (0.0248 ug/mL)			1.82			<LOQ (0.00975 ug/mL)		
	C90865M	<LOQ (0.0248 ug/mL)			2.51			<LOQ (0.00975 ug/mL)		
	C90871M	<LOQ (0.0248 ug/mL)			1.67			<LOQ (0.00975 ug/mL)		
	C90872M	<LOQ (0.0248 ug/mL)			3.01			<LOQ (0.00975 ug/mL)		
	C90882M	<LOQ (0.0248 ug/mL)		NA	1.49		22.2	<LOQ (0.00975 ug/mL)		NA
	C90886M	<LOQ (0.0248 ug/mL)	<LOQ	NA	1.68	2.03	0.449	<LOQ (0.00975 ug/mL)	<LOQ	NA
	C91252F	<LOQ (0.0248 ug/mL)			3.85			<LOQ (0.00975 ug/mL)		
	C91254F	<LOQ (0.0248 ug/mL)			3.40			<LOQ (0.00975 ug/mL)		
	C91264F	<LOQ (0.0248 ug/mL)			3.31			<LOQ (0.00975 ug/mL)		
	C91267F	<LOQ (0.0248 ug/mL)			4.80			<LOQ (0.00975 ug/mL)		
	C91283F	<LOQ (0.0248 ug/mL)			3.98			<LOQ (0.00975 ug/mL)		
	C91286F	<LOQ (0.0248 ug/mL)			2.98			<LOQ (0.00975 ug/mL)		
	C91292F	<LOQ (0.0248 ug/mL)			5.50			<LOQ (0.00975 ug/mL)		
	C91295F	<LOQ (0.0248 ug/mL)			4.32			<LOQ (0.00975 ug/mL)		
	C91301F	<LOQ (0.0248 ug/mL)		NA	4.28		20.1	<LOQ (0.00975 ug/mL)		NA
	C91303F	<LOQ (0.0248 ug/mL)	<LOQ	NA	3.14	3.96	0.796	<LOQ (0.00975 ug/mL)	<LOQ	NA
Group 4	C90916M	<LOQ (0.0248 ug/mL)			7.18			<LOQ (0.00975 ug/mL)		
Mid-High Dose	C90931M	<LOQ (0.0248 ug/mL)			8.47			<LOQ (0.00975 ug/mL)		
100 mg/kg	C90937M	<LOQ (0.0248 ug/mL)			8.17			<LOQ (0.00975 ug/mL)		
	C90940M	<LOQ (0.0248 ug/mL)			6.18			<LOQ (0.00975 ug/mL)		
	C90944M	<LOQ (0.0248 ug/mL)			6.98			<LOQ (0.00975 ug/mL)		
	C90945M	<LOQ (0.0248 ug/mL)			5.66			<LOQ (0.00975 ug/mL)		
	C90954M	<LOQ (0.0248 ug/mL)			7.38			<LOQ (0.00975 ug/mL)		
	C90956M	<LOQ (0.0248 ug/mL)			5.84			<LOQ (0.00975 ug/mL)		
	C90957M	<LOQ (0.0248 ug/mL)		NA	6.14		19.7	<LOQ (0.00975 ug/mL)		NA
	C90963M	<LOQ (0.0248 ug/mL)	<LOQ	NA	4.09	6.61	1.30	<LOQ (0.00975 ug/mL)	<LOQ	NA
	C91312F	<LOQ (0.0248 ug/mL)			4.13			<LOQ (0.00975 ug/mL)		
	C91323F	<LOQ (0.0248 ug/mL)			6.40			<LOQ (0.00975 ug/mL)		
	C91328F	<LOQ (0.0248 ug/mL)			4.41			<LOQ (0.00975 ug/mL)		
	C91339F	<LOQ (0.0248 ug/mL)			8.84			<LOQ (0.00975 ug/mL)		
	C91340F	<LOQ (0.0248 ug/mL)			5.14			<LOQ (0.00975 ug/mL)		
	C91351F	0.0256			7.40			<LOQ (0.00975 ug/mL)		
	C91354F	<LOQ (0.0248 ug/mL)			7.72			<LOQ (0.00975 ug/mL)		
	C91357F	<LOQ (0.0248 ug/mL)			4.36			<LOQ (0.00975 ug/mL)		
	C91369F	<LOQ (0.0248 ug/mL)		NA	4.96		33.2	<LOQ (0.00975 ug/mL)		NA
	C91371F	<LOQ (0.0248 ug/mL)	<LOQ	NA	10.2	6.36	2.11	<LOQ (0.00975 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N{(H)CH2COO}
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQs (0.00492 & 0.00974 ug/mL) to include std correction factors
new LOQs are 0.00394 & 0.00781 ug/mL. LAC 02/19/01

Date Entered/By:	04/11/00, 04/12/00, 04/17/00, 06/02/00, 06/07/00, 07/11/00, 08/15/00, 09/01/00 CSH/LAC
Date Verified/ By:	02/22/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number: Amelia 062498, Soup 020199, Ruby 100699
Instrument Software/Version: Masslynx 3.3 & 3.4
Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Dates of Extraction/Analyst: 2/25/00 RWW
Dates of Analysis/Analyst: 03/22/00, 03/30/00, 04/10/00, 04/18/00, 04/20/00, 04/21/00, 05/01/00, 05/24/00, 08/18/00, 08/21/00, 08/23/00 CSH/MMH/IAS
Date of Data Reduction/Analyst: 04/12/00, 04/13/00, 04/19/00, 04/20/00, 04/21/00, 04/25/00, 05/03/00, 05/25/00, 08/21/00, 08/22/00, 08/25/00 IAS/MMH/HOJ

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor	Surrogate Verified	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
					lot 171							lot L-15709							
Method Blk	RBS02250-H2O Blk-1-1	1	NA	0.9275	0.8640	1	0.00	D082100007	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	R033000004	<LOQ (0.00493 ug/mL)		
	RBS02250-H2O Blk-1-2	1	NA	0.9275	0.8640	1	0.00	D042100018	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	D050100018	<LOQ (0.00493 ug/mL)		
	RBS02250-H2O Blk-1-3	1	NA	0.9275	0.8640	1	0.00	D042100019	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	D050100019	<LOQ (0.00493 ug/mL)		
	RBS02250-H2O Blk-1-4	1	NA	0.9275	0.8640	1	0.00	D042100020	< LOQ (0.00394 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100020	<LOQ (0.00493 ug/mL)	<LOQ	NA
Matrix Blk	RBS02250-Sera Blk-1-1	1	NA	0.9275	0.8640	1	0.00	D082100008	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	R033000005	<LOQ (0.00493 ug/mL)		
	RBS02250-Sera Blk-1-2	1	NA	0.9275	0.8640	1	0.00	D042100021	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	D050100021	<LOQ (0.00493 ug/mL)		
	RBS02250-Sera Blk-1-3	1	NA	0.9275	0.8640	1	0.00	D042100022	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	D050100022	<LOQ (0.00493 ug/mL)		
	RBS02250-Sera Blk-1-4	1	NA	0.9275	0.8640	1	0.00	D042100026	< LOQ (0.00394 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100023	<LOQ (0.00493 ug/mL)	<LOQ	NA
Matrix Blk	RTS02250-Sera Blk-1-1	1	NA	0.9275	0.8640	1	0.00	D082100009	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	R033000006	<LOQ (0.00493 ug/mL)		
	RTS02250-Sera Blk-1-2	1	NA	0.9275	0.8640	1	0.00	D042100027	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	D050100024	<LOQ (0.00493 ug/mL)		
	RTS02250-Sera Blk-1-3	1	NA	0.9275	0.8640	1	0.00	D042100028	< LOQ (0.00394 ug/mL)			Unknown	NA	1	0.00	D050100025	<LOQ (0.00493 ug/mL)		
	RTS02250-Sera Blk-1-4	1	NA	0.9275	0.8640	1	0.00	D042100029	< LOQ (0.00394 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100026	<LOQ (0.00493 ug/mL)	<LOQ	NA
QC-250 ng/mL	RTS02250-MS-1-1	1	NA	NA	NA	1	186	R033000018	75%			NA	NA	1	206	R033000018	83%		
	RTS02250-MS-1-2	1	NA	NA	NA	1	188	R033000019	76%		6%	NA	NA	1	216	R033000019	87%		3%
	RTS02250-MS-1-3	1	NA	NA	NA	1	168	R033000020	68%	73%	4%	NA	NA	1	214	R033000020	86%	85%	2%
	RTS02250-MS-1-1	1	NA	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA		
	RTS02250-MS-1-2	1	NA	NA	NA	NA	NA	NA	NA		NA	NA	NA	NA	NA	NA	NA		NA
	RTS02250-MS-1-3	1	NA	NA	NA	1	194	D050100035	78% 2nd	78%	NA	NA	NA	NA	NA	NA	NA	NA	NA
	RTS02250-MSD-2-1	1	NA	NA	NA	1	240	D03300082	97%			NA	NA	1	232	D03300082	94%		
	RTS02250-MSD-2-2	1	NA	NA	NA	1	269	D03300083	109%		7%	NA	NA	1	253	D03300083	102%		8%
	RTS02250-MSD-2-3	1	NA	NA	NA	1	278	D03300084	112%	106%	8%	NA	NA	1	275	D03300084	111%	102%	9%
	RTS02250-MSD-2-1	1	NA	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA		
	RTS02250-MSD-2-2	1	NA	NA	NA	NA	NA	NA	NA		NA	NA	NA	NA	NA	NA	NA		NA
	RTS02250-MSD-2-3	1	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 1 Control 0.0 mg/kg	C90712M	1	NA	0.9275	0.8640	1	12.6	D050100042	0.0101			Unknown	NA	1	1.49	R033000023	<LOQ (0.00493 ug/mL)		
	C90714M	1	NA	0.9275	0.8640	1	36.0	R033000024	0.0288			Unknown	NA	1	1.79	R033000024	<LOQ (0.00493 ug/mL)		
	C90723M	1	NA	0.9275	0.8640	1	15.1	D050100044	0.0121			Unknown	NA	1	1.45	R033000025	<LOQ (0.00493 ug/mL)		
	C90727M	1	NA	0.9275	0.8640	1	1.57	D082100020	< LOQ (0.00394 ug/mL)			Unknown	NA	1	1.19	R033000026	<LOQ (0.00493 ug/mL)		
	C90730M	1	NA	0.9275	0.8640	1	8.78	D082100021	0.00704			Unknown	NA	1	1.37	R033000027	<LOQ (0.00493 ug/mL)		
	C90751M	1	NA	0.9275	0.8640	1	11.0	D050100050	0.0088			Unknown	NA	1	1.49	R033000030	<LOQ (0.00493 ug/mL)		
	C90752M	1	NA	0.9275	0.8640	1	59.6	R033000031	0.0477			Unknown	NA	1	1.99	R033000031	<LOQ (0.00493 ug/mL)		
	C90754M	1	NA	0.9275	0.8640	1	40.6	R033000032	0.0325			Unknown	NA	1	1.36	R033000032	<LOQ (0.00493 ug/mL)		
	C90755M	1	Confirmed High	0.9275	0.8640	1	17.0	D050100053	0.0136			Unknown	Confirmed High	1	0.00	R033000033	<LOQ (0.00493 ug/mL)		
	C90758M	1	NA	0.9275	0.8640	1	13.6	D050100054	0.0109			Unknown	NA	1	1.49	R033000034	<LOQ (0.00493 ug/mL)		
	C90759M	1	Confirmed High	0.9275	0.8640	1	38.4	R033000037	0.0307			Unknown	Confirmed High	1	0.960	R041000040	<LOQ (0.00493 ug/mL)		
	C90764M	1	Confirmed High	0.9275	0.8640	1	24.1	D050100059	0.0193			Unknown	Confirmed High	1	0.000	R041000041	<LOQ (0.00493 ug/mL)		
	C90766M	1	Confirmed High	0.9275	0.8640	1	8.20	D082100022	0.00657			Unknown	Confirmed High	1	0.870	R041000042	<LOQ (0.00493 ug/mL)		
	C90767M	1	NA	0.9275	0.8640	1	18.5	D050100061	0.0148		70.3	Unknown	NA	1	0.00	R041000043	<LOQ (0.00493 ug/mL)		NA
	C90774M	1	NA	0.9275	0.8640	1	49.1	R033000041	0.0393	0.0191	0.0134	Unknown	NA	1	1.04	R041000044	<LOQ (0.00493 ug/mL)	<LOQ	NA
	C91121F	1	NA	0.9275	0.8640	1	127	R033000044	0.101			Unknown	NA	1	0.880	R041000048	<LOQ (0.00493 ug/mL)		
	C91122F	1	Confirmed High	0.9275	0.8640	1	173	R033000045	0.138			Unknown	Confirmed High	1	1.04	R041000049	<LOQ (0.00493 ug/mL)		
	C91123F	1	Confirmed High	0.9275	0.8640	1	73.7	R033000046	0.0590			Unknown	Confirmed High	1	0.830	R041000050	<LOQ (0.00493 ug/mL)		
	C91124F	1	Confirmed High	0.9275	0.8640	1	126	R033000047	0.101			Unknown	Confirmed High	1	1.15	R041000051	<LOQ (0.00493 ug/mL)		
	C91125F	1	Confirmed High	0.9275	0.8640	1	75.7	R033000048	0.0606			Unknown	Confirmed High	1	1.010	R041000052	<LOQ (0.00493 ug/mL)		
	C91131F	1	NA	0.9275	0.8640	1	98.4	R033000051	0.0788			Unknown	NA	1	0.950	R041000056	<LOQ (0.00493 ug/mL)		
	C91132F	1	NA	0.9275	0.8640	1	31.4	R033000052	0.0251			Unknown	NA	1	0.970	R041000057	<LOQ (0.00493 ug/mL)		
	C91133F	1	Confirmed High	0.9275	0.8640	1	154	R033000053	0.123			Unknown	Confirmed High	1	1.10	R041000058	<LOQ (0.00493 ug/mL)		
	C91152F	1	Confirmed High	0.9275	0.8640	1	168	R033000054	0.134			Unknown	Confirmed High	1	1.18	R041000059	<LOQ (0.00493 ug/mL)		
	C91153F	1	Confirmed High	0.9275	0.8640	1	37.6	R033000055	0.0301			Unknown	Confirmed High	1	0.900	R041000060	<LOQ (0.00493 ug/mL)		
	C91156F	1	Confirmed High	0.9275	0.8640	1	44.9	R033000058	0.0360			Unknown	Confirmed High	1	1.18	R041000064	<LOQ (0.00493 ug/mL)		
	C91158F	1	Confirmed High	0.9275	0.8640	1	49.9	R033000059	0.0400			Unknown	Confirmed High	1	0.00	R041000065	<LOQ (0.00493 ug/mL)		
	C91178F	1	NA	0.9275	0.8640	1	76.6	R033000060	0.0614			Unknown	NA	1	0.00	R041000066	<LOQ (0.00493 ug/mL)		
	C91180F	1	Confirmed High	0.9275	0.8640	1	67.0	R033000061	0.0537		49.9	Unknown	Confirmed High	1	1.04	R041000067	<LOQ (0.00493 ug/mL)		NA
	C91189F	1	Confirmed High	0.9275	0.8640	1	111	R033000062	0.089	0.0755	0.0376	Unknown	Confirmed High	1	1.10	R041000068	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90781M	1	NA	0.9275	0.8640	10	206	R050200016	1.65			Unknown	Confirmed High	1	12.8	R041000072	<LOQ (0.00493 ug/mL)		
	C90792M	1	NA	0.9275	0.8640	10	277	R050200017	3.03			Unknown	NA	1	16.1	R041000073	<LOQ (0.00493 ug/mL)		
	C90793M	1	NA	0.9275	0.8640	1	459	R033000067	0.367			Unknown	NA	1	22.3	R041000074	<LOQ (0.00493 ug/mL)		
	C90798M	1	NA	0.9275	0.8640	10	153	R050200019	1.23			Unknown	NA	1	12.8	R041000075	<LOQ (0.00493 ug/mL)		
	C90799M	1	NA	0.9275	0.8640	10	278	R050200020	2.23			Unknown	Confirmed High	1	13.3	R041000076	<LOQ (0.00493 ug/mL)		
	C90800M	1	NA	0.9275	0.8640	10	363	R050200025	2.91			Unknown	2nd Analysis OK	1	15.2	R041000080	<LOQ (0.00493 ug/mL)		
	C90812M	1	Confirmed High	0.9275	0.8640	1	841	A041800051	0.674			Unknown	Confirmed High	1	13.3	R041000081	<LOQ (0.00493 ug/mL)		
	C90822M	1	Confirmed High	0.9275	0.8640	1	705	A041800052	0.565			Unknown	Confirmed High	1	11.8	R041000082	<LOQ (0.00493 ug/mL)		
	C90829M	1	NA	0.9275	0.8640	10	304	R050200027	2.43		60.6	Unknown	Confirmed High	1	13.8	R041000083	<LOQ (0.00493 ug/mL)		NA
	C90832M	1	NA	0.9275	0.8640	10	440	R050200028	3.53	1.86	1.128	Unknown	Confirmed High	1	23.6	R041000084	<LOQ (0.00493 ug/mL)	<LOQ	NA
	C91194F	1	NA	0.9275	0.8640	10	350	R050200029	2.81			Unknown	Confirmed High	1	28.6	R041000088	<LOQ (0.00493 ug/mL)		
	C91197F	1	NA	0.9275	0.8640	10	620	R050200034	4.97			Unknown	Confirmed High	1	34.5	R041000089	<LOQ (0.00493 ug/mL)		
	C91199F	1	NA	0.9275	0.8640	10	615	R050200035	4.93			Unknown	Confirmed High	1	32.4	R041000090	<LOQ (0.00493 ug/mL)		
	C91200F	1	NA	0.9275	0.8640	10	459	R050200036	3.68			Unknown	Confirmed High	1	19.2	R041000091	<LOQ (0.00493 ug/mL)		
	C91206F	1	NA	0.9275	0.8640	10	292	D041700086	2.34			Unknown	Confirmed High	1	15.9	R041000092	<LOQ (0.00493 ug/mL)		
	C91227F	1	NA	0.9275	0.8640	10	729	R050200038	5.84			Unknown	Confirmed High	1	27.4	R041000096	<LOQ (0.00493 ug/mL)		
	C91229F	1	NA	0.9275	0.8640	10	831	R050200043	6.66			Unknown	Confirmed High	1	26.1	R041000097	<LOQ (0.00493 ug/mL)		
	C91231F	1	NA	0.9275	0.8640	10	370	R050200044	2.97			Unknown	NA	1	27.4	R041000098	<LOQ (0.00493 ug/mL)		
	C91244F	1	NA	0.9275	0.8640	10	849	R050200045	6.80		34.9	Unknown	NA	1	34.5	R041000099	<LOQ (0.00493 ug/mL)		NA
	C91250F	1	NA	0.9275	0.8640	10	713	R050200046	5.72	4.67	1.63	Unknown	NA	1	30.8	R041000100	<LOQ (0.00493 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL LAC 02/19/01

Date Entered/By: 04/17/00, 04/20/00, 04/24/0, 04/25/00, 05/04/00, 05/05/00, 06/12/00, 09/01/00, 09/18/00, 11/06/00 CSH/MMH/LAC
Date Verified/ By: 02/23/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number: Amelia 062498, Soup 020199, Ruby 100699
Instrument Software/Version: Masslynx 3.3 & 3.4
Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Dates of Extraction/Analyst: 2/25/00 RWW
Dates of Analysis/Analyst: 03/22/00, 03/30/00, 04/10/00, 04/18/00, 04/20/00, 04/21/00, 05/01/00, 05/24/00, 08/18/00, 08/21/00, 08/23/00 CSH/MMH/IAS
Date of Data Reduction/Analyst: 04/12/00, 04/13/00, 04/19/00, 04/20/00, 04/21/00, 04/25/00, 05/03/00, 05/25/00, 08/21/00, 08/22/00, 08/25/00 IAS/MMH/HOJ

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Dilution Factor	PFOSAA Conc. ng/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	EtFOSE-OH Purity Correction Factor	Surrogate Verified	EtFOSE-OH Dilution Factor	EtFOSE-OH Conc. ng/mL	Filename	Concentration of EtFOSE-OH ug/mL or % Rec	Mean EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD
		lot T-7121.1								lot Unknown/SD013							
Method Blk	RBS02250-H2O Blk-1-1	Unknown	NA	1	0.00	D082100007	<LOQ (0.0248 ug/mL)			Unknown	NA	1	2.89	A032200014	<LOQ (0.00977 ug/mL)		
	RBS02250-H2O Blk-1-2	Unknown	NA	1	0.00	D050100018	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.890	D050100018	<LOQ (0.00977 ug/mL)		
	RBS02250-H2O Blk-1-3	Unknown	NA	1	0.00	D050100019	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	D050100019	<LOQ (0.00977 ug/mL)		
	RBS02250-H2O Blk-1-4	Unknown	NA	1	0.00	D050100020	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100020	<LOQ (0.00977 ug/mL)	<LOQ	NA
Matrix Blk	RBS02250-Sera Blk-1-1	Unknown	NA	1	0.00	D082100008	<LOQ (0.0248 ug/mL)			Unknown	NA	1	2.57	A032200021	<LOQ (0.00977 ug/mL)		
	RBS02250-Sera Blk-1-2	Unknown	NA	1	0.00	D050100021	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	D050100021	<LOQ (0.00977 ug/mL)		
	RBS02250-Sera Blk-1-3	Unknown	NA	1	0.00	D050100022	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	D050100022	<LOQ (0.00977 ug/mL)		
	RBS02250-Sera Blk-1-4	Unknown	NA	1	0.00	D050100023	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100023	<LOQ (0.00977 ug/mL)	<LOQ	NA
Matrix Blk	RTS02250-Sera Blk-1-1	Unknown	NA	1	0.00	D082100009	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	A032200028	<LOQ (0.00977 ug/mL)		
	RTS02250-Sera Blk-1-2	Unknown	NA	1	0.00	D050100024	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	D050100024	<LOQ (0.00977 ug/mL)		
	RTS02250-Sera Blk-1-3	Unknown	NA	1	0.00	D050100025	<LOQ (0.0248 ug/mL)			Unknown	NA	1	0.00	D050100025	<LOQ (0.00977 ug/mL)		
	RTS02250-Sera Blk-1-4	Unknown	NA	1	0.00	D050100026	<LOQ (0.0248 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100026	<LOQ (0.00977 ug/mL)	<LOQ	NA
QC-250 ng/mL	RTS02250-MS-1-1	NA	NA	1	249	R033000018	100%			NA	NA	1	78.2	R033000018	32%		
	RTS02250-MS-1-2	NA	NA	1	242	R033000019	98%		5%	NA	NA	1	98.3	R033000019	40%		12%
	RTS02250-MS-1-3	NA	NA	1	224	R033000020	90%	96%	5%	NA	NA	1	95.4	R033000020	38%	37%	4%
	RTS02250-MS-1-1	NA	NA	NA	NA	NA	NA			NA	NA	1	69.5	D050100030	28% 2nd		
	RTS02250-MS-1-2	NA	NA	NA	NA	NA	NA		NA	NA	NA	1	78.4	D050100034	32% 2nd		10%
	RTS02250-MS-1-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	1	64.9	D050100035	26% 2nd	29%	3%
	RTS02250-MSD-2-1	NA	NA	1	268	D042100034	108%			NA	NA	1	95.6	D03300082	39%		
	RTS02250-MSD-2-2	NA	NA	1	278	D042100036	112%		4%	NA	NA	1	115	D03300083	46%		10%
	RTS02250-MSD-2-3	NA	NA	1	291	D042100041	117%	113%	5%	NA	NA	1	114	D03300084	46%	44%	4%
	RTS02250-MSD-2-1	NA	NA	NA	NA	NA	NA			NA	NA	1	87.2	D050100036	35% 2nd		
	RTS02250-MSD-2-2	NA	NA	NA	NA	NA	NA		NA	NA	NA	1	70.6	D050100037	28% 2nd		13%
	RTS02250-MSD-2-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	1	91.2	D050100038	37% 2nd	33%	4%
Group 1 Control 0.0 mg/kg	C90712M	Unknown	NA	1	0.00	R041700018	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000024	<LOQ (0.00977 ug/mL)		
	C90714M	Unknown	NA	1	3.79	R041700019	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000025	<LOQ (0.00977 ug/mL)		
	C90723M	Unknown	NA	1	0.00	R041700020	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000026	<LOQ (0.00977 ug/mL)		
	C90727M	Unknown	Confirmed High	1	0.00	R041700021	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000027	<LOQ (0.00977 ug/mL)		
	C90730M	Unknown	NA	1	0.00	R041700022	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000028	<LOQ (0.00977 ug/mL)		
	C90751M	Unknown	NA	1	5.75	R041700026	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000032	<LOQ (0.00977 ug/mL)		
	C90752M	Unknown	NA	1	0.00	R041700027	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000033	<LOQ (0.00977 ug/mL)		
	C90754M	Unknown	NA	1	0.00	R041700028	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000034	<LOQ (0.00977 ug/mL)		
	C90755M	Unknown	Confirmed High	1	0.00	R041700029	<LOQ (0.0097 ug/mL)			Unknown	Confirmed High	1	0.00	R041000035	<LOQ (0.00977 ug/mL)		
	C90758M	Unknown	NA	1	0.00	R041700030	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	R041000036	<LOQ (0.00977 ug/mL)		
	C90759M	Unknown	Confirmed High	1	0.00	R041700034	<LOQ (0.0097 ug/mL)			Unknown	Confirmed High	1	0.00	R041000040	<LOQ (0.00977 ug/mL)		
	C90764M	Unknown	Confirmed High	1	0.00	R041700035	<LOQ (0.0097 ug/mL)			Unknown	Confirmed High	1	0.00	R041000041	<LOQ (0.00977 ug/mL)		
	C90766M	Unknown	Confirmed High	1	0.00	R041700036	<LOQ (0.0097 ug/mL)			Unknown	Confirmed High	1	0.00	R041000042	<LOQ (0.00977 ug/mL)		
	C90767M	Unknown	NA	1	0.00	R041700037	<LOQ (0.0097 ug/mL)		NA	Unknown	NA	1	0.00	R041000043	<LOQ (0.00977 ug/mL)		NA
	C90774M	Unknown	NA	1	0.00	R041700038	<LOQ (0.0097 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	R041000044	<LOQ (0.00977 ug/mL)	<LOQ	NA
	C91121F	Unknown	NA	1	0.00	R041700042	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	A041800018	<LOQ (0.00493 ug/mL)		
	C91122F	Unknown	Confirmed High	1	0.00	R041700043	<LOQ (0.0097 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800019	<LOQ (0.00493 ug/mL)		
	C91123F	Unknown	Confirmed High	1	0.00	R041700044	<LOQ (0.0097 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800020	<LOQ (0.00493 ug/mL)		
	C91124F	Unknown	Confirmed High	1	17.44	R041700045	0.0174			Unknown	2nd Analysis OK	1	0.00	A041800021	<LOQ (0.00493 ug/mL)		
	C91125F	Unknown	Confirmed High	1	0.00	R041700046	<LOQ (0.0097 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800022	<LOQ (0.00493 ug/mL)		
	C91131F	Unknown	NA	1	0.00	R041700050	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	A041800026	<LOQ (0.00493 ug/mL)		
	C91132F	Unknown	NA	1	0.00	R041700051	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	A041800027	<LOQ (0.00493 ug/mL)		
	C91133F	Unknown	Confirmed High	1	0.00	R041700052	<LOQ (0.0097 ug/mL)			Unknown	Confirmed High	1	0.00	A041800028	<LOQ (0.00493 ug/mL)		
	C91152F	Unknown	Confirmed High	1	13.64	R041700053	0.0136			Unknown	2nd Analysis OK	1	0.00	A041800029	<LOQ (0.00493 ug/mL)		
	C91153F	Unknown	Confirmed High	1	0.00	R041700054	<LOQ (0.0097 ug/mL)			Unknown	Confirmed High	1	0.00	A041800030	<LOQ (0.00493 ug/mL)		
	C91156F	Unknown	Confirmed High	1	0.00	R041700058	<LOQ (0.0097 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800034	<LOQ (0.00493 ug/mL)		
	C91158F	Unknown	Confirmed High	1	0.00	R041700059	<LOQ (0.0097 ug/mL)			Unknown	Confirmed High	1	0.00	A041800035	<LOQ (0.00493 ug/mL)		
	C91178F	Unknown	NA	1	0.00	R041700060	<LOQ (0.0097 ug/mL)			Unknown	NA	1	0.00	A041800036	<LOQ (0.00493 ug/mL)		
	C91180F	Unknown	Confirmed High	1	0.00	R041700061	<LOQ (0.0097 ug/mL)		NA	Unknown	2nd Analysis OK	1	0.00	A041800037	<LOQ (0.00493 ug/mL)		NA
	C91189F	Unknown	Confirmed High	1	0.00	R041700062	<LOQ (0.0097 ug/mL)	<LOQ	NA	Unknown	2nd Analysis OK	1	0.00	A041800038	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90781M	Unknown	Confirmed High	1	342	R041000072	0.342			Unknown	2nd Analysis OK	1	0.00	A041800042	<LOQ (0.00493 ug/mL)		
	C90792M	Unknown	NA	1	301	R041000073	0.301			Unknown	NA	1	0.00	A041800043	<LOQ (0.00493 ug/mL)		
	C90793M	Unknown	NA	1	504	R041000074	0.504			Unknown	NA	1	0.00	A041800044	<LOQ (0.00493 ug/mL)		
	C90798M	Unknown	NA	1	366	R041000075	0.366			Unknown	NA	1	0.00	A041800045	<LOQ (0.00493 ug/mL)		
	C90799M	Unknown	Confirmed High	1	378	R041000076	0.378			Unknown	Confirmed High	1	0.00	A041800046	<LOQ (0.00493 ug/mL)		
	C90800M	Unknown	2nd Analysis OK	1	289	R041000080	0.289			Unknown	2nd Analysis OK	1	0.880	A041800050	<LOQ (0.00493 ug/mL)		
	C90812M	Unknown	NA	10	123	R052400022	1.23			Unknown	Confirmed High	1	0.00	A041800051	<LOQ (0.00493 ug/mL)		
	C90822M	Unknown	Confirmed High	1	462	R041000082	0.462			Unknown	Confirmed High	1	0.00	A041800052	<LOQ (0.00493 ug/mL)		
	C90829M	Unknown	Confirmed High	1	333	R041000083	0.333		83.2	Unknown	Confirmed High	1	0.00	A041800053	<LOQ (0.00493 ug/mL)		NA
	C90832M	Unknown	NA	10	177	R052400024	1.77	0.597	0.497	Unknown	Confirmed High	1	0.00	A041800054	<LOQ (0.00493 ug/mL)	<LOQ	NA
	C91194F	Unknown	2nd Analysis OK	1	809	D042100058	0.809			Unknown	NA	1	4.27	R050200047	<LOQ(0.00977 ug/mL)		
	C91197F	Unknown	2nd Analysis OK	1	870	D042100059	0.870			Unknown	NA	1	4.49	R050200052	<LOQ(0.00977 ug/mL)		
	C91199F	Unknown	2nd Analysis OK	1	680	D041700084	0.680			Unknown	NA	1	4.35	R050200053	<LOQ(0.00977 ug/mL)		
	C91200F	Unknown	2nd Analysis OK	1	322	D041700085	0.322			Unknown	NA	1	2.17	R050200054	<LOQ(0.00977 ug/mL)		
	C91206F	Unknown	2nd Analysis OK	1	365	D041700086	0.365			Unknown	NA	1	3.29	R050200055	<LOQ(0.00977 ug/mL)		
	C91227F	Unknown	Confirmed High	1	629	D041700090	0.629			Unknown	NA	1	3.11	R050200056	<LOQ(0.00977 ug/mL)		
	C91229F	Unknown	2nd Analysis OK	1	582	D041700091	0.582			Unknown	NA	1	4.78	R050200061	<LOQ(0.00977 ug/mL)		
	C91231F	Unknown	NA	1	341	D041700092	0.341			Unknown	NA	1	3.64	R050200062	<LOQ(0.00977 ug/mL)		
	C91244F	Unknown	NA	1	590	D041700093	0.590		35.7	Unknown	NA	1	3.99	R050200063	<LOQ(0.00977 ug/mL)		NA
	C91250F	Unknown	NA	1	375	D041700094	0.375	0.556	0.199	Unknown	NA	1	5.40	R050200064	<LOQ(0.00977 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL LAC 02/19/01

Date Entered/By: 04/17/00, 04/20/00, 04/24/0, 04/25/00, 05-04/00, 05/05/00, 06/12/00, 09/01/00, 09/18/00, 11/06/00 CSH/MMH/LAC
Date Verified/ By: 02/23/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: ETS-8-4.1 & ETS-8-5 1
Analytical Equipment System Number: Amelia 062498, Soup 020199, Ruby 100699
Instrument Software/Version: Masslynx 3.3 & 3.4
Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Dates of Extraction/Analyst: 2/25/00 RWW
Dates of Analysis/Analyst: 03/22/00, 03/30/00, 04/10/00, 04/18/00, 04/20/00, 04/21/00, 05/01/00, 05/24/00, 08/18/00, 08/21/00, 08/23/00 CSH/MMH/IAS
Date of Data Reduction/Analyst: 04/12/00, 04/13/00, 04/19/00, 04/20/00, 04/21/00, 04/25/00, 05/03/00, 05/25/00, 08/21/00, 08/22/00, 08/25/00 IAS/MMH/HOJ

Sample Data

WEEK 53 RAT SERA lot NB113047-80 lot 529

Group Dose	Sample #	M556 Purity Correction Factor	Surrogate Verified	M556 Dilution Factor	M556 Conc. ng/mL	Filename	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Dilution Factor	PFOSEA Conc. ng/mL	Filename	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	RBS02250-H2O Blk-1-1	Unknown	NA	1	0.00	D042100004	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	A032200014	<LOQ (0.00492 ug/mL)		
	RBS02250-H2O Blk-1-2	Unknown	NA	1	0.03	D050100018	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	D050100018	<LOQ (0.00492 ug/mL)		
	RBS02250-H2O Blk-1-3	Unknown	NA	1	0.00	D050100019	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	D050100019	<LOQ (0.00492 ug/mL)		
	RBS02250-H2O Blk-1-4	Unknown	NA	1	0.00	D050100020	< LOQ (0.00494 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100020	<LOQ (0.00492 ug/mL)	<LOQ	NA
Matrix Blk	RBS02250-Sera Blk-1-1	Unknown	NA	1	0.00	D042100005	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	A032200021	<LOQ (0.00492 ug/mL)		
	RBS02250-Sera Blk-1-2	Unknown	NA	1	0.00	D050100021	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	D050100021	<LOQ (0.00492 ug/mL)		
	RBS02250-Sera Blk-1-3	Unknown	NA	1	0.00	D050100022	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	D050100022	<LOQ (0.00492 ug/mL)		
	RBS02250-Sera Blk-1-4	Unknown	NA	1	0.00	D050100026	< LOQ (0.00494 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100023	<LOQ (0.00492 ug/mL)	<LOQ	NA
Matrix Blk	RTS02250-Sera Blk-1-1	Unknown	NA	1	0.00	D042100006	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	A032200028	<LOQ (0.00492 ug/mL)		
	RTS02250-Sera Blk-1-2	Unknown	NA	1	0.00	D050100027	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	D050100024	<LOQ (0.00492 ug/mL)		
	RTS02250-Sera Blk-1-3	Unknown	NA	1	0.00	D050100028	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	D050100025	<LOQ (0.00492 ug/mL)		
	RTS02250-Sera Blk-1-4	Unknown	NA	1	0.00	D050100029	< LOQ (0.00494 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100026	<LOQ (0.00492 ug/mL)	<LOQ	NA
QC-250 ng/mL	RTS02250-MS-1-1	NA	NA	1	316	R041000018	127%			NA	NA	1	174	R033000018	70%		
	RTS02250-MS-1-2	NA	NA	1	282	R041000019	114%		9%	NA	NA	1	159	R033000019	64%		21%
	RTS02250-MS-1-3	NA	NA	1	336	R041000020	135%	125%	11%	NA	NA	1	114	R033000020	46%	60%	13%
	RTS02250-MS-1-1	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA NA		
	RTS02250-MS-1-2	NA	NA	NA	NA	NA	NA		NA	NA	NA	1	125	R041000019	51% 2nd		34%
	RTS02250-MS-1-3	NA	NA	1	305	D042100037	123% 2nd	123%	NA	NA	NA	1	76.4	R041000020	31% 2nd	41%	14%
	RTS02250-MSD-2-1	NA	NA	1	307	D042100034	124%			NA	NA	1	157	D03300082	63%		
	RTS02250-MSD-2-2	NA	NA	1	308	D042100036	124%		4%	NA	NA	1	188	D03300083	76%		11%
	RTS02250-MSD-2-3	NA	NA	1	329	D042100041	133%	127%	5%	NA	NA	1	192	D03300084	78%	72%	8%
	RTS02250-MSD-2-1	NA	NA	NA	NA	NA	NA			NA	NA	1	157	D050100036	63% 2nd		
	RTS02250-MSD-2-2	NA	NA	NA	NA	NA	NA		NA	NA	NA	NA	NA	NA	NA		NA
	RTS02250-MSD-2-3	NA	NA	1	325	D050100038	131% 2nd	131%	NA	NA	NA	NA	NA	NA	NA	63%	NA
Group 1 Control 0.0 mg/kg	C90712M	Unknown	NA	1	1.36	D050100042	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	R041000024	<LOQ (0.00492 ug/mL)		
	C90714M	Unknown	NA	1	0.710	D050100043	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	R041000025	<LOQ (0.00492 ug/mL)		
	C90723M	Unknown	NA	1	0.00	D050100044	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	R041000026	<LOQ (0.00492 ug/mL)		
	C90727M	Unknown	NA	1	0.00	D050100045	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	D050100045	<LOQ (0.00492 ug/mL)		
	C90730M	Unknown	NA	1	0.00	D050100046	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	D050100046	<LOQ (0.00492 ug/mL)		
	C90751M	Unknown	NA	1	0.340	D050100050	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	R041000032	<LOQ (0.00492 ug/mL)		
	C90752M	Unknown	NA	1	0.450	D050100051	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	R041000033	<LOQ (0.00492 ug/mL)		
	C90754M	Unknown	NA	1	0.380	D050100052	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	R041000034	<LOQ (0.00492 ug/mL)		
	C90755M	Unknown	Confirmed High	1	0.00	D050100053	< LOQ (0.00494 ug/mL)			Unknown	Confirmed High	1	0.00	D050100053	<LOQ (0.00492 ug/mL)		
	C90758M	Unknown	NA	1	0.00	D050100054	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	R041000036	<LOQ (0.00492 ug/mL)		
	C90759M	Unknown	Confirmed High	1	0.33	D050100058	< LOQ (0.00494 ug/mL)			Unknown	Confirmed High	1	0.00	D050100058	<LOQ (0.00492 ug/mL)		
	C90764M	Unknown	Confirmed High	1	0.000	D050100059	< LOQ (0.00494 ug/mL)			Unknown	Confirmed High	1	0.00	D050100059	<LOQ (0.00492 ug/mL)		
	C90766M	Unknown	Confirmed High	1	0.00	D050100060	< LOQ (0.00494 ug/mL)			Unknown	Confirmed High	1	0.00	D050100060	<LOQ (0.00492 ug/mL)		
	C90767M	Unknown	NA	1	0.00	D050100061	< LOQ (0.00494 ug/mL)		NA	Unknown	NA	1	0.00	D050100061	<LOQ (0.00492 ug/mL)		NA
	C90774M	Unknown	NA	1	0.00	D050100062	< LOQ (0.00494 ug/mL)	<LOQ	NA	Unknown	NA	1	0.00	D050100062	<LOQ (0.00492 ug/mL)	<LOQ	NA
	C91121F	Unknown	NA	1	1.78	D050100066	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	A041800018	<LOQ (0.00492 ug/mL)		
	C91122F	Unknown	Confirmed High	1	0.820	D050100067	< LOQ (0.00494 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800019	<LOQ (0.00492 ug/mL)		
	C91123F	Unknown	Confirmed High	1	0.320	D050100068	< LOQ (0.00494 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800020	<LOQ (0.00492 ug/mL)		
	C91124F	Unknown	Confirmed High	1	1.15	D050100069	< LOQ (0.00494 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800021	<LOQ (0.00492 ug/mL)		
	C91125F	Unknown	Confirmed High	1	0.00	D050100070	< LOQ (0.00494 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800022	<LOQ (0.00492 ug/mL)		
	C91131F	Unknown	NA	1	1.01	D050100074	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	A041800026	<LOQ (0.00492 ug/mL)		
	C91132F	Unknown	NA	1	0.00	D050100075	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	A041800027	<LOQ (0.00492 ug/mL)		
	C91133F	Unknown	Confirmed High	1	0.260	D050100076	< LOQ (0.00494 ug/mL)			Unknown	Confirmed High	1	0.00	A041800028	<LOQ (0.00492 ug/mL)		
	C91152F	Unknown	Confirmed High	1	0.00	D050100077	< LOQ (0.00494 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800029	<LOQ (0.00492 ug/mL)		
	C91153F	Unknown	Confirmed High	1	0.00	D050100078	< LOQ (0.00494 ug/mL)			Unknown	Confirmed High	1	0.00	A041800030	<LOQ (0.00492 ug/mL)		
	C91156F	Unknown	Confirmed High	1	1.55	D050100082	< LOQ (0.00494 ug/mL)			Unknown	2nd Analysis OK	1	0.00	A041800034	<LOQ (0.00492 ug/mL)		
	C91158F	Unknown	Confirmed High	1	0.220	D050100083	< LOQ (0.00494 ug/mL)			Unknown	Confirmed High	1	0.00	A041800035	<LOQ (0.00492 ug/mL)		
	C91178F	Unknown	NA	1	0.00	D050100084	< LOQ (0.00494 ug/mL)			Unknown	NA	1	0.00	A041800036	<LOQ (0.00492 ug/mL)		
	C91180F	Unknown	Confirmed High	1	0.530	D050100085	< LOQ (0.00494 ug/mL)		NA	Unknown	2nd Analysis OK	1	0.00	A041800037	<LOQ (0.00492 ug/mL)		NA
	C91189F	Unknown	Confirmed High	1	0.360	D050100086	< LOQ (0.00494 ug/mL)	<LOQ	NA	Unknown	2nd Analysis OK	1	0.00	A041800038	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90781M	Unknown	Confirmed High	1	344	R041000072	0.344			Unknown	2nd Analysis OK	1	0.00	A041800042	<LOQ (0.00492 ug/mL)		
	C90792M	Unknown	NA	1	376	R041000073	0.376			Unknown	NA	1	0.00	A041800043	<LOQ (0.00492 ug/mL)		
	C90793M	Unknown	NA	1	189	R041000074	0.189			Unknown	NA	1	0.00	A041800044	<LOQ (0.00492 ug/mL)		
	C90798M	Unknown	NA	1	294	R041000075	0.294			Unknown	NA	1	0.00	A041800045	<LOQ (0.00492 ug/mL)		
	C90799M	Unknown	Confirmed High	1	424	R041000076	0.424			Unknown	Confirmed High	1	0.00	A041800046	<LOQ (0.00492 ug/mL)		
	C90800M	Unknown	2nd Analysis OK	1	429	R041000080	0.429			Unknown	2nd Analysis OK	1	0.00	A041800050	<LOQ (0.00492 ug/mL)		
	C90812M	Unknown	Confirmed High	1	341	R041000081	0.341			Unknown	Confirmed High	1	0.00	A041800051	<LOQ (0.00492 ug/mL)		
	C90822M	Unknown	Confirmed High	1	286	R041000082	0.286			Unknown	Confirmed High	1	0.00	A041800052	<LOQ (0.00492 ug/mL)		
	C90829M	Unknown	Confirmed High	1	436	R041000083	0.436		29.5	Unknown	Confirmed High	1	0.00	A041800053	<LOQ (0.00492 ug/mL)		NA
	C90832M	Unknown	Confirmed High	1	594	R041000084	0.594	0.371	0.110	Unknown	Confirmed High	1	0.00	A041800054	<LOQ (0.00492 ug/mL)	<LOQ	NA
	C91194F	Unknown	Confirmed High	1	441	R041000088	0.441			Unknown	Confirmed High	1	0.00	R041000088	<LOQ (0.0492 ug/mL)		
	C91197F	Unknown	Confirmed High	1	694	R041000089	0.694			Unknown	Confirmed High	1	0.00	R041000089	<LOQ (0.0492 ug/mL)		
	C91199F	Unknown	Confirmed High	1	521	R041000090	0.521			Unknown	Confirmed High	1	0.00	R041000090	<LOQ (0.0492 ug/mL)		
	C91200F	Unknown	Confirmed High	1	403	R041000091	0.403			Unknown	Confirmed High	1	0.00	R041000091	<LOQ (0.0492 ug/mL)		
	C91206F	Unknown	Confirmed High	1	167	R041000092	0.167			Unknown	Confirmed High	1	0.00	R041000092	<LOQ (0.0492 ug/mL)		
	C91227F	Unknown	Confirmed High	1	418	R041000096	0.418			Unknown	Confirmed High	1	0.00	R041000096	<LOQ (0.0492 ug/mL)		
	C91229F	Unknown	Confirmed High	1	408	R041000097	0.408			Unknown	NA	1	0.00	R041000097	<LOQ (0.0492 ug/mL)		
	C91231F	Unknown	NA	1	476	R041000098	0.476			Unknown	NA	1	0.00	R041000098	<LOQ (0.0492 ug/mL)		
	C91244F	Unknown	NA	1	504	R041000099	0.504		30.4	Unknown	NA	1	0.00	R041000099	<LOQ (0.0492 ug/mL)		NA
	C91250F	Unknown	NA	1	356	R041000100	0.356	0.439	0.134	Unknown	NA	1	0.00	R041000100	<LOQ (0.0492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01

Date Entered/By: 04/17/00, 04/20/00, 04/24/0, 04/25/00, 05/04/00, 05/05/00, 06/12/00, 09/01/00, 09/18/00, 11/06/00 CSH/MMH/LAC
Date Verified/ By: 02/23/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number: Amelia 062498, Soup 020199, Ruby 100699
Instrument Software/Version: Masslynx 3.3 & 3.4
Filename: See Attachments
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Dates of Extraction/Analyst: 2/25/00 RWW
Dates of Analysis/Analyst: 03/22/00, 03/30/00, 04/10/00, 04/18/00, 04/20/00, 04/21/00, 05/01/00, 05/24/00, 08/18/00, 08/21/00, 08/23/00 CSH/MMH/IAS
Date of Data Reduction/Analyst: 04/12/00, 04/13/00, 04/19/00, 04/20/00, 04/21/00, 04/25/00, 05/03/00, 05/25/00, 08/21/00, 08/22/00, 08/25/00 IAS/MMH/HOJ

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of EtFOSE-OH ug/mL or % Rec	Mean EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	RBS02250-H2O Blk-1-1	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RBS02250-H2O Blk-1-2	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RBS02250-H2O Blk-1-3	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RBS02250-H2O Blk-1-4	< LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	< LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
Matrix Blk	RBS02250-Sera Blk-1-1	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RBS02250-Sera Blk-1-2	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RBS02250-Sera Blk-1-3	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RBS02250-Sera Blk-1-4	< LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	< LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
Matrix Blk	RTS02250-Sera Blk-1-1	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RTS02250-Sera Blk-1-2	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RTS02250-Sera Blk-1-3	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	RTS02250-Sera Blk-1-4	< LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	< LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
QC-250 ng/mL	RTS02250-MS-1-1	75%			83%			100%			32%			127%			70%		
	RTS02250-MS-1-2	76%		6%	87%		3%	98%		5%	40%		12%	114%		9%	64%		21%
	RTS02250-MS-1-3	68%	73%	4%	86%	85%	2%	90%	96%	5%	38%		4%	135%	125%	11%	46%	60%	13%
	RTS02250-MS-1-1	NA			NA			NA			28% 2nd			NA			NA		
	RTS02250-MS-1-2	NA		NA	NA		NA	NA		NA	32% 2nd		10%	NA		NA	51% 2nd		34%
	RTS02250-MS-1-3	78% 2nd	78%	NA	NA	NA	NA	NA	NA	NA	26% 2nd	29%	3%	123% 2nd	123%	NA	31% 2nd	41%	14%
	RTS02250-MSD-2-1	97%			94%			108%			39%			124%			63%		
	RTS02250-MSD-2-2	109%		7%	102%		8%	112%		4%	46%		10%	124%		4%	76%		11%
	RTS02250-MSD-2-3	112%	106%	8%	111%	102%	9%	117%	113%	5%	46%	44%	4%	133%	127%	5%	78%	72%	8%
	RTS02250-MSD-2-1	NA			NA			NA			35% 2nd			NA			63% 2nd		
	RTS02250-MSD-2-2	NA		NA	NA		NA	NA		NA	28% 2nd		13%	NA		NA	NA		NA
	RTS02250-MSD-2-3	NA	NA	NA	NA	NA	NA	NA	NA	NA	37% 2nd	33%	4%	131% 2nd	131%	NA	NA	63%	NA
Group 1 Control 0.0 mg/kg	C90712M	0.0101			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90714M	0.0288			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90723M	0.0121			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90727M	< LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90730M	0.0070			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90751M	0.0088			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90752M	0.0477			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90754M	0.0325			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90755M	0.0136			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90758M	0.0109			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90759M	0.0307			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90764M	0.0193			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90766M	0.0066			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00977 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90767M	0.0148		70.3	<LOQ (0.00493 ug/mL)		NA	<LOQ (0.0097 ug/mL)		NA	<LOQ (0.00977 ug/mL)		NA	< LOQ (0.00494 ug/mL)		NA	<LOQ (0.00492 ug/mL)		NA
	C90774M	0.0393	0.0191	0.0134	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0097 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL)	<LOQ	NA	< LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
	C91121F	0.101			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91122F	0.138			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91123F	0.0590			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91124F	0.101			<LOQ (0.00493 ug/mL)			0.02			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91125F	0.0606			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91131F	0.0788			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91132F	0.0251			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91133F	0.123			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91152F	0.134			<LOQ (0.00493 ug/mL)			0.01			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91153F	0.0301			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91156F	0.0360			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91158F	0.0400			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91178F	0.0614			<LOQ (0.00493 ug/mL)			<LOQ (0.0097 ug/mL)			<LOQ (0.00493 ug/mL)			< LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91180F	0.0537		49.9	<LOQ (0.00493 ug/mL)		NA	<LOQ (0.0097 ug/mL)		NA	<LOQ (0.00493 ug/mL)		NA	< LOQ (0.00494 ug/mL)		NA	<LOQ (0.00492 ug/mL)		NA
	C91189F	0.089	0.0755	0.0376	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0097 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	< LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90781M	1.65			<LOQ (0.00493 ug/mL)			0.342			<LOQ (0.00493 ug/mL)			0.344			<LOQ (0.00492 ug/mL)		
	C90792M	3.03			<LOQ (0.00493 ug/mL)			0.301			<LOQ (0.00493 ug/mL)			0.376			<LOQ (0.00492 ug/mL)		
	C90793M	0.367			<LOQ (0.00493 ug/mL)			0.504			<LOQ (0.00493 ug/mL)			0.189			<LOQ (0.00492 ug/mL)		
	C90798M	1.23			<LOQ (0.00493 ug/mL)			0.366			<LOQ (0.00493 ug/mL)			0.294			<LOQ (0.00492 ug/mL)		
	C90799M	2.23			<LOQ (0.00493 ug/mL)			0.378			<LOQ (0.00493 ug/mL)			0.424			<LOQ (0.00492 ug/mL)		
	C90800M	2.91			<LOQ (0.00493 ug/mL)			0.289			<LOQ (0.00493 ug/mL)			0.429			<LOQ (0.00492 ug/mL)		
	C90812M	0.674			<LOQ (0.00493 ug/mL)			1.230			<LOQ (0.00493 ug/mL)			0.341			<LOQ (0.00492 ug/mL)		
	C90822M	0.565			<LOQ (0.00493 ug/mL)			0.462			<LOQ (0.00493 ug/mL)			0.286			<LOQ (0.00492 ug/mL)		
	C90829M	2.43		60.6	<LOQ (0.00493 ug/mL)		NA	0.333		83.2	<LOQ (0.00493 ug/mL)		NA	0.436		29.5	<LOQ (0.00492 ug/mL)		NA
	C90832M	3.53	1.86	1.128	<LOQ (0.00493 ug/mL)	<LOQ	NA	1.770	0.597	0.497	<LOQ (0.00493 ug/mL)	<LOQ	NA	0.594	0.371	0.110	<LOQ (0.00492 ug/mL)	<LOQ	NA
	C91194F	2.81			<LOQ (0.00493 ug/mL)			0.809			<LOQ(0.00977 ug/mL)			0.441			<LOQ (0.0492 ug/mL)		
	C91197F	4.97			<LOQ (0.00493 ug/mL)			0.870			<LOQ(0.00977 ug/mL)			0.694			<LOQ (0.0492 ug/mL)		
	C91199F	4.93			<LOQ (0.00493 ug/mL)			0.680			<LOQ(0.00977 ug/mL)			0.521			<LOQ (0.0492 ug/mL)		
	C91200F	3.68			<LOQ (0.00493 ug/mL)			0.322			<LOQ(0.00977 ug/mL)			0.403			<LOQ (0.0492 ug/mL)		
	C91206F	2.34			<LOQ (0.00493 ug/mL)			0.365			<LOQ(0.00977 ug/mL)			0.167			<LOQ (0.0492 ug/mL)		
	C91227F	5.84			<LOQ (0.00493 ug/mL)			0.629			<LOQ(0.00977 ug/mL)			0.418			<LOQ (0.0492 ug/mL)		
	C91229F	6.66			<LOQ (0.00493 ug/mL)			0.582			<LOQ(0.00977 ug/mL)			0.408			<LOQ (0.0492 ug/mL)		
	C91231F	2.97			<LOQ (0.00493 ug/mL)			0.341			<LOQ(0.00977 ug/mL)			0.476			<LOQ (0.0492 ug/mL)		
	C91244F	6.80		34.9	<LOQ (0.00493 ug/mL)		NA	0.590		35.7	<LOQ(0.00977 ug/mL)		NA	0.504		30.4	<LOQ (0.0492 ug/mL)		NA
	C91250F	5.72	4.67	1.63	<LOQ (0.00493 ug/mL)	<LOQ	NA	0.375	0.556	0.199	<LOQ(0.00977 ug/mL)	<LOQ	NA	0.356	0.439	0.134	<LOQ (0.0492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug mL. LAC 02/19/01

Date Entered/By: 04/17/00, 04/20/00, 04/24/0, 04/25/00, 05/04/00, 05/05/00, 06/12/00, 09/01/00, 09/18/00, 11/06/00 CSH MMH/LAC
Date Verified/ By: 02/23/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number: Amelia 062498, Soup 020199, Ruby 100699
Instrument Software/Version: Masslynx 3.3 & 3.4
Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Dates of Extraction/Analyst: 2/25/00 RWW
Dates of Analysis/Analyst: 03/22/00, 03/30/00, 04/10/00, 04/18/00, 04/20/00, 04/21/00, 05/01/00, 05/30/00, 08/18/00, 08/21/00, 08/23/00, 02/27/01 CSH/MMH/IAS
Date of Data Reduction/Analyst: 04/12/00, 04/13/00, 04/19/00, 04/20/00, 04/21/00, 04/25/00, 05/03/00, 05/31/00, 08/21/00, 08/22/00, 08/25/00, 03/13/01 IAS/MMH/HOJ

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Std Correction Factor	PFOS Purity Correction Factor (lot 171)	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor (lot L-15709)	Surrogate Verified	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90841M	1	NA	0.9275	0.8640	100	208	D042100020	16.7			Unknown	NA	1	201	R041000104	0.201		
Mid Dose	C90850M	1	NA	0.9275	0.8640	100	256	D042100021	20.5			Unknown	NA	1	209	R041000105	0.209		
30.0 mg/kg	C90854M	1	NA	0.9275	0.8640	100	330	D042100022	26.4			Unknown	NA	1	338	R041000106	0.338		
	C90855M	1	NA	0.9275	0.8640	100	179	D042100023	14.3			Unknown	NA	1	293	R041000107	0.293		
	C90856M	1	NA	0.9275	0.8640	100	222	D042100024	17.8			Unknown	NA	1	181	R041000108	0.181		
	C90859M	1	NA	0.9275	0.8640	100	306	D042100028	24.5			Unknown	NA	1	231	R041000112	0.231		
	C90873M	1	NA	0.9275	0.8640	100	341	D042100029	27.3			Unknown	NA	1	252	R041000113	0.252		
	C90879M	1	NA	0.9275	0.8640	100	45	D042100030	3.58			Unknown	NA	1	125	R041000114	0.125		
	C90881M	1	NA	0.9275	0.8640	100	151	D042100031	12.1		39.8	Unknown	NA	1	180	R041000115	0.180		31.6
	C90896M	1	NA	0.9275	0.8640	100	214	D042100032	17.2	18.0	7.18	Unknown	NA	1	365	R041000116	0.365	0.237	0.0751
	C91255F	1	NA	0.9275	0.8640	100	701	D042100036	56.2			Unknown	NA	1	393	D03300087	0.393		
	C91258F	1	NA	0.9275	0.8640	100	342	D042100037	27.4			Unknown	NA	1	268	D03300088	0.268		
	C91264F	1	NA	0.9275	0.8640	100	368	D042100038	29.5			Unknown	NA	1	324	D03300089	0.324		
	C91269F	1	NA	0.9275	0.8640	100	491	D042100039	39.3			Unknown	NA	1	489	D03300090	0.489		
	C91270F	1	NA	0.9275	0.8640	100	142	D042100040	11.4			Unknown	NA	1	485	D03300091	0.485		
	C91274F	1	NA	0.9275	0.8640	100	442	D042100044	35.4			Unknown	NA	1	404	D03300094	0.404		
	C91289F	1	NA	0.9275	0.8640	100	475	D042100045	38.1			Unknown	NA	1	279	D03300095	0.279		
	C91294F	1	NA	0.9275	0.8640	100	363	D042100046	29.1			Unknown	NA	1	283	D03300096	0.283		
	C91297F	1	NA	0.9275	0.8640	100	480	D042100047	38.5		33.8	Unknown	NA	1	415	D03300097	0.415		22.5
	C91305F	1	NA	0.9275	0.8640	100	392	D042100048	31.4	33.6	11.4	Unknown	NA	1	323	D03300098	0.323	0.366	0.0824
Group 4	C90902M	1	NA	0.9275	0.8640	100	880	R053000030	70.5			Unknown	NA	1	615	D03300101	0.615		
Mid-High Dose	C90908M	1	NA	0.9275	0.8640	100	697	D042000015	55.8			Unknown	NA	1	275	D03300102	0.275		
100 mg/kg	C90914M	1	NA	0.9275	0.8640	100	680	D042000016	54.5			Unknown	NA	1	382	D03300103	0.382		
	C90918M	1	NA	0.9275	0.8640	100	837	R053000033	67.1			Unknown	NA	1	662	D03300104	0.662		
	C90923M	1	NA	0.9275	0.8640	100	777	D042000017	62.3			Unknown	NA	1	305	D03300105	0.305		
	C90926M	1	NA	0.9275	0.8640	100	613	D042000018	49.1			Unknown	NA	1	223	D03300108	0.223		
	C90927M	1	NA	0.9275	0.8640	100	751	D042000019	60.2			Unknown	NA	1	363	D03300109	0.363		
	C90934M	1	NA	0.9275	0.8640	100	464	D042000023	37.2			Unknown	NA	1	247	D03300110	0.247		
	C90936M	1	NA	0.9275	0.8640	100	640	D042000024	51.3			Unknown	NA	1	331	D03300111	0.331		
	C90944M	1	NA	0.9275	0.8640	100	761	D042000025	60.9			Unknown	NA	1	270	D03300112	0.270		
	C90946M	1	NA	0.9275	0.8640	100	750	D042000026	60.1			Unknown	NA	1	231	D03300115	0.231		
	C90950M	1	NA	0.9275	0.8640	1000	330	D081800023	264			Unknown	NA	1	474	D03300116	0.474		
	C90951M	1	NA	0.9275	0.8640	1000	274	D081800024	220			Unknown	NA	1	343	D03300117	0.343		
	C90956M	1	NA	0.9275	0.8640	100	614	D042000027	49.2		80.1	Unknown	NA	1	203	D03300118	0.203		38.7
	C90957M	1	NA	0.9275	0.8640	100	911	R053000034	73.0	82.4	66.0	Unknown	NA	1	430	D03300119	0.430	0.357	0.138
	C91314F	1	NA	0.9275	0.8640	1000	218	R053000023	175			Unknown	NA	10	105	D042000047	1.05		
	C91319F	1	NA	0.9275	0.8640	100	917	D042100051	73.5			Unknown	NA	10	120	D042000048	1.20		
	C91323F	1	NA	0.9275	0.8640	1000	274	R053000016	220			Unknown	?	1	305	D033000105	0.305		
	C91326F	1	NA	0.9275	0.8640	1000	184	R053000024	148			Unknown	NA	1	517	A041800068	0.517		
	C91334F	1	NA	0.9275	0.8640	100	824	D042100053	66.0			Unknown	NA	1	746	A041800069	0.746		
	C91346F	1	NA	0.9275	0.8640	1000	167	R053000025	134			Unknown	NA	1	577	A041800070	0.577		
	C91347F	1	NA	0.9275	0.8640	1000	395	R053000017	317			Unknown	NA	10	112	D010227020	1.12		
	C91352F	1	NA	0.9275	0.8640	1000	395	R053000018	317			Unknown	NA	10	109	D010227021	1.09		
	C91356F	1	NA	0.9275	0.8640	1000	164	R053000026	132			Unknown	NA	10	203	D042000055	2.03		
	C91359F	1	NA	0.9275	0.8640	1000	174	R053000039	139			Unknown	NA	1	637	A041800075	0.637		
	C91363F	1	NA	0.9275	0.8640	1000	154	R053000027	123			Unknown	NA	10	103	D042000057	1.03		
	C91367F	1	NA	0.9275	0.8640	1000	354	R053000019	284			Unknown	NA	10	159	D010227022	1.59		
	C91371F	1	NA	0.9275	0.8640	1000	228	R053000020	183			Unknown	NA	10	224	D010227023	2.24		
	C91372F	1	NA	0.9275	0.8640	100	698	D042100045	55.9		53.6	Unknown	NA	10	106	D042000058	1.06		48.9
	C91373F	1	NA	0.9275	0.8640	100	951	R053000038	76.2	163	87.2	Unknown	NA	10	125	D042000059	1.25	1.10	0.536

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

+ Recovery confirmed high with 08/21/00 analysis. LAC09/18/00
Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01
* CCVs were out; data may be biased low.

Date Entered/By: 04/17/00, 04/20/00, 04/24/0, 0/4/25/00, 05/04/00, 05/05/00, 06/01/00, 09/01/00, 09/18/00, 11/06/00, 03/07/01, 03/22/01 CSH/MMH/LAC
Date Verified/ By: 02/23/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Soup 020199, Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	2/25/00 RWW
Dates of Analysis/Analyst:	03/22/00, 03/30/00, 04/10/00, 04/18/00, 04/20/00, 04/21/00, 05/01/00, 05/30/00, 08/18/00, 08/21/00, 08/23/00, 02/27/01 CSH/MMH/IAS
Date of Data Reduction/Analyst:	04/12/00, 04/13/00, 04/19/00, 04/20/00, 04/21/00, 04/25/00, 05/03/00, 05/31/00, 08/21/00, 08/22/00, 08/25/00, 03/13/01 IAS/MMH/HOJ

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Dilution Factor	PFOSAA Conc. ng/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	EtFOSE-OH Purity Correction Factor	Surrogate Verified	EtFOSE-OH Dilution Factor	EtFOSE-OH Conc. ng/mL	Filename	Concentration of EtFOSE-OH ug/mL or % Rec	Mean EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD
		lot T-7121.1								lot Unknown/SD013							
Group 3	C90841M	Unknown	NA	10	431	A042100052	4.31			Unknown	NA	1	0.000	R041000104	<LOQ(0.00977 ug/mL) *		
Mid Dose	C90850M	Unknown	NA	10	323	A042100053	3.23			Unknown	NA	1	0.000	R041000105	<LOQ(0.00977 ug/mL) *		
30.0 mg/kg	C90854M	Unknown	NA	10	749	A042100054	7.49			Unknown	NA	1	0.000	R041000106	<LOQ(0.00977 ug/mL) *		
	C90855M	Unknown	NA	10	600	A042100055	6.00			Unknown	NA	1	0.000	R041000107	<LOQ(0.00977 ug/mL) *		
	C90856M	Unknown	NA	10	321	A042100056	3.21			Unknown	NA	1	0.000	R041000108	<LOQ(0.00977 ug/mL) *		
	C90859M	Unknown	NA	10	419	A042100060	4.19			Unknown	NA	1	0.000	R041000112	<LOQ(0.00977 ug/mL) *		
	C90873M	Unknown	NA	10	621	A042100061	6.21			Unknown	NA	1	0.000	R041000113	<LOQ(0.00977 ug/mL) *		
	C90879M	Unknown	NA	10	417	A042100062	4.17			Unknown	NA	1	0.000	R041000114	<LOQ(0.00977 ug/mL) *		
	C90881M	Unknown	NA	10	235	A042100063	2.35		35.9	Unknown	NA	1	0.000	R041000115	<LOQ(0.00977 ug/mL) *		NA
	C90896M	Unknown	NA	10	682	A042100064	6.82	4.80	1.72	Unknown	NA	1	0.000	R041000116	<LOQ(0.00977 ug/mL) *	<LOQ	NA
	C91255F	Unknown	NA	10	678	A042100068	6.78			Unknown	NA	1	24.1	D03300087	<LOQ (0.0248 ug/mL)		
	C91258F	Unknown	NA	10	306	A042100069	3.06			Unknown	NA	1	0.00	D03300088	<LOQ (0.0248 ug/mL)		
	C91264F	Unknown	NA	10	317	A042100070	3.17			Unknown	NA	1	26.6	D03300089	0.0266		
	C91269F	Unknown	NA	10	508	A042100071	5.08			Unknown	NA	1	0.00	D03300090	<LOQ (0.0248 ug/mL)		
	C91270F	Unknown	NA	10	457	A042100072	4.57			Unknown	NA	1	22.9	D03300091	<LOQ (0.0248 ug/mL)		
	C91274F	Unknown	NA	10	430	A042100076	4.30			Unknown	NA	1	23.7	D03300094	<LOQ (0.0248 ug/mL)		
	C91289F	Unknown	NA	10	310	A042100077	3.10			Unknown	NA	1	23.1	D03300095	<LOQ (0.0248 ug/mL)		
	C91294F	Unknown	NA	10	347	A042100078	3.47			Unknown	NA	1	24.5	D03300096	<LOQ (0.0248 ug/mL)		
	C91297F	Unknown	NA	10	435	A042100079	4.35		27.0	Unknown	NA	1	22.0	D03300097	<LOQ (0.0248 ug/mL)		9.76
	C91305F	Unknown	NA	10	431	A042100080	4.31	4.22	1.14	Unknown	NA	1	32.8	D03300098	0.0328	0.0258	0.00252
Group 4	C90902M	Unknown	NA	100	227	D082100023	22.7			Unknown	NA	1	31.8	D03300101	0.0318		
Mid-High Dose	C90908M	Unknown	NA	10	527.9	D042000031	5.28			Unknown	NA	1	27.4	D03300102	0.0274		
100 mg/kg	C90914M	Unknown	NA	10	590.7	D042000032	5.91			Unknown	NA	1	31.2	D03300103	0.0312		
	C90918M	Unknown	NA	100	152	D082100027	15.2			Unknown	NA	1	23.5	D03300104	<LOQ (0.0248 ug/mL)		
	C90923M	Unknown	NA	10	738	D042000033	7.4			Unknown	NA	1	24.0	D03300105	<LOQ (0.0248 ug/mL)		
	C90926M	Unknown	NA	10	502.6	D042000034	5.03			Unknown	NA	1	23.7	D03300108	<LOQ (0.0248 ug/mL)		
	C90927M	Unknown	NA	10	596.2	D042000035	5.96			Unknown	NA	1	29.0	D03300109	0.0290		
	C90934M	Unknown	NA	10	468.3	D042000039	4.68			Unknown	NA	1	28.1	D03300110	0.0281		
	C90936M	Unknown	NA	10	532.2	D042000040	5.32			Unknown	NA	1	31.6	D03300111	0.0316		
	C90944M	Unknown	NA	10	511.9	D042000041	5.12			Unknown	NA	1	25.6	D03300112	0.0256		
	C90946M	Unknown	NA	10	492.5	D042000042	4.92			Unknown	NA	1	3.77	A041800058	<LOQ (0.00493 ug/mL)		
	C90950M	Unknown	NA	100	220	D082100028	22.0			Unknown	NA	1	14.6	A041800059	0.0146		
	C90951M	Unknown	NA	100	118	D082100029	11.8			Unknown	NA	1	10.1	A041800060	0.0101		
	C90956M	Unknown	NA	10	396.0	D042000043	3.96		69.2	Unknown	NA	1	7.15	A041800061	0.00715		46.2
	C90957M	Unknown	NA	100	99.4	D082100030	9.94	9.0	6.23	Unknown	NA	1	6.10	A041800062	0.00610	0.0215	0.00992
	C91314F	Unknown	NA	10	837	D042000047	8.37			Unknown	NA	1	18.1	A041800066	0.0181		
	C91319F	Unknown	NA	10	702	D042000048	7.02			Unknown	NA	1	22.7	A041800067	0.0227		
	C91323F	Unknown	NA	100	201	D082300020	20.1			Unknown	?	1	24.0	D033000105	<LOQ (0.0248 ug/mL)		
	C91326F	Unknown	NA	10	647	D042000049	6.47			Unknown	NA	1	12.5	A041800068	0.0125		
	C91334F	Unknown	NA	10	655	D042000050	6.55			Unknown	NA	1	20.8	A041800069	0.0208		
	C91346F	Unknown	NA	10	672	D042000051	6.72			Unknown	NA	1	15.7	A041800070	0.0157		
	C91347F	Unknown	NA	100	152	D082300021	15.2			Unknown	NA	1	28.3	D010227027	0.0283		
	C91352F	Unknown	NA	100	215	D082300022	21.5			Unknown	NA	1	23.6	D010227028	0.0236		
	C91356F	Unknown	NA	100	223	D082100031	22.3			Unknown	NA	1	39.6	A041800074	0.0396		
	C91359F	Unknown	NA	10	825	D042000056	8.25			Unknown	NA	1	14.4	A041800075	0.0144		
	C91363F	Unknown	NA	10	818	D042000057	8.18			Unknown	NA	1	18.4	A041800076	0.0184		
	C91367F	Unknown	NA	100	230	D082300023	23.0			Unknown	NA	1	36.9	D010227029	0.0369		
	C91371F	Unknown	NA	100	197	D082300024	19.7			Unknown	NA	1	40.7	D010227030	0.0407		
	C91372F	Unknown	NA	10	978	D042000058	9.78		51.3	Unknown	NA	1	15.6	A041800077	0.0156		41.7
	C91373F	Unknown	NA	10	958	D042000059	9.58	12.8	6.59	Unknown	NA	1	15.7	A041800078	0.0157	0.0231	0.00963

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

+ Recovery confirmed high with 08/21/00 analysis. LAC 09/18/00
Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01
* CCVs were out; data may be biased low.

Date Entered/By: 04/17/00, 04/20/00, 04/24/00, 04/25/00, 05/04/00, 05/05/00, 06/01/00, 09/01/00, 09/18/00, 11/06/00, 03/07/01, 03/22/01 CSH/MMH/LAC
Date Verified/ By: 02/23/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Soup 020199, Ruby 100699
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	2/25/00 RWW
Dates of Analysis/Analyst:	03/22/00, 03/30/00, 04/10/00, 04/18/00, 04/20/00, 04/21/00, 05/01/00, 05/30/00, 08/18/00, 08/21/00, 08/23/00, 02/27/01 CSH/MMH/IAS
Date of Data Reduction/Analyst:	04/12/00, 04/13/00, 04/19/00, 04/20/00, 04/21/00, 04/25/00, 05/03/00, 05/31/00, 08/21/00, 08/22/00, 08/25/00, 03/13/01 IAS/MMH/HOJ

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	M556 Purity Correction Factor (lot NB113047-80)	Surrogate Verified	M556 Dilution Factor	M556 Conc. ng/mL	Filename	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	PFOSEA Purity Correction Factor (lot 529)	Surrogate Verified	PFOSEA Dilution Factor	PFOSEA Conc. ng/mL	Filename	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3 Mid Dose 30.0 mg/kg	C90841M	Unknown	NA	10	517	A042100052	5.17			Unknown	NA	1	0.00	R04100104	<LOQ (0.0492 ug/mL)		
	C90850M	Unknown	NA	10	356	A042100053	3.56			Unknown	NA	1	0.00	R04100105	<LOQ (0.0492 ug/mL)		
	C90854M	Unknown	NA	10	529	A042100054	5.29			Unknown	NA	1	0.00	R04100106	<LOQ (0.0492 ug/mL)		
	C90855M	Unknown	NA	10	346	A042100055	3.46			Unknown	NA	1	0.00	R04100107	<LOQ (0.0492 ug/mL)		
	C90856M	Unknown	NA	10	417	A042100056	4.17			Unknown	NA	1	0.00	R04100108	<LOQ (0.0492 ug/mL)		
	C90859M	Unknown	NA	10	528	A042100060	5.28			Unknown	NA	1	0.00	R04100112	<LOQ (0.0492 ug/mL)		
	C90873M	Unknown	NA	10	357	A042100061	3.57			Unknown	NA	1	0.00	R04100113	<LOQ (0.0492 ug/mL)		
	C90879M	Unknown	NA	10	242	A042100062	2.42			Unknown	NA	1	0.00	R04100114	<LOQ (0.0492 ug/mL)		
	C90881M	Unknown	NA	10	381	A042100063	3.81		24.3	Unknown	NA	1	0.00	R04100115	<LOQ (0.0492 ug/mL)		NA
	C90896M	Unknown	NA	10	324	A042100064	3.24	4.00	0.970	Unknown	NA	1	0.00	R04100116	<LOQ (0.0492 ug/mL)	<LOQ	NA
	C91255F	Unknown	NA	10	407	A042100068	4.07			Unknown	NA	1	0.00	D03300087	<LOQ (0.0247 ug/mL)		
	C91258F	Unknown	NA	10	264	A042100069	2.64			Unknown	NA	1	0.00	D03300088	<LOQ (0.0247 ug/mL)		
	C91264F	Unknown	NA	10	391	A042100070	3.91			Unknown	NA	1	0.00	D03300089	<LOQ (0.0247 ug/mL)		
	C91269F	Unknown	NA	10	545	A042100071	5.45			Unknown	NA	1	0.00	D03300090	<LOQ (0.0247 ug/mL)		
	C91270F	Unknown	NA	10	468	A042100072	4.68			Unknown	NA	1	0.00	D03300091	<LOQ (0.0247 ug/mL)		
	C91274F	Unknown	NA	10	396	A042100076	3.96			Unknown	NA	1	0.00	D03300094	<LOQ (0.0247 ug/mL)		
	C91289F	Unknown	NA	10	321	A042100077	3.21			Unknown	NA	1	0.00	D03300095	<LOQ (0.0247 ug/mL)		
	C91294F	Unknown	NA	10	425	A042100078	4.25			Unknown	NA	1	0.00	D03300096	<LOQ (0.0247 ug/mL)		
	C91297F	Unknown	NA	10	430	A042100079	4.30		18.8	Unknown	NA	1	0.00	D03300097	<LOQ (0.0247 ug/mL)		NA
	C91305F	Unknown	NA	10	451	A042100080	4.51	4.10	0.771	Unknown	NA	1	17.8	D03300098	<LOQ (0.0247 ug/mL)	<LOQ	NA
Group 4 Mid-High Dose 100 mg/kg	C90902M	Unknown	NA	100	224	D082100024	22.4			Unknown	NA	1	0.00	D03300101	<LOQ (0.0247 ug/mL)		
	C90908M	Unknown	NA	10	761	D042000031	7.6			Unknown	NA	1	0.00	D03300102	<LOQ (0.0247 ug/mL)		
	C90914M	Unknown	NA	10	877	D042000032	8.8			Unknown	NA	1	13.1	D03300103	<LOQ (0.0247 ug/mL)		
	C90918M	Unknown	NA	100	201	D082100027	20.1			Unknown	NA	1	0.00	D03300104	<LOQ (0.0247 ug/mL)		
	C90923M	Unknown	NA	10	604	D042000033	6.0			Unknown	NA	1	0.00	D03300105	<LOQ (0.0247 ug/mL)		
	C90926M	Unknown	NA	10	579.9	D042000034	5.80			Unknown	NA	1	0.00	D03300108	<LOQ (0.0247 ug/mL)		
	C90927M	Unknown	NA	10	901	D042000035	9.0			Unknown	NA	1	0.00	D03300109	<LOQ (0.0247 ug/mL)		
	C90934M	Unknown	NA	10	787	D042000039	7.9			Unknown	NA	1	0.00	D03300110	<LOQ (0.0247 ug/mL)		
	C90936M	Unknown	NA	10	624.6	D042000040	6.25			Unknown	NA	1	0.00	D03300111	<LOQ (0.0247 ug/mL)		
	C90944M	Unknown	NA	10	762	D042000041	7.6			Unknown	NA	1	15.0	D03300112	<LOQ (0.0247 ug/mL)		
	C90946M	Unknown	NA	10	532.7	D042000042	5.33			Unknown	NA	1	0.00	A041800058	<LOQ (0.00492 ug/mL)		
	C90950M	Unknown	NA	100	149	D082100028	14.9			Unknown	NA	1	0.00	A041800059	<LOQ (0.00492 ug/mL)		
	C90951M	Unknown	NA	100	164	D082100029	16.4			Unknown	NA	1	0.00	A041800060	<LOQ (0.00492 ug/mL)		
	C90956M	Unknown	NA	10	645.7	D042000043	6.46		52.7	Unknown	NA	1	0.00	A041800061	<LOQ (0.00492 ug/mL)		NA
	C90957M	Unknown	NA	100	116	D082100030	11.6	10.4	5.48	Unknown	NA	1	0.00	A041800062	<LOQ (0.00492 ug/mL)	<LOQ	NA
	C91314F	Unknown	NA	10	697	D042000047	6.97			Unknown	NA	1	0.00	A041800066	<LOQ (0.00492 ug/mL)		
	C91319F	Unknown	NA	10	761	D042000048	7.61			Unknown	NA	1	0.00	A041800067	<LOQ (0.00492 ug/mL)		
	C91323F	Unknown	NA	100	198	D082300020	19.8			Unknown	NA	1	0.00	D03300105	<LOQ (0.0247 ug/mL)		
	C91326F	Unknown	NA	10	574	D042000049	5.74			Unknown	NA	1	0.00	A041800068	<LOQ (0.00492 ug/mL)		
	C91334F	Unknown	NA	10	927	D042000050	9.27			Unknown	NA	1	0.00	A041800069	<LOQ (0.00492 ug/mL)		
	C91346F	Unknown	NA	10	648	D042000051	6.48			Unknown	NA	1	0.00	A041800070	<LOQ (0.00492 ug/mL)		
	C91347F	Unknown	NA	100	147	D082300021	14.7			Unknown	NA	1	0.00	D010227027	<LOQ (0.00999 ug/mL)		
	C91352F	Unknown	NA	100	178	D082300022	17.8			Unknown	NA	1	2.52	D010227028	<LOQ (0.00999 ug/mL)		
	C91356F	Unknown	NA	100	258	D082100031	25.8			Unknown	NA	1	0.00	A041800074	<LOQ (0.00492 ug/mL)		
	C91359F	Unknown	NA	10	708	D042000056	7.08			Unknown	NA	1	0.00	A041800075	<LOQ (0.00492 ug/mL)		
	C91363F	Unknown	NA	10	835	D042000057	8.35			Unknown	NA	1	0.00	A041800076	<LOQ (0.00492 ug/mL)		
	C91367F	Unknown	NA	100	176	D082300023	17.6			Unknown	NA	1	5.60	D010227029	<LOQ (0.00999 ug/mL)		
	C91371F	Unknown	NA	100	251	D082300024	25.1			Unknown	NA	1	0.00	D010227030	<LOQ (0.00999 ug/mL)		
	C91372F	Unknown	NA	10	901	D042000058	9.01		55.5	Unknown	NA	1	0.00	A041800077	<LOQ (0.00492 ug/mL)		NA
	C91373F	Unknown	NA	10	723	D042000059	7.23	12.6	6.98	Unknown	NA	1	0.00	A041800078	<LOQ (0.00492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

+ Recovery confirmed high with 08/21/00 analysis. LAC 09/18/00
Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01
* CCVs were out; data may be biased low.

Date Entered/By:	04/17/00, 04/20/00, 04/24/0, 0/4/25/00, 05/04/00, 05/05/00, 06/01/00, 09/01/00, 09/18/00, 11/06/00, 03/07/01, 03/22/01 CSH/MMH/LAC
Date Verified/By:	02/23/01 KJH / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Serum
Method/Revision: ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number: Amelia 062498, Soup 020199, Ruby 100699
Instrument Software/Version: Masslynx 3.3 & 3.4
Filename: See Attachments
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Dates of Extraction/Analyst: 2/25/00 RWW
Dates of Analysis/Analyst: 03/22/00, 03/30/00, 04/10/00, 04/18/00, 04/20/00, 04/21/00, 05/01/00, 05/30/00, 08/18/00, 08/21/00, 08/23/00, 02/27/01 CSH/MMH/IAS
Date of Data Reduction/Analyst: 04/12/00, 04/13/00, 04/19/00, 04/20/00, 04/21/00, 04/25/00, 05/03/00, 05/31/00, 08/21/00, 08/22/00, 08/25/00, 03/13/01 IAS/MMH/HOJ

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of EtFOSE-OH ug/mL or % Rec	Mean EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90841M	16.7			0.201			4.31			<LOQ(0.00977 ug/mL) *			5.17			<LOQ (0.0492 ug/mL)		
Mid Dose	C90850M	20.5			0.209			3.23			<LOQ(0.00977 ug/mL) *			3.56			<LOQ (0.0492 ug/mL)		
30.0 mg/kg	C90854M	26.4			0.338			7.49			<LOQ(0.00977 ug/mL) *			5.29			<LOQ (0.0492 ug/mL)		
	C90855M	14.3			0.293			6.00			<LOQ(0.00977 ug/mL) *			3.46			<LOQ (0.0492 ug/mL)		
	C90856M	17.8			0.181			3.21			<LOQ(0.00977 ug/mL) *			4.17			<LOQ (0.0492 ug/mL)		
	C90859M	24.5			0.231			4.19			<LOQ(0.00977 ug/mL) *			5.28			<LOQ (0.0492 ug/mL)		
	C90873M	27.3			0.252			6.21			<LOQ(0.00977 ug/mL) *			3.57			<LOQ (0.0492 ug/mL)		
	C90879M	3.58			0.125			4.17			<LOQ(0.00977 ug/mL) *			2.42			<LOQ (0.0492 ug/mL)		
	C90881M	12.1		39.8	0.180		31.6	2.35		35.9	<LOQ(0.00977 ug/mL) *		NA	3.81		24.3	<LOQ (0.0492 ug/mL)		NA
	C90896M	17.2	18.0	7.18	0.365	0.237	0.0751	6.82	4.80	1.72	<LOQ(0.00977 ug/mL) *	<LOQ	NA	3.24	4.00	0.970	<LOQ (0.0492 ug/mL)	<LOQ	NA
	C91255F	56.2			0.393			6.78			<LOQ (0.0248 ug/mL)			4.07			<LOQ (0.0247 ug/mL)		
	C91258F	27.4			0.268			3.06			<LOQ (0.0248 ug/mL)			2.64			<LOQ (0.0247 ug/mL)		
	C91264F	29.5			0.324			3.17			0.0266			3.91			<LOQ (0.0247 ug/mL)		
	C91269F	39.3			0.489			5.08			<LOQ (0.0248 ug/mL)			5.45			<LOQ (0.0247 ug/mL)		
	C91270F	11.4			0.485			4.57			<LOQ (0.0248 ug/mL)			4.68			<LOQ (0.0247 ug/mL)		
	C91274F	35.4			0.404			4.30			<LOQ (0.0248 ug/mL)			3.96			<LOQ (0.0247 ug/mL)		
	C91289F	38.1			0.279			3.10			<LOQ (0.0248 ug/mL)			3.21			<LOQ (0.0247 ug/mL)		
	C91294F	29.1			0.283			3.47			<LOQ (0.0248 ug/mL)			4.25			<LOQ (0.0247 ug/mL)		
	C91297F	38.5		33.8	0.415		22.5	4.35		27.0	<LOQ (0.0248 ug/mL)		9.76	4.30		18.8	<LOQ (0.0247 ug/mL)		NA
	C91305F	31.4	33.6	11.4	0.323	0.366	0.0824	4.31	4.22	1.14	0.0328	0.0258	0.00252	4.51	4.10	0.771	<LOQ (0.0247 ug/mL)	<LOQ	NA
Group 4	C90902M	70.5			0.615			22.7			0.0318			22.4			<LOQ (0.0247 ug/mL)		
Mid-High Dose	C90908M	55.8			0.275			5.28			0.0274			7.6			<LOQ (0.0247 ug/mL)		
100 mg/kg	C90914M	54.5			0.382			5.91			0.0312			8.8			<LOQ (0.0247 ug/mL)		
	C90918M	67.1			0.662			15.2			<LOQ (0.0248 ug/mL)			20.1			<LOQ (0.0247 ug/mL)		
	C90923M	62.3			0.305			7.4			<LOQ (0.0248 ug/mL)			6.0			<LOQ (0.0247 ug/mL)		
	C90926M	49.1			0.223			5.03			<LOQ (0.0248 ug/mL)			5.80			<LOQ (0.0247 ug/mL)		
	C90927M	60.2			0.363			5.96			0.0290			9.0			<LOQ (0.0247 ug/mL)		
	C90934M	37.2			0.247			4.68			0.0281			7.9			<LOQ (0.0247 ug/mL)		
	C90936M	51.3			0.331			5.32			0.0316			6.25			<LOQ (0.0247 ug/mL)		
	C90944M	60.9			0.270			5.12			0.0256			7.6			<LOQ (0.0247 ug/mL)		
	C90946M	60.1			0.231			4.92			<LOQ (0.00493 ug/mL)			5.33			<LOQ (0.00492 ug/mL)		
	C90950M	264			0.474			22.0			0.0146			14.9			<LOQ (0.00492 ug/mL)		
	C90951M	220			0.343			11.8			0.0101			16.4			<LOQ (0.00492 ug/mL)		
	C90956M	49.2		80.1	0.203		38.7	3.96		69.2	0.0072		46.2	6.46		52.7	<LOQ (0.00492 ug/mL)		NA
	C90957M	73.0	82.4	66.0	0.430	0.357	0.138	9.94	9.0	6.23	0.0061	0.0215	0.00992	11.6	10.4	5.48	<LOQ (0.00492 ug/mL)	<LOQ	NA
	C91314F	175			1.05			8.37			0.0181			6.97			<LOQ (0.00492 ug/mL)		
	C91319F	73.5			1.20			7.02			0.0227			7.61			<LOQ (0.00492 ug/mL)		
	C91323F	220			0.31			20.1			<LOQ (0.0248 ug/mL)			19.8			<LOQ (0.0247 ug/mL)		
	C91326F	148			0.517			6.47			0.0125			5.74			<LOQ (0.00492 ug/mL)		
	C91334F	66.0			0.746			6.55			0.0208			9.27			<LOQ (0.00492 ug/mL)		
	C91346F	134			0.577			6.72			0.0157			6.48			<LOQ (0.00492 ug/mL)		
	C91347F	317			1.12			15.2			0.0283			14.7			<LOQ (0.00999 ug/mL)		
	C91352F	317			1.09			21.5			0.0236			17.8			<LOQ (0.00999 ug/mL)		
	C91356F	132			2.03			22.3			0.0396			25.8			<LOQ (0.00492 ug/mL)		
	C91359F	139			0.637			8.25			0.0144			7.08			<LOQ (0.00492 ug/mL)		
	C91363F	123			1.03			8.18			0.0184			8.35			<LOQ (0.00492 ug/mL)		
	C91367F	284			1.59			23.0			0.0369			17.6			<LOQ (0.00999 ug/mL)		
	C91371F	183			2.24			19.7			0.0407			25.1			<LOQ (0.00999 ug/mL)		
	C91372F	55.9		53.6	1.06		48.9	9.78		51.3	0.0156		41.7	9.01		55.5	<LOQ (0.00492 ug/mL)		NA
	C91373F	76.2	163	87.2	1.25	1.095	0.536	9.58	12.8	6.59	0.0157	0.0231	0.0096	7.23	12.6	6.98	<LOQ (0.00492 ug/mL)	<LOQ	NA

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PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
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new LOQ is 0.00394 ug/mL. LAC 02/19/01
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Date Entered/By: 04/17/00, 04/20/00, 04/24/0, 04/25/00, 05/04/00, 05/05/00, 06/01/00, 09/01/00, 09/18/00, 11/06/00, 03/07/01, 03/22/01 CSH/MMH/LAC
Date Verified/ By: 02/23/01 KJH / 04/30/01 LAC
Purity Entered/Verified: 02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 SAL/RWW
Dates of Analysis/Analyst:	02/18/00, 03/15/00, 4/18/00, 4/19/00, 06/19/00, 06/20/00, 08/16/00, 08/23/00 MMH/IAS/CSH
Date of Data Reduction/Analyst:	02/21/00, 03/16/00, 4/19/00, 4/20/00, 06/20/00, 06/21/00, 08/18/00, 08/25/00, 04/11/01 MMH/IAS/HOJ
Sample Data	**Box** 00-009

WEEK 105 RAT SERA lot 171

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec	Average PFOS ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	020700-H2O Blk-5	1	NA	0.9275	0.8640	1	0.00	D082300049	<LOQ (0.00394 ug/mL)		
	020700-H2O Blk-6	1	NA	0.9275	0.8640	1	0.00	D082300050	<LOQ (0.00394 ug/mL)	<LOQ	NA
	020800-H2O Blk-1	1	NA	0.9275	0.8640	1	0.00	D081600020	<LOQ (0.00394 ug/mL)		
	020800-H2O Blk-2	1	NA	0.9275	0.8640	1	0.00	D031500025	<LOQ (0.00394 ug/mL)	<LOQ	NA
	020900-H2O Blk-5	1	NA	0.9275	0.8640	1	0.00	A062000005	<LOQ (0.00394 ug/mL)		
	020900-H2O Blk-6	1	NA	0.9275	0.8640	1	0.00	A062000019	<LOQ (0.00394 ug/mL)	<LOQ	NA
Matrix Blk	020700-RBS Blk-5	1	NA	0.9275	0.8640	1	0.00	D082300051	<LOQ (0.00394 ug/mL)		
	020700-RBS Blk-6	1	NA	0.9275	0.8640	1	0.00	D082300052	<LOQ (0.00394 ug/mL)	<LOQ	NA
	020800-RBS Blk-1	1	X	0.9275	0.8640	1	0.00	A031500006	<LOQ (0.00394 ug/mL)		
	020800-RBS-Blk-2	1	NA	0.9275	0.8640	1	0.00	D031500026	<LOQ (0.00394 ug/mL)	<LOQ	NA
	020800-RTS Blk-1	1	X	0.9275	0.8640	1	0.760	A031500007	<LOQ (0.00394 ug/mL)		
	020800-RTS-Blk-2	1	NA	0.9275	0.8640	1	0.230	D031500027	<LOQ (0.00394 ug/mL)	<LOQ	NA
	020900-RBS Blk-5	1	NA	0.9275	0.8640	1	0.00	A062000006	0.00		
	020900-RBS-Blk-6	1	NA	0.9275	0.8640	1	0.00	A062000020	0.00	<LOQ	NA
	020900-RTS Blk-5	1	NA	0.9275	0.8640	1	0.00	A062000007	0.00		
	020900-RTS-Blk-6	1	Confirmed High	0.9275	0.8640	1	0.00	A062000021	0.00	<LOQ	NA
QC-250 ppb	020700-RTS-MS-5	1	NA	NA	NA	1	300	A022600017	121%		
	020700-RTS-MSD-5	1	NA	NA	NA	1	300	A022600018	121%	121%	0%
	020700-RTS-MS-5	1	NA	NA	NA	NA	NA	NA	NA		
	020700-RTS-MSD-5	1	NA	NA	NA	NA	NA	NA	NA	NA	NA
	020700-RTS-MS-6	1	NA	NA	NA	1	297	A022600019	120%		
	020700-RTS-MSD-6	1	NA	NA	NA	1	249	A022600020	101%	110%	17%
	020700-RTS-MS-6	1	NA	NA	NA	NA	NA	NA	NA		
	020700-RTS-MSD-6	1	NA	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-1	1	NA	NA	NA	1	198	A021800017	80%		
	020800-RTS-MSD-1	1	NA	NA	NA	1	206	D081600027	83%	82%	4%
	020800-RTS-MS-1	1	NA	NA	NA	NA	NA	NA	NA		
	020800-RTS-MSD-1	1	NA	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-2	1	NA	NA	NA	1	200	A021800018	81%		
	020800-RTS-MSD-2	1	NA	NA	NA	1	190	D081600023	77%	79%	5%
	020800-RTS-MS-2	1	NA	NA	NA	NA	NA	NA	NA		
	020800-RTS-MSD-2	1	NA	NA	NA	NA	NA	NA	NA	NA	NA
	C90778M-MS-5	1	NA	NA	NA	4	343	A062000054	72%		
	C90778M-MSD-6	1	NA	NA	NA	4	345	A062000055	73%	72%	1%
	020900-RTS-MS-5	1	NA	NA	NA	1	239	A201800019	97%		
	020900-RTS-MSD-6	1	NA	NA	NA	1	249	A021800020	101%	99%	4%
	020900-RTS-MS-5	1	NA	NA	NA	NA	NA	NA	NA		
	020900-RTS-MSD-6	1	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 1	C90712M	1	NA	0.9275	0.8640	1	3.62	D031500043	<LOQ (0.00394 ug/mL)		
Control	C90716M	1	NA	0.9275	0.8640	1	0.00	D031500057	<LOQ (0.00394 ug/mL)		
0.0 mg/kg	C90722M	1	NA	0.9275	0.8640	1	8.49	D031500058	0.00680		
	C90724M	1	NA	0.9275	0.8640	1	14.5	A021800039	0.0116		
	C90726M	1	NA	0.9275	0.8640	1	3.36	D031500044	<LOQ (0.00394 ug/mL)		
	C90728M	1	NA	0.9275	0.8640	1	16.4	A021800040	0.0131		
	C90735M	1	NA	0.9275	0.8640	1	2.65	D031500061	<LOQ (0.00394 ug/mL)		
	C90742M	1	NA	0.9275	0.8640	1	5.40	D031500064	0.00433		
	C90743M	1	NA	0.9275	0.8640	1	1.48	D031500045	<LOQ (0.00394 ug/mL)		
	C90747M	1	NA	0.9275	0.8640	1	8.06	D031500065	0.00646		
	C90750M	1	NA	0.9275	0.8640	1	62.9	A021800026	0.0504		
	C90758M	1	NA	0.9275	0.8640	1	2.53	D031500047	<LOQ (0.00394 ug/mL)		
	C90765M	1	NA	0.9275	0.8640	1	1.01	D031500066	<LOQ (0.00394 ug/mL)		131
	C90778M	1	NA	0.9275	0.8640	1	13.4	A021800058	0.0107	0.00936	0.0123
Group 1	C91127F	1	NA	0.9275	0.8640	1	51.6	A021800047	0.0413		
Control	C91131F	1	NA	0.9275	0.8640	1	42.7	A021800048	0.0342		
0.0 mg/kg	C91138F	1	NA	0.9275	0.8640	1	11.9	A021800059	0.00951		
	C91144F	1	NA	0.9275	0.8640	1	108	A021800030	0.0868		
	C91148F	1	NA	0.9275	0.8640	1	27.0	A021800051	0.0217		
	C91157F	1	NA	0.9275	0.8640	1	21.4	A021800031	0.0171		
	C91159F	1	NA	0.9275	0.8640	1	41.5	A021800052	0.0333		
	C91166F	1	NA	0.9275	0.8640	1	21.4	A021800032	0.0171		
	C91172F	1	NA	0.9275	0.8640	1	10.8	A021800053	0.00865		
	C91176F	1	NA	0.9275	0.8640	1	0.00	D031500074	<LOQ (0.00394 ug/mL)		
	C91183F	1	NA	0.9275	0.8640	1	23.6	A021800055	0.0189		
	C91188F	1	NA	0.9275	0.8640	1	69.9	A021800033	0.0560		87.2
	C91190F	1	NA	0.9275	0.8640	1	0.00	D031500054	<LOQ (0.00394 ug/mL)	0.0271	0.0237
Group 2	C90781M	1	NA	0.9275	0.8640	1	102	A021800081	0.0819		
Low Dose	C90782M	1	NA	0.9275	0.8640	1	506	A021800060	0.405		
3.00 mg/kg	C90783M	1	NA	0.9275	0.8640	10	189	A031500027	1.52		
	C90785M	1	NA	0.9275	0.8640	1	83.0	A021800064	0.0665		
	C90787M	1	NA	0.9275	0.8640	10	158	A031500028	1.26		
	C90788M	1	NA	0.9275	0.8640	10	460	A031500029	3.69		
	C90791M	1	NA	0.9275	0.8640	15	157	A031500026	1.89		
	C90794M	1	NA	0.9275	0.8640	10	273	A031500033	2.19		
	C90799M	1	NA	0.9275	0.8640	1	627	A021800082	0.502		
	C90804M	1	NA	0.9275	0.8640	1	398	A021800067	0.319		
	C90809M	1	NA	0.9275	0.8640	15	382	A031500023	4.59		
	C90810M	1	NA	0.9275	0.8640	1	177	A021800068	0.142		
	C90811M	1	NA	0.9275	0.8640	1	379	A021800085	0.304		
	C90812M	1	NA	0.9275	0.8640	1	218	A021800086	0.175		
	C90815M	1	NA	0.9275	0.8640	1	408	A021800087	0.327		
	C90823M	1	NA	0.9275	0.8640	10	108	A031500035	0.864		
	C90829M	1	NA	0.9275	0.8640	10	177	A031500036	1.42		
	C90830M	1	NA	0.9275	0.8640	50	114	R041800049	4.56		
	C90832M	1	NA	0.9275	0.8640	10	109	A031500040	0.872		
	C90835M	1	NA	0.9275	0.8640	10	36.6	A031500041	0.293		
	C90838M	1	NA	0.9275	0.8640	1	229	A021800074	0.184		120
	C90840M	1	NA	0.9275	0.8640	1	268	A021800089	0.215	1.18	1.41
Group 2	C91197F	1	NA	0.9275	0.8640	10	198	A031500042	1.59		
Low Dose	C91201F	1	NA	0.9275	0.8640	10	187	A031500043	1.50		
3.00 mg/kg	C91207F	1	NA	0.9275	0.8640	15	490	A031500022	5.89		
	C91216F	1	E	E	0.8640	E	E	E	E		
	C91220F	1	NA	0.9275	0.8640	15	401	A031500021	4.82		
	C91221F	1	NA	0.9275	0.8640	10	119	A031500048	0.955		
	C91227F	1	NA	0.9275	0.8640	50	103	R041800051	4.12		
	C91232F	1	NA	0.9275	0.8640	15	384	A031500020	4.62		
	C91235F	1	NA	0.9275	0.8640	80	156	R041800050	9.98		
	C91236F	1	NA	0.9275	0.8640	50	92.3	R041800052	3.70		
	C91242F	1	NA	0.9275	0.8640	10	372	A031500055	2.98		70.7
	C91243F	1	NA	0.9275	0.8640	10	141	A031500056	1.13	3.75	2.65

A= CCVs analyzed bracketing these data were within criteria but had peak areas slightly above the valid linear range. LAC 05/03/01
E = Lost during extraction, no sample remaining to reextract.

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/17/00, 5/16/00, 06/22/00, 06/23/00, 06/26/00, 09/05/00, 10/17/00, 04/13/01 LAC/CSH 4/17/01 mmh
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 SAL/RWW
Dates of Analysis/Analyst:	02/18/00, 03/15/00, 4/18/00, 4/19/00, 06/19/00, 06/20/00, 08/16/00, 08/23/00 MMH/IAS/CSH
Date of Data Reduction/Analyst:	02/21/00, 03/16/00, 4/19/00, 4/20/00, 06/20/00, 06/21/00, 08/18/00, 08/25/00, 04/11/01 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot L-15709

Group Dose	Sample #	PFOSA Purity Correction Factor	Surrogate Verified	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Average PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	020700-H2O Blk-5	Unknown	NA	1	0.000	D081600016	<LOQ (0.00493 ug/mL)		
	020700-H2O Blk-6	Unknown	NA	1	0.000	D081600017	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020800-H2O Blk-1	Unknown	2nd Analysis OK	1	0.000	D081600020	<LOQ (0.00493 ug/mL)		
	020800-H2O Blk-2	Unknown	NA	1	0.920	A021800110	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020900-H2O Blk-5	Unknown	NA	1	0.000	A062000005	<LOQ (0.00493 ug/mL)		
	020900-H2O Blk-6	Unknown	NA	1	0.000	A062000019	<LOQ (0.00493 ug/mL)	<LOQ	NA
Matrix Blk	020700-RBS Blk-5	Unknown	NA	1	0.000	D081600018	<LOQ (0.00493 ug/mL)		
	020700-RBS Blk-6	Unknown	NA	1	0.000	D081500019	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020800-RBS Blk-1	Unknown	X	1	0.790	A021800004	<LOQ (0.00493 ug/mL)		
	020800-RBS-Blk-2	Unknown	NA	1	0.710	A021800111	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020800-RTS Blk-1	Unknown	X	1	0.980	A021800005	<LOQ (0.00493 ug/mL)		
	020800-RTS-Blk-2	Unknown	NA	1	0.790	A021800112	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020900-RBS Blk-5	Unknown	NA	1	0.000	A062000006	<LOQ (0.00493 ug/mL)		
	020900-RBS-Blk-6	Unknown	NA	1	0.000	A062000020	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020900-RTS Blk-5	Unknown	NA	1	0.000	A062000007	<LOQ (0.00493 ug/mL)		
	020900-RTS-Blk-6	Unknown	Confirmed High	1	0.000	A062000021	<LOQ (0.00493 ug/mL)	<LOQ	NA
QC-250 ppb	020700-RTS-MS-5	NA	NA	1	349	A022500017	141%		
	020700-RTS-MSD-5	NA	NA	1	340	A022500018	137%	139%	3%
	020700-RTS-MS-5	NA	NA	1	305	D082300057	123% 2nd		
	020700-RTS-MSD-5	NA	NA	1	310	D082300058	125% 2nd	124%	2%
	020700-RTS-MS-6	NA	NA	1	354	A022500019	143%		
	020700-RTS-MSD-6	NA	NA	1	346	A022500020	140%	141%	2%
	020700-RTS-MS-6	NA	NA	1	318	D082300059	128% 2nd		
	020700-RTS-MSD-6	NA	NA	1	308	D082300060	124% 2nd	126%	3%
	020800-RTS-MS-1	NA	NA	1	230	A021800017	93%		
	020800-RTS-MSD-1	NA	NA	1	272	D081600027	110%	101%	17%
	020800-RTS-MS-1	NA	NA	NA	NA	NA	NA		
	020800-RTS-MSD-1	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-2	NA	NA	1	229	A021800018	93%		
	020800-RTS-MSD-2	NA	NA	1	259	D081600023	105%	99%	12%
	020800-RTS-MS-2	NA	NA	NA	NA	NA	NA		
	020800-RTS-MSD-2	NA	NA	NA	NA	NA	NA	NA	NA
	C90778M-MS-5	NA	NA	4	342	D081600024	72%		
	C90778M-MSD-6	NA	NA	4	341	D081600025	72%	72%	0%
	020900-RTS-MS-5	NA	NA	1	265	A201800019	107%		
	020900-RTS-MSD-6	NA	NA	1	257	A021800020	104%	105%	3%
	020900-RTS-MS-5	NA	NA	NA	NA	NA	NA		
	020900-RTS-MSD-6	NA	NA	NA	NA	NA	NA	NA	NA
Group 1 Control 0.0 mg/kg	C90712M	Unknown	NA	1	0.770	A021800023	<LOQ (0.00493 ug/mL)		
	C90716M	Unknown	NA	1	0.940	A021800037	<LOQ (0.00493 ug/mL)		
	C90722M	Unknown	NA	1	0.800	A021800038	<LOQ (0.00493 ug/mL)		
	C90724M	Unknown	NA	1	0.960	A021800039	<LOQ (0.00493 ug/mL)		
	C90726M	Unknown	NA	1	0.830	A021800024	<LOQ (0.00493 ug/mL)		
	C90728M	Unknown	NA	1	0.930	A021800040	<LOQ (0.00493 ug/mL)		
	C90735M	Unknown	NA	1	0.800	A021800041	<LOQ (0.00493 ug/mL)		
	C90742M	Unknown	NA	1	0.960	A021800044	<LOQ (0.00493 ug/mL)		
	C90743M	Unknown	NA	1	0.990	A021800025	<LOQ (0.00493 ug/mL)		
	C90747M	Unknown	NA	1	0.950	A021800045	<LOQ (0.00493 ug/mL)		
	C90750M	Unknown	NA	1	0.770	A021800026	<LOQ (0.00493 ug/mL)		
	C90758M	Unknown	NA	1	0.670	A021800027	<LOQ (0.00493 ug/mL)		
	C90765M	Unknown	NA	1	0.620	A021800046	<LOQ (0.00493 ug/mL)		NA
	C90778M	Unknown	NA	1	0.960	A021800058	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	Unknown	NA	1	0.900	A021800047	<LOQ (0.00493 ug/mL)		
	C91131F	Unknown	NA	1	0.930	A021800048	<LOQ (0.00493 ug/mL)		
	C91138F	Unknown	NA	1	0.790	A021800059	<LOQ (0.00493 ug/mL)		
	C91144F	Unknown	NA	1	1.55	A021800030	<LOQ (0.00493 ug/mL)		
	C91148F	Unknown	NA	1	1.05	A021800051	<LOQ (0.00493 ug/mL)		
	C91157F	Unknown	NA	1	1.08	A021800031	<LOQ (0.00493 ug/mL)		
	C91159F	Unknown	NA	1	0.740	A021800052	<LOQ (0.00493 ug/mL)		
	C91166F	Unknown	NA	1	0.770	A021800032	<LOQ (0.00493 ug/mL)		
	C91172F	Unknown	NA	1	0.770	A021800053	<LOQ (0.00493 ug/mL)		
	C91176F	Unknown	NA	1	0.750	A021800054	<LOQ (0.00493 ug/mL)		
	C91183F	Unknown	NA	1	0.860	A021800055	<LOQ (0.00493 ug/mL)		
	C91188F	Unknown	NA	1	0.860	A021800033	<LOQ (0.00493 ug/mL)		NA
	C91190F	Unknown	NA	1	0.920	A021800034	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.00 mg/kg	C90781M	Unknown	NA	1	6.70	A021800081	0.00670		
	C90782M	Unknown	NA	1	12.8	A021800060	0.0128		
	C90783M	Unknown	NA	1	30.4	A021800061	0.0304		
	C90785M	Unknown	NA	1	21.7	A021800064	0.0217		
	C90787M	Unknown	NA	1	12.5	A021800065	0.0125		
	C90788M	Unknown	NA	1	39.5	A021800100	0.0395		
	C90791M	Unknown	NA	1	13.9	A021800101	0.0139		
	C90794M	Unknown	NA	1	37.4	A021800066	0.0374		
	C90799M	Unknown	NA	1	9.00	A021800082	0.00900		
	C90804M	Unknown	NA	1	7.28	A021800067	0.00728		
	C90809M	Unknown	NA	1	33.9	A021800102	0.0339		
	C90810M	Unknown	NA	1	21.3	A021800068	0.0213		
	C90811M	Unknown	NA	1	2.70	A021800085	<LOQ (4.93 ng/mL)		
	C90812M	Unknown	NA	1	5.90	A021800086	0.00590		
	C90815M	Unknown	NA	1	5.15	A021800087	0.00515		
	C90823M	Unknown	NA	1	6.67	A021800088	0.00667		
	C90829M	Unknown	NA	1	22.7	A021800071	0.0227		
	C90830M	Unknown	NA	1	37.0	A021800072	0.0370		
	C90832M	Unknown	NA	1	31.4	A021800073	0.0314		
	C90835M	Unknown	NA	1	19.6	A021800103	0.0196		
	C90838M	Unknown	NA	1	11.5	A021800074	0.0115		64.0
	C90840M	Unknown	NA	1	5.74	A021800089	0.00574	0.0187	0.0119
Group 2 Low Dose 3.00 mg/kg	C91197F	Unknown	NA	1	17.0	A021800092	0.0170		
	C91201F	Unknown	NA	1	13.3	A021800093	0.0133		
	C91207F	Unknown	NA	1	49.2	A021800075	0.0492		
	C91216F	Unknown	E	E	E	E	E		
	C91220F	Unknown	NA	1	50.4	A021800106	0.0504		
	C91221F	Unknown	NA	1	9.43	A021800094	0.00943		
	C91227F	Unknown	NA	1	16.6	A021800095	0.0166		
	C91232F	Unknown	NA	1	253	A021800078	0.253		
	C91235F	Unknown	NA	1	309	A021800079	0.309		
	C91236F	Unknown	NA	1	129	A021800096	0.129		
	C91242F	Unknown	NA	1	62.1	A021800099	0.0621		123
	C91243F	Unknown	NA	1	22.0	A021800080	0.0220	0.0846	0.104

A= CCVs analyzed bracketing these data were within criteria but had peak areas slightly above the valid linear range. LAC 05/03/01

E = Lost during extraction, no sample remaining to reextract.

PFOS = Perfluorooctanesulfonate

PFOSA = Perfluorooctanesulfonamide

PFOSAA = Perfluorooctanesulfonamidoacetate

EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

M556 = C8F17SO2N((H)CH2COO)

PFOSEA = Perfuoroactane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/17/00, 5/16/00, 06/22/00, 06/23/00, 06/26/00, 09/05/00, 10/17/00, 04/13/01 LAC/CSH 4/17/01 mmh
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 SAL/RWW
Dates of Analysis/Analyst:	02/18/00, 03/15/00, 4/18/00, 4/19/00, 06/19/00, 06/20/00, 08/16/00, 08/23/00 MMH/IAS/CSH
Date of Data Reduction/Analyst:	02/21/00, 03/16/00, 4/19/00, 4/20/00, 06/20/00, 06/21/00, 08/18/00, 08/25/00, 04/11/01 MMH/IAS/HOJ
Sample Data	Box 00-009

WEEK 105 RAT SERA lot T-7121.1

Group Dose	Sample #	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Dilution Factor	PFOSAA Conc. ng/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Average PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	020700-H2O Blk-5	Unknown	NA	1	0.00	D081600016	<LOQ (0.00493 ug/mL) A		
	020700-H2O Blk-6	Unknown	NA	1	0.00	D081600017	<LOQ (0.00493 ug/mL) A	<LOQ	NA
	020800-H2O Blk-1	Unknown	2nd Analysis OK	1	0.00	A021800003	<LOQ (0.00493 ug/mL)		
	020800-H2O Blk-2	Unknown	NA	1	0.00	A021800110	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020900-H2O Blk-5	Unknown	NA	1	0.00	A062000005	<LOQ (0.0248 ug/mL)		
	020900-H2O Blk-6	Unknown	NA	1	0.00	A062000019	<LOQ (0.0248 ug/mL)	<LOQ	NA
Matrix Blk	020700-RBS Blk-5	Unknown	NA	1	0.00	D081600018	<LOQ (0.00493 ug/mL) A		
	020700-RBS Blk-6	Unknown	NA	1	0.00	D081600019	<LOQ (0.00493 ug/mL) A	<LOQ	NA
	020800-RBS Blk-1	Unknown	X	1	0.00	A021800004	<LOQ (0.00493 ug/mL)		
	020800-RBS-Blk-2	Unknown	NA	1	0.00	A021800111	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020800-RTS Blk-1	Unknown	X	1	0.00	A021800005	<LOQ (0.00493 ug/mL)		
	020800-RTS-Blk-2	Unknown	NA	1	0.00	A021800112	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020900-RBS Blk-5	Unknown	NA	1	0.00	A062000006	<LOQ (0.0248 ug/mL)		
	020900-RBS-Blk-6	Unknown	NA	1	0.00	A062000020	<LOQ (0.0248 ug/mL)	<LOQ	NA
	020900-RTS Blk-5	Unknown	NA	1	0.00	A062000007	<LOQ (0.0248 ug/mL)		
	020900-RTS-Blk-6	Unknown	Confirmed High	1	0.00	A062000021	<LOQ (0.0248 ug/mL)	<LOQ	NA
QC-250 ppb	020700-RTS-MS-5	NA	NA	1	310	A022500017	125%		
	020700-RTS-MSD-5	NA	NA	1	306	A022500018	123%	124%	2%
	020700-RTS-MS-5	NA	NA	NA	NA	NA	NA		
	020700-RTS-MSD-5	NA	NA	NA	NA	NA	NA	NA	NA
	020700-RTS-MS-6	NA	NA	1	312	A022600019	126%		
	020700-RTS-MSD-6	NA	NA	1	305	A022600020	123%	124%	2%
	020700-RTS-MS-6	NA	NA	NA	NA	NA	NA		
	020700-RTS-MSD-6	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-1	NA	NA	1	281	A021800017	113%		
	020800-RTS-MSD-1	NA	NA	1	259	D081600027	105% A*	109%	8%
	020800-RTS-MS-1	NA	NA	NA	NA	NA	NA		
	020800-RTS-MSD-1	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-2	NA	NA	1	296	A021800018	119%		
	020800-RTS-MSD-2	NA	NA	1	247	D081600023	100% A	110%	18%
	020800-RTS-MS-2	NA	NA	NA	NA	NA	NA		
	020800-RTS-MSD-2	NA	NA	NA	NA	NA	NA	NA	NA
	C90778M-MS-5	NA	NA	4	338	A062100054	71%		
	C90778M-MSD-6	NA	NA	4	333	A062100055	70%	70%	1%
	020900-RTS-MS-5	NA	NA	1	334	A021800019	135%		
	020900-RTS-MSD-6	NA	NA	1	357	A021800020	144%	139%	7%
	020900-RTS-MS-5	NA	NA	1	307	D031500039	124% 2nd		
	020900-RTS-MSD-6	NA	NA	1	313	D031500040	126% 2nd	125%	2%
Group 1 Control 0.0 mg/kg	C90712M	Unknown	NA	1	2.52	A021800023	<LOQ (0.00493 ug/mL)		
	C90716M	Unknown	NA	1	0.00	A021800037	<LOQ (0.00493 ug/mL)		
	C90722M	Unknown	NA	1	0.00	A021800038	<LOQ (0.00493 ug/mL)		
	C90724M	Unknown	NA	1	0.00	A021800039	<LOQ (0.00493 ug/mL)		
	C90726M	Unknown	NA	1	0.00	A021800024	<LOQ (0.00493 ug/mL)		
	C90728M	Unknown	NA	1	0.00	A021800040	<LOQ (0.00493 ug/mL)		
	C90735M	Unknown	NA	1	0.00	A021800041	<LOQ (0.00493 ug/mL)		
	C90742M	Unknown	NA	1	0.00	A021800044	<LOQ (0.00493 ug/mL)		
	C90743M	Unknown	NA	1	0.00	A021800025	<LOQ (0.00493 ug/mL)		
	C90747M	Unknown	NA	1	0.00	A021800045	<LOQ (0.00493 ug/mL)		
	C90750M	Unknown	NA	1	0.00	A021800026	<LOQ (0.00493 ug/mL)		
	C90758M	Unknown	NA	1	0.00	A021800027	<LOQ (0.00493 ug/mL)		
	C90765M	Unknown	NA	1	0.00	A021800046	<LOQ (0.00493 ug/mL)		NA
	C90778M	Unknown	NA	1	0.00	A021800058	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	Unknown	NA	1	0.00	A021800047	<LOQ (0.00493 ug/mL)		
	C91131F	Unknown	NA	1	0.00	A021800048	<LOQ (0.00493 ug/mL)		
	C91138F	Unknown	NA	1	0.00	A021800059	<LOQ (0.00493 ug/mL)		
	C91144F	Unknown	NA	1	0.00	A021800030	<LOQ (0.00493 ug/mL)		
	C91148F	Unknown	NA	1	0.00	A021800051	<LOQ (0.00493 ug/mL)		
	C91157F	Unknown	NA	1	0.00	A021800031	<LOQ (0.00493 ug/mL)		
	C91159F	Unknown	NA	1	2.87	A021800052	<LOQ (0.00493 ug/mL)		
	C91166F	Unknown	NA	1	0.00	A021800032	<LOQ (0.00493 ug/mL)		
	C91172F	Unknown	NA	1	0.00	A021800053	<LOQ (0.00493 ug/mL)		
	C91176F	Unknown	NA	1	0.00	A021800054	<LOQ (0.00493 ug/mL)		
	C91183F	Unknown	NA	1	0.00	A021800055	<LOQ (0.00493 ug/mL)		
	C91188F	Unknown	NA	1	0.00	A021800033	<LOQ (0.00493 ug/mL)		NA
	C91190F	Unknown	NA	1	3.68	A021800034	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.00 mg/kg	C90781M	Unknown	NA	1	141	R041800017	0.141		
	C90782M	Unknown	NA	1	538	A021800060	0.538		
	C90783M	Unknown	NA	1	642	A021800061	0.642		
	C90785M	Unknown	NA	1	189	A021800064	0.189		
	C90787M	Unknown	NA	1	289	A021800065	0.289		
	C90788M	Unknown	NA	10	84.5	A031500029	0.845		
	C90791M	Unknown	NA	10	80.7	A021800101	0.807		
	C90794M	Unknown	NA	1	670	A021800066	0.670		
	C90799M	Unknown	NA	1	212	R041800021	0.212		
	C90804M	Unknown	NA	1	214	A021800067	0.214		
	C90809M	Unknown	NA	10	93.6	A031500034	0.936		
	C90810M	Unknown	NA	1	764	A021800068	0.764		
	C90811M	Unknown	NA	1	105	R041800024	0.105		
	C90812M	Unknown	NA	1	617	R041900025	0.617		
	C90815M	Unknown	NA	1	278	R041900026	0.278		
	C90823M	Unknown	NA	1	174	R041800027	0.174		
	C90829M	Unknown	NA	1	475	R041900028	0.475		
	C90830M	Unknown	NA	1	579	R041900031	0.579		
	C90832M	Unknown	NA	10	156	A031500040	1.56		
	C90835M	Unknown	NA	1	684	A021800103	0.684		
	C90838M	Unknown	NA	1	769	R041900033	0.769		62.4
	C90840M	Unknown	NA	1	685	R041900034	0.685	0.553	0.345
Group 2 Low Dose 3.00 mg/kg	C91197F	Unknown	NA	1	327	A021800092	0.327		
	C91201F	Unknown	NA	1	368	A021800093	0.368		
	C91207F	Unknown	NA	10	63.1	A031500044	0.631		
	C91216F	Unknown	E	E	E	E	E		
	C91220F	Unknown	NA	10	130	A031500047	1.30		
	C91221F	Unknown	NA	1	380	A021800094	0.380		
	C91227F	Unknown	NA	1	609	A021800095	0.609		
	C91232F	Unknown	NA	10	360	A031500050	3.60		
	C91235F	Unknown	NA	10	439	A031500051	4.39		
	C91236F	Unknown	NA	10	272	A031500054	2.72		
	C91242F	Unknown	NA	10	137	A031500055	1.37		94.1
	C91243F	Unknown	NA	1	896	R041900046	0.896	1.51	1.42

A= CCVs analyzed bracketing these data were within criteria but had peak areas slightly above the valid linear range. LAC 05/03/01
E = Lost during extraction, no sample remaining to reextract.

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
MS56 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL LAC 02/19/01

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/17/00, 5/16/00, 06/22/00, 06/23/00, 06/26/00, 09/05/00, 10/17/00, 04/13/01 LAC/CSH 4/17/01 mmh
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Maxslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 SAL/RWW
Dates of Analysis/Analyst:	02/18/00, 03/15/00, 4/18/00, 4/19/00, 06/19/00, 06/20/00, 08/16/00, 08/23/00 MMH/IAS/CSH
Date of Data Reduction/Analyst:	02/21/00, 03/16/00, 4/19/00, 4/20/00, 06/20/00, 06/21/00, 08/18/00, 08/25/00, 04/11/01 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot Unknown/SD013

Group Dose	Sample #	EtFOSE-OH Purity Correction Factor	Surrogate Verified	EtFOSE-OH Dilution Factor	EtFOSE-OH Conc. ug/mL	Filename	Concentration of EtFOSE-OH ug/mL or % Rec	Average EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	020700-H2O Blk-5	Unknown	NA	1	0.00	D081600016	<LOQ (0.00977 ug/mL)		
	020700-H2O Blk-6	Unknown	NA	1	0.00	D081600017	<LOQ (0.00977 ug/mL)	<LOQ	NA
	020800-H2O Blk-1	Unknown	2nd Analysis OK	1	0.00	D081600020	<LOQ (0.00977 ug/mL)		
	020800-H2O Blk-2	Unknown	NA	1	0.00	A021800110	<LOQ (0.00977 ug/mL)	<LOQ	NA
	020900-H2O Blk-5	Unknown	NA	1	0.00	A062000005	<LOQ (0.0248 ug/mL)		
	020900-H2O Blk-6	Unknown	NA	1	0.00	A062000019	<LOQ (0.0248 ug/mL)	<LOQ	NA
Matrix Blk	020700-RBS Blk-5	Unknown	NA	1	0.00	D081600018	<LOQ (0.00977 ug/mL)		
	020700-RBS Blk-6	Unknown	NA	1	0.00	D081600019	<LOQ (0.00977 ug/mL)	<LOQ	NA
	020800-RBS Blk-1	Unknown	X	1	0.00	A021800004	<LOQ (0.00977 ug/mL)		
	020800-RBS-Blk-2	Unknown	NA	1	0.00	A021800111	<LOQ (0.00977 ug/mL)	<LOQ	NA
	020800-RTS Blk-1	Unknown	X	1	0.00	A021800005	<LOQ (0.00977 ug/mL)		
	020800-RTS-Blk-2	Unknown	NA	1	0.00	A021800112	<LOQ (0.00977 ug/mL)	<LOQ	NA
	020900-RBS Blk-5	Unknown	NA	1	0.00	A062000006	<LOQ (0.0248 ug/mL)		
	020900-RBS-Blk-6	Unknown	NA	1	0.00	A062000020	<LOQ (0.0248 ug/mL)	<LOQ	NA
	020900-RTS Blk-5	Unknown	NA	1	0.00	A062000007	<LOQ (0.0248 ug/mL)		
	020900-RTS-Blk-6	Unknown	Confirmed High	1	0.00	A062000021	<LOQ (0.0248 ug/mL)	<LOQ	NA
QC-250 ppb	020700-RTS-MS-5	NA	NA	1	83.8	A022500017	34%		
	020700-RTS-MSD-5	NA	NA	1	69.2	A022500018	28%	31%	19%
	020700-RTS-MS-5	NA	NA	NA	NA	NA	NA		
	020700-RTS-MSD-5	NA	NA	NA	NA	NA	NA	NA	NA
	020700-RTS-MS-6	NA	NA	1	82.2	A022500019	33%		
	020700-RTS-MSD-6	NA	NA	1	65.0	A022500020	26%	30%	23%
	020700-RTS-MS-6	NA	NA	NA	NA	NA	NA		
	020700-RTS-MSD-6	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-1	NA	NA	1	51.8	A021800017	21%		
	020800-RTS-MSD-1	NA	NA	1	63.2	D081600027	25%	23%	20%
	020800-RTS-MS-1	NA	NA	1	80.6	D031500037	32% 2nd		
	020800-RTS-MSD-1	NA	NA	NA	NA	NA	NA	32%	NA
	020800-RTS-MS-2	NA	NA	1	53.2	A021800018	21%		
	020800-RTS-MSD-2	NA	NA	1	62.8	D081600023	25%	23%	17%
	020800-RTS-MS-2	NA	NA	1	93.2	D031500038	38% 2nd		
	020800-RTS-MSD-2	NA	NA	NA	NA	NA	NA	38%	NA
	C90778M-MS-5	NA	NA	4	582	A062100054	122%		
	C90778M-MSD-6	NA	NA	4	606	A062100055	127%	125%	4%
	020900-RTS-MS-5	NA	NA	1	64.5	A021800019	26%		
	020900-RTS-MSD-6	NA	NA	1	71.1	A021800020	29%	27%	10%
	020900-RTS-MS-5	NA	NA	1	92.9	D031500039	37% 2nd		
	020900-RTS-MSD-6	NA	NA	1	97.0	D031500040	39% 2nd	38%	4%
Group 1	C90712M	Unknown	NA	1	0.00	A021800023	<LOQ (0.00977 ug/mL)		
Control	C90716M	Unknown	NA	1	0.00	A021800037	<LOQ (0.00977 ug/mL)		
0.0 mg/kg	C90722M	Unknown	NA	1	0.00	A021800038	<LOQ (0.00977 ug/mL)		
	C90724M	Unknown	NA	1	0.00	A021800039	<LOQ (0.00977 ug/mL)		
	C90726M	Unknown	NA	1	0.00	A021800024	<LOQ (0.00977 ug/mL)		
	C90728M	Unknown	NA	1	0.00	A021800040	<LOQ (0.00977 ug/mL)		
	C90735M	Unknown	NA	1	0.00	A021800041	<LOQ (0.00977 ug/mL)		
	C90742M	Unknown	NA	1	0.00	A021800044	<LOQ (0.00977 ug/mL)		
	C90743M	Unknown	NA	1	0.00	A021800025	<LOQ (0.00977 ug/mL)		
	C90747M	Unknown	NA	1	0.00	A021800045	<LOQ (0.00977 ug/mL)		
	C90750M	Unknown	NA	1	0.00	A021800026	<LOQ (0.00977 ug/mL)		
	C90758M	Unknown	NA	1	0.00	A021800027	<LOQ (0.00977 ug/mL)		
	C90765M	Unknown	NA	1	0.00	A021800046	<LOQ (0.00977 ug/mL)		NA
	C90778M	Unknown	NA	1	0.00	A021800058	<LOQ (0.00977 ug/mL)	<LOQ	NA
Group 1	C91127F	Unknown	NA	1	0.00	A021800047	<LOQ (0.00977 ug/mL)		
Control	C91131F	Unknown	NA	1	0.00	A021800048	<LOQ (0.00977 ug/mL)		
0.0 mg/kg	C91138F	Unknown	NA	1	0.00	A021800059	<LOQ (0.00977 ug/mL)		
	C91144F	Unknown	NA	1	2.54	A021800030	<LOQ (0.00977 ug/mL)		
	C91148F	Unknown	NA	1	0.00	A021800051	<LOQ (0.00977 ug/mL)		
	C91157F	Unknown	NA	1	0.00	A021800031	<LOQ (0.00977 ug/mL)		
	C91159F	Unknown	NA	1	0.00	A021800052	<LOQ (0.00977 ug/mL)		
	C91166F	Unknown	NA	1	0.00	A021800032	<LOQ (0.00977 ug/mL)		
	C91172F	Unknown	NA	1	0.00	A021800053	<LOQ (0.00977 ug/mL)		
	C91176F	Unknown	NA	1	0.00	A021800054	<LOQ (0.00977 ug/mL)		
	C91183F	Unknown	NA	1	0.00	A021800055	<LOQ (0.00977 ug/mL)		
	C91188F	Unknown	NA	1	0.00	A021800033	<LOQ (0.00977 ug/mL)		NA
	C91190F	Unknown	NA	1	0.00	A021800034	<LOQ (0.00977 ug/mL)	<LOQ	NA
Group 2	C90781M	Unknown	NA	1	0.00	A021800081	<LOQ (0.00977 ug/mL)		
Low Dose	C90782M	Unknown	NA	1	0.00	A021800060	<LOQ (0.00977 ug/mL)		
3.00 mg/kg	C90783M	Unknown	NA	1	2.81	A021800061	<LOQ (0.00977 ug/mL)		
	C90785M	Unknown	NA	1	0.00	A021800064	<LOQ (0.00977 ug/mL)		
	C90787M	Unknown	NA	1	0.00	A021800065	<LOQ (0.00977 ug/mL)		
	C90788M	Unknown	NA	1	2.19	A021800100	<LOQ (0.00977 ug/mL)		
	C90791M	Unknown	NA	1	0.00	A021800101	<LOQ (0.00977 ug/mL)		
	C90794M	Unknown	NA	1	0.00	A021800066	<LOQ (0.00977 ug/mL)		
	C90799M	Unknown	NA	1	0.00	A021800082	<LOQ (0.00977 ug/mL)		
	C90804M	Unknown	NA	1	0.00	A021800067	<LOQ (0.00977 ug/mL)		
	C90809M	Unknown	NA	1	2.45	A021800102	<LOQ (0.00977 ug/mL)		
	C90810M	Unknown	NA	1	1.89	A021800068	<LOQ (0.00977 ug/mL)		
	C90811M	Unknown	NA	1	0.00	A021800085	<LOQ (0.00977 ug/mL)		
	C90812M	Unknown	NA	1	0.00	A021800086	<LOQ (0.00977 ug/mL)		
	C90815M	Unknown	NA	1	0.00	A021800087	<LOQ (0.00977 ug/mL)		
	C90823M	Unknown	NA	1	0.00	A021800088	<LOQ (0.00977 ug/mL)		
	C90829M	Unknown	NA	1	0.00	A021800071	<LOQ (0.00977 ug/mL)		
	C90830M	Unknown	NA	1	2.76	A021800072	<LOQ (0.00977 ug/mL)		
	C90832M	Unknown	NA	1	0.00	A021800073	<LOQ (0.00977 ug/mL)		
	C90835M	Unknown	NA	1	0.00	A021800103	<LOQ (0.00977 ug/mL)		
	C90838M	Unknown	NA	1	2.50	A021800074	<LOQ (0.00977 ug/mL)		NA
	C90840M	Unknown	NA	1	0.00	A021800089	<LOQ (0.00977 ug/mL)	<LOQ	NA
Group 2	C91197F	Unknown	NA	1	0.00	A021800092	<LOQ (0.00977 ug/mL)		
Low Dose	C91201F	Unknown	NA	1	0.00	A021800093	<LOQ (0.00977 ug/mL)		
3.00 mg/kg	C91207F	Unknown	NA	1	0.00	A021800075	<LOQ (0.00977 ug/mL)		
	C91216F	Unknown	E	E	E	E	E		
	C91220F	Unknown	NA	1	2.40	A021800106	<LOQ (0.00977 ug/mL)		
	C91221F	Unknown	NA	1	0.00	A021800094	<LOQ (0.00977 ug/mL)		
	C91227F	Unknown	NA	1	0.00	A021800095	<LOQ (0.00977 ug/mL)		
	C91232F	Unknown	NA	1	7.15	A021800078	<LOQ (0.00977 ug/mL)		
	C91235F	Unknown	NA	1	7.45	A021800079	<LOQ (0.00977 ug/mL)		
	C91236F	Unknown	NA	1	4.03	A021800096	<LOQ (0.00977 ug/mL)		
	C91242F	Unknown	NA	1	3.07	A021800099	<LOQ (0.00977 ug/mL)		NA
	C91243F	Unknown	NA	1	0.00	A021800080	<LOQ (0.00977 ug/mL)	<LOQ	NA

A= CCVs analyzed bracketing these data were within criteria but had peak areas slightly above the valid linear range. LAC 05/03/01
E = Lost during extraction, no sample remaining to reextract.
PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/17/00, 5/16/00, 06/22/00, 06/23/00, 06/26/00, 09/05/00, 10/17/00, 04/13/01 LAC/CSH 4/17/01 mmh
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98 1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 SAL/RWW
Dates of Analysis/Analyst:	02/18/00, 03/15/00, 4/18/00, 4/19/00, 06/19/00, 06/20/00, 08/16/00, 08/23/00 MMH/IAS/CSH
Date of Data Reduction/Analyst:	02/21/00, 03/16/00, 4/19/00, 4/20/00, 06/20/00, 06/21/00, 08/18/00, 08/25/00, 04/11/01 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot NB113047-80

Group Dose	Sample #	M556 Purity Correction Factor	Surrogate Verified	M556 Dilution Factor	M556 Conc. ng/mL	Filename	Concentration of M556 ug/mL or % Rec	Average M556 ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	020700-H2O Blk-5	Unknown	NA	1	0.190	D081600016	<LOQ (0.00977 ug/mL) A		
	020700-H2O Blk-6	Unknown	NA	1	0.00	D081600017	<LOQ (0.00977 ug/mL) A	<LOQ	NA
	020800-H2O Blk-1	Unknown	2nd Analysis OK	1	0.00	D081600020	<LOQ (0.00977 ug/mL) A		
	020800-H2O Blk-2	Unknown	NA	1	0.00	D031500025	<LOQ (0.00494 ug/mL)	<LOQ	NA
	020900-H2O Blk-5	Unknown	NA	1	0.00	A061900005	<LOQ (0.00494 ug/mL)		
	020900-H2O Blk-6	Unknown	NA	1	0.00	A061900019	<LOQ (0.00494 ug/mL)	<LOQ	NA
Matrix Blk	020700-RBS Blk-5	Unknown	NA	1	0.00	D081600018	<LOQ (0.00977 ug/mL) A		
	020700-RBS Blk-6	Unknown	NA	1	0.00	D081600019	<LOQ (0.00977 ug/mL) A	<LOQ	NA
	020800-RBS Blk-1	Unknown	X	1	0.00	A031500006	<LOQ (0.00494 ug/mL)		
	020800-RBS-Blk-2	Unknown	NA	1	0.00	D031500026	<LOQ (0.00494 ug/mL)	<LOQ	NA
	020800-RTS Blk-1	Unknown	X	1	0.69	A031500007	<LOQ 0.00497 ug/mL)		
	020800-RTS-Blk-2	Unknown	NA	1	0.00	D031500027	<LOQ 0.00497 ug/mL)	<LOQ	NA
	020900-RBS Blk-5	Unknown	NA	1	0.00	A031500007	<LOQ (0.00494 ug/mL)		
	020900-RBS-Blk-6	Unknown	NA	1	0.00	D031500027	<LOQ (0.00494 ug/mL)	<LOQ	NA
	020900-RTS Blk-5	Unknown	NA	1	0.0900	A061900007	<LOQ (0.00494 ug/mL)		
	020900-RTS-Blk-6	Unknown	Confirmed High	1	0.00	A061900021	<LOQ (0.00494 ug/mL)	<LOQ	NA
QC-250 ppb	020700-RTS-MS-5	NA	NA	1	380	A022500016	153%		
	020700-RTS-MSD-5	NA	NA	1	381	A022500017	153%	153%	0%
	020700-RTS-MS-5	NA	NA	1	345	D082300057	139% 2nd		
	020700-RTS-MSD-5	NA	NA	1	359	D082300058	145% 2nd	142%	4%
	020700-RTS-MS-6	NA	NA	1	363	A022500018	146%		
	020700-RTS-MSD-6	NA	NA	1	352	A022500019	142%	144%	3%
	020700-RTS-MS-6	NA	NA	1	354	D082300059	143% 2nd		
	020700-RTS-MSD-6	NA	NA	1	350	D082300060	141% 2nd	142%	1%
	020800-RTS-MS-1	NA	NA	1	249	A021800017	100%		
	020800-RTS-MSD-1	NA	NA	1	252	D081600027	102% A	101%	1%
	020800-RTS-MS-1	NA	NA	NA	NA	NA	NA		
	020800-RTS-MSD-1	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-2	NA	NA	1	247	A021800018	100%		
	020800-RTS-MSD-2	NA	NA	1	230	D081600023	93% A	96%	7%
	020800-RTS-MS-2	NA	NA	NA	NA	NA	NA		
	020800-RTS-MSD-2	NA	NA	NA	NA	NA	NA	NA	NA
	C90778M-MS-5	NA	NA	4	445	A062000054	93%		
	C90778M-MSD-6	NA	NA	4	409	A062000055	86%	90%	8%
	020900-RTS-MS-5	NA	NA	1	340	A021800019	137%		
	020900-RTS-MSD-6	NA	NA	1	347	A021800020	139%	138%	1%
	020900-RTS-MS-5	NA	NA	1	324	D031500039	131% 2nd		
	020900-RTS-MSD-6	NA	NA	1	318	D031500040	127% 2nd	129%	3%
Group 1 Control 0.0 mg/kg	C90712M	Unknown	NA	1	0.600	D031500043	<LOQ (0.00494 ug/mL)		
	C90716M	Unknown	NA	1	0.380	D031500057	<LOQ (0.00494 ug/mL)		
	C90722M	Unknown	NA	1	0.00	D031500058	<LOQ (0.00494 ug/mL)		
	C90724M	Unknown	NA	1	0.00	D031500059	<LOQ (0.00494 ug/mL)		
	C90726M	Unknown	NA	1	0.00	D031500044	<LOQ (0.00494 ug/mL)		
	C90728M	Unknown	NA	1	0.00	D031500060	<LOQ (0.00494 ug/mL)		
	C90735M	Unknown	NA	1	0.00	D031500061	<LOQ (0.00494 ug/mL)		
	C90742M	Unknown	NA	1	0.00	D031500064	<LOQ (0.00494 ug/mL)		
	C90743M	Unknown	NA	1	0.00	D031500045	<LOQ (0.00494 ug/mL)		
	C90747M	Unknown	NA	1	0.00	D031500065	<LOQ (0.00494 ug/mL)		
	C90750M	Unknown	NA	1	0.00	D031500046	<LOQ (0.00494 ug/mL)		
	C90758M	Unknown	NA	1	0.00	D031500047	<LOQ (0.00494 ug/mL)		
	C90765M	Unknown	NA	1	0.00	D031500066	<LOQ (0.00494 ug/mL)		NA
	C90778M	Unknown	NA	1	0.00	D031500078	<LOQ (0.00494 ug/mL)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	Unknown	NA	1	0.00	D031500067	<LOQ (0.00494 ug/mL)		
	C91131F	Unknown	NA	1	0.00	D031500068	<LOQ (0.00494 ug/mL)		
	C91138F	Unknown	NA	1	0.00	D031500079	<LOQ (0.00494 ug/mL)		
	C91144F	Unknown	NA	1	0.00	D031500050	<LOQ (0.00494 ug/mL)		
	C91148F	Unknown	NA	1	0.00	D031500071	<LOQ (0.00494 ug/mL)		
	C91157F	Unknown	NA	1	0.00	D031500051	<LOQ (0.00494 ug/mL)		
	C91159F	Unknown	NA	1	0.00	D031500072	<LOQ (0.00494 ug/mL)		
	C91166F	Unknown	NA	1	0.00	D031500052	<LOQ (0.00494 ug/mL)		
	C91172F	Unknown	NA	1	0.00	D031500073	<LOQ (0.00494 ug/mL)		
	C91176F	Unknown	NA	1	0.00	D031500074	<LOQ (0.00494 ug/mL)		
	C91183F	Unknown	NA	1	0.00	D031500075	<LOQ (0.00494 ug/mL)		
	C91188F	Unknown	NA	1	0.00	D031500053	<LOQ (0.00494 ug/mL)		NA
	C91190F	Unknown	NA	1	0.00	D031500054	<LOQ (0.00494 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.00 mg/kg	C90781M	Unknown	NA	1	44.5	A021800081	0.0445		
	C90782M	Unknown	NA	1	123	A021800060	0.123		
	C90783M	Unknown	NA	1	264	A021800061	0.264		
	C90785M	Unknown	NA	1	36.2	A021800064	0.0362		
	C90787M	Unknown	NA	1	76.8	A021800065	0.0768		
	C90788M	Unknown	NA	1	534	A021800100	0.534		
	C90791M	Unknown	NA	1	195	A021800101	0.195		
	C90794M	Unknown	NA	1	217	A021800066	0.217		
	C90799M	Unknown	NA	1	161	A021800082	0.161		
	C90804M	Unknown	NA	1	21.5	A021800067	0.0215		
	C90809M	Unknown	NA	1	255	A021800102	0.255		
	C90810M	Unknown	NA	1	55.8	A021800068	0.0558		
	C90811M	Unknown	NA	1	91.6	A021800085	0.0916		
	C90812M	Unknown	NA	1	61.0	A021800086	0.0610		
	C90815M	Unknown	NA	1	112	A021800087	0.112		
	C90823M	Unknown	NA	1	175	A021800088	0.175		
	C90829M	Unknown	NA	1	145	A021800071	0.145		
	C90830M	Unknown	NA	1	276	A021800072	0.276		
	C90832M	Unknown	NA	1	122	A021800073	0.122		
	C90835M	Unknown	NA	1	49.9	A021800103	0.0499		
	C90838M	Unknown	NA	1	95.0	A021800074	0.0950		79.4
	C90840M	Unknown	NA	1	88.8	A021800089	0.0888	0.145	0.115
Group 2 Low Dose 3.00 mg/kg	C91197F	Unknown	NA	1	285	A021800092	0.285		
	C91201F	Unknown	NA	1	200	A021800093	0.200		
	C91207F	Unknown	NA	1	300	A021800075	0.300		
	C91216F	Unknown	E	E	E	E	E		
	C91220F	Unknown	NA	1	355	A021800106	0.355		
	C91221F	Unknown	NA	1	118	A021800094	0.118		
	C91227F	Unknown	NA	1	164	A021800095	0.164		
	C91232F	Unknown	NA	10	159	A031500050	1.59		
	C91235F	Unknown	NA	10	190	A031500051	1.90		
	C91236F	Unknown	NA	10	143	A031500054	1.43		
	C91242F	Unknown	NA	10	98.0	A031500055	0.980		98.0
	C91243F	Unknown	NA	1	166	A021800080	0.166	0.680	0.667

A= CCVs analyzed bracketing these data were within criteria but had peak areas slightly above the valid linear range. LAC 05/03/01
E = Lost during extraction, no sample remaining to reextract.
PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/17/00, 5/16/00, 06/22/00, 06/23/00, 06/26/00, 09/05/00, 10/17/00, 04/13/01 LAC/CSH 4/17/01 mmh
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 SAL/RWW
Dates of Analysis/Analyst:	02/18/00, 03/15/00, 4/18/00, 4/19/00, 06/19/00, 06/20/00, 08/16/00, 08/23/00 MMH/IAS/CSH
Date of Data Reduction/Analyst:	02/21/00, 03/16/00, 4/19/00, 4/20/00, 06/20/00, 06/21/00, 08/18/00, 08/25/00, 04/11/01 MMH/IAS/HOJ
Sample Data	Box 00-009

WEEK 105 RAT SERA lot 529

Group Dose	Sample #	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Dilution Factor	PFOSEA Conc. ng/mL	Filename	Concentration of PFOSEA ug/mL or % Rec	Average PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	020700-H2O Blk-5	Unknown	NA	1	0.00	D081600016	<LOQ (0.00492 ug/mL)		
	020700-H2O Blk-6	Unknown	NA	1	0.120	D081600017	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020800-H2O Blk-1	Unknown	2nd Analysis OK	1	0.00	D081600020	<LOQ (0.00492 ug/mL)		
	020800-H2O Blk-2	Unknown	NA	1	0.00	A021800110	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020900-H2O Blk-5	Unknown	NA	1	0.00	A061900005	<LOQ (0.00492 ug/mL)		
	020900-H2O Blk-6	Unknown	NA	1	0.00	A061900019	<LOQ (0.00492 ug/mL)	<LOQ	NA
Matrix Blk	020700-RBS Blk-5	Unknown	NA	1	0.00	D081600018	<LOQ (0.00492 ug/mL)		
	020700-RBS Blk-6	Unknown	NA	1	0.00	D081600019	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020800-RBS Blk-1	Unknown	X	1	0.00	A021800004	<LOQ (0.00492 ug/mL)		
	020800-RBS-Blk-2	Unknown	NA	1	0.00	A021800111	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020800-RTS Blk-1	Unknown	X	1	0.00	A021800005	<LOQ (0.00492 ug/mL)		
	020800-RTS-Blk-2	Unknown	NA	1	0.00	A021800112	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020900-RBS Blk-5	Unknown	NA	1	0.00	A061900006	<LOQ (0.00492 ug/mL)		
	020900-RBS-Blk-6	Unknown	NA	1	0.00	A061900020	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020900-RTS Blk-5	Unknown	NA	1	0.00	A061900007	<LOQ (0.00492 ug/mL)		
	020900-RTS-Blk-6	Unknown	Confirmed High	1	0.00	A061900021	<LOQ (0.00492 ug/mL)	<LOQ	NA
QC-250 ppb	020700-RTS-MS-5	NA	NA	1	196	A022600017	79%		
	020700-RTS-MSD-5	NA	NA	1	186	A022600018	75%	77%	5%
	020700-RTS-MS-5	NA	NA	NA	NA	NA	NA		
	020700-RTS-MSD-5	NA	NA	NA	NA	NA	NA	NA	NA
	020700-RTS-MS-6	NA	NA	1	196	A022600019	79%		
	020700-RTS-MSD-6	NA	NA	1	150	A022600020	60%	70%	27%
	020700-RTS-MS-6	NA	NA	NA	NA	NA	NA		
	020700-RTS-MSD-6	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-1	NA	NA	1	114	A021800017	46%		
	020800-RTS-MSD-1	NA	NA	1	161	D081600027	65%	55%	34%
	020800-RTS-MS-1	NA	NA	1	105	D031500037	42% 2nd		
	020800-RTS-MSD-1	NA	NA	NA	NA	NA	NA	42%	NA
	020800-RTS-MS-2	NA	NA	1	115	A021800018	46%		
	020800-RTS-MSD-2	NA	NA	1	138	D081600023	56%	51%	18%
	020800-RTS-MS-2	NA	NA	1	112	D031500038	45% 2nd		
	020800-RTS-MSD-2	NA	NA	NA	NA	NA	NA	45%	NA
	C90778M-MS-5	NA	NA	4	398	A062000054	83%		
	C90778M-MSD-6	NA	NA	4	416	A062000055	87%	85%	4%
	020900-RTS-MS-5	NA	NA	1	150	A021800019	61%		
	020900-RTS-MSD-6	NA	NA	1	150	A021800020	60%	60%	1%
	020900-RTS-MS-5	NA	NA	1	142	D031500039	57% 2nd		
	020900-RTS-MSD-6	NA	NA	1	141	D031500040	56% 2nd	57%	2%
Group 1 Control 0.0 mg/kg	C90712M	Unknown	NA	1	0.00	A021800023	<LOQ (0.00492 ug/mL)		
	C90716M	Unknown	NA	1	0.00	A021800037	<LOQ (0.00492 ug/mL)		
	C90722M	Unknown	NA	1	0.00	A021800038	<LOQ (0.00492 ug/mL)		
	C90724M	Unknown	NA	1	0.00	A021800039	<LOQ (0.00492 ug/mL)		
	C90726M	Unknown	NA	1	0.00	A021800024	<LOQ (0.00492 ug/mL)		
	C90728M	Unknown	NA	1	0.00	A021800040	<LOQ (0.00492 ug/mL)		
	C90735M	Unknown	NA	1	0.00	A021800041	<LOQ (0.00492 ug/mL)		
	C90742M	Unknown	NA	1	0.00	A021800044	<LOQ (0.00492 ug/mL)		
	C90743M	Unknown	NA	1	0.00	A021800025	<LOQ (0.00492 ug/mL)		
	C90747M	Unknown	NA	1	0.00	A021800045	<LOQ (0.00492 ug/mL)		
	C90750M	Unknown	NA	1	0.00	A021800026	<LOQ (0.00492 ug/mL)		
	C90758M	Unknown	NA	1	0.00	A021800027	<LOQ (0.00492 ug/mL)		
	C90765M	Unknown	NA	1	0.00	A021800046	<LOQ (0.00492 ug/mL)		NA
	C90778M	Unknown	NA	1	0.00	A021800058	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	Unknown	NA	1	0.00	A021800047	<LOQ (0.00492 ug/mL)		
	C91131F	Unknown	NA	1	0.00	A021800048	<LOQ (0.00492 ug/mL)		
	C91138F	Unknown	NA	1	0.00	A021800059	<LOQ (0.00492 ug/mL)		
	C91144F	Unknown	NA	1	0.00	A021800030	<LOQ (0.00492 ug/mL)		
	C91148F	Unknown	NA	1	0.00	A021800051	<LOQ (0.00492 ug/mL)		
	C91157F	Unknown	NA	1	0.00	A021800031	<LOQ (0.00492 ug/mL)		
	C91159F	Unknown	NA	1	0.00	A021800052	<LOQ (0.00492 ug/mL)		
	C91166F	Unknown	NA	1	0.00	A021800032	<LOQ (0.00492 ug/mL)		
	C91172F	Unknown	NA	1	0.00	A021800053	<LOQ (0.00492 ug/mL)		
	C91176F	Unknown	NA	1	0.00	A021800054	<LOQ (0.00492 ug/mL)		
	C91183F	Unknown	NA	1	0.00	A021800055	<LOQ (0.00492 ug/mL)		
	C91188F	Unknown	NA	1	3.03	A021800033	<LOQ (0.00492 ug/mL)		NA
	C91190F	Unknown	NA	1	0.00	A021800034	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.00 mg/kg	C90781M	Unknown	NA	1	0.00	A021800081	<LOQ (0.00492 ug/mL)		
	C90782M	Unknown	NA	1	0.00	A021800060	<LOQ (0.00492 ug/mL)		
	C90783M	Unknown	NA	1	0.00	A021800061	<LOQ (0.00492 ug/mL)		
	C90785M	Unknown	NA	1	0.00	A021800064	<LOQ (0.00492 ug/mL)		
	C90787M	Unknown	NA	1	0.00	A021800065	<LOQ (0.00492 ug/mL)		
	C90788M	Unknown	NA	1	0.00	A021800100	<LOQ (0.00492 ug/mL)		
	C90791M	Unknown	NA	1	0.00	A021800101	<LOQ (0.00492 ug/mL)		
	C90794M	Unknown	NA	1	0.00	A021800066	<LOQ (0.00492 ug/mL)		
	C90799M	Unknown	NA	1	0.00	A021800082	<LOQ (0.00492 ug/mL)		
	C90804M	Unknown	NA	1	0.00	A021800067	<LOQ (0.00492 ug/mL)		
	C90809M	Unknown	NA	1	0.00	A021800102	<LOQ (0.00492 ug/mL)		
	C90810M	Unknown	NA	1	0.00	A021800068	<LOQ (0.00492 ug/mL)		
	C90811M	Unknown	NA	1	0.00	A021800085	<LOQ (0.00492 ug/mL)		
	C90812M	Unknown	NA	1	0.00	A021800086	<LOQ (0.00492 ug/mL)		
	C90815M	Unknown	NA	1	0.00	A021800087	<LOQ (0.00492 ug/mL)		
	C90823M	Unknown	NA	1	0.00	A021800088	<LOQ (0.00492 ug/mL)		
	C90829M	Unknown	NA	1	0.00	A021800071	<LOQ (0.00492 ug/mL)		
	C90830M	Unknown	NA	1	0.00	A021800072	<LOQ (0.00492 ug/mL)		
	C90832M	Unknown	NA	1	0.00	A021800073	<LOQ (0.00492 ug/mL)		
	C90835M	Unknown	NA	1	0.00	A021800103	<LOQ (0.00492 ug/mL)		
	C90838M	Unknown	NA	1	0.00	A021800074	<LOQ (0.00492 ug/mL)		NA
	C90840M	Unknown	NA	1	0.00	A021800089	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.00 mg/kg	C91197F	Unknown	NA	1	0.00	A021800092	<LOQ (0.00492 ug/mL)		
	C91201F	Unknown	NA	1	0.00	A021800093	<LOQ (0.00492 ug/mL)		
	C91207F	Unknown	NA	1	0.00	A021800075	<LOQ (0.00492 ug/mL)		
	C91216F	Unknown	E	E	E	E	E		
	C91220F	Unknown	NA	1	0.00	A021800106	<LOQ (0.00492 ug/mL)		
	C91221F	Unknown	NA	1	0.00	A021800094	<LOQ (0.00492 ug/mL)		
	C91227F	Unknown	NA	1	0.00	A021800095	<LOQ (0.00492 ug/mL)		
	C91232F	Unknown	NA	1	0.00	A021800078	<LOQ (0.00492 ug/mL)		
	C91235F	Unknown	NA	1	0.00	A021800079	<LOQ (0.00492 ug/mL)		
	C91236F	Unknown	NA	1	0.00	A021800096	<LOQ (0.00492 ug/mL)		
	C91242F	Unknown	NA	1	0.00	A021800099	<LOQ (0.00492 ug/mL)		NA
	C91243F	Unknown	NA	1	0.00	A021800080	<LOQ (0.00492 ug/mL)	<LOQ	NA

A= CCVs analyzed bracketing these data were within criteria but had peak areas slightly above the valid linear range. LAC 05/03/01
E = Lost during extraction, no sample remaining to reextract.

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M56 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/17/00, 5/16/00, 06/22/00, 06/23/00, 06/26/00, 09/05/00, 10/17/00, 04/13/01 LAC/CSH 4/17/01 mmh
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 SAL/RWW
Dates of Analysis/Analyst:	02/18/00, 03/15/00, 4/18/00, 4/19/00, 06/19/00, 06/20/00, 08/16/00, 08/23/00 MMH/IAS/CSH
Date of Data Reduction/Analyst:	02/21/00, 03/16/00, 4/19/00, 4/20/00, 06/20/00, 06/21/00, 08/18/00, 08/25/00, 04/11/01 MMH/IAS/HOJ

Sample Data
WEEK 105 RAT SERA

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Average PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Average PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Average PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	020700-H2O Blk-5	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL) A		
	020700-H2O Blk-6	<LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL) A	<LOQ	NA
	020800-H2O Blk-1	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	020800-H2O Blk-2	<LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020900-H2O Blk-5	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	020900-H2O Blk-6	<LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA
Matrix Blk	020700-RBS Blk-5	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL) A		
	020700-RBS Blk-6	<LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL) A	<LOQ	NA
	020800-RBS Blk-1	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	020800-RBS-Blk-2	<LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020800-RTS Blk-1	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	020800-RTS-Blk-2	<LOQ (0.00394 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA
	020900-RBS Blk-5	0.00			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	020900-RBS-Blk-6	0.00	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA
	020900-RTS Blk-5	0.00			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	020900-RTS-Blk-6	0.00	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA
QC-250 ppb	020700-RTS-MS-5	121%			141%			125%		
	020700-RTS-MSD-5	121%	121%	0%	137%	139%	3%	123%	124%	2%
	020700-RTS-MS-5	NA			123% 2nd			NA		
	020700-RTS-MSD-5	NA	NA	NA	125% 2nd	124%	2%	NA	NA	NA
	020700-RTS-MS-6	120%			143%			126%		
	020700-RTS-MSD-6	101%	110%	17%	140%	141%	2%	123%	124%	2%
	020700-RTS-MS-6	NA			128% 2nd			NA		
	020700-RTS-MSD-6	NA	NA	NA	124% 2nd	126%	3%	NA	NA	NA
	020800-RTS-MS-1	80%			93%			113%		
	020800-RTS-MSD-1	83%	82%	4%	110%	101%	17%	105% A	109%	8%
	020800-RTS-MS-1	NA			NA			NA		
	020800-RTS-MSD-1	NA	NA	NA	NA	NA	NA	NA	NA	NA
	020800-RTS-MS-2	81%			93%			119%		
	020800-RTS-MSD-2	77%	79%	5%	105%	99%	12%	100% A	110%	18%
	020800-RTS-MS-2	NA			NA			NA		
	020800-RTS-MSD-2	NA	NA	NA	NA	NA	NA	NA	NA	NA
	C90778M-MS-5	72%			72%			71%		
	C90778M-MSD-6	73%	72%	1%	72%	72%	0%	70%	70%	1%
	020900-RTS-MS-5	97%			107%			135%		
	020900-RTS-MSD-6	101%	99%	4%	104%	105%	3%	144%	139%	7%
	020900-RTS-MS-5	NA			NA			124% 2nd		
	020900-RTS-MSD-6	NA	NA	NA	NA	NA	NA	126% 2nd	125%	2%
Group 1 Control 0.0 mg/kg	C90712M	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90716M	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90722M	0.01			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90724M	0.0116			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90726M	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90728M	0.0131			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90735M	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90742M	0.00			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90743M	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90747M	0.01			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90750M	0.0504			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90758M	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C90765M	<LOQ (0.00394 ug/mL)		131	<LOQ (0.00493 ug/mL)		NA	<LOQ (0.00493 ug/mL)		NA
	C90778M	0.0107	0.00936	0.0123	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	0.0413			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91131F	0.0342			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91138F	0.0095			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91144F	0.087			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91148F	0.0217			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91157F	0.0171			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91159F	0.0333			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91166F	0.0171			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91172F	0.0086			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91176F	<LOQ (0.00394 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91183F	0.0189			<LOQ (0.00493 ug/mL)			<LOQ (0.00493 ug/mL)		
	C91188F	0.0560		87.2	<LOQ (0.00493 ug/mL)		NA	<LOQ (0.00493 ug/mL)		NA
	C91190F	<LOQ (0.00394 ug/mL)	0.0271	0.0237	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.00 mg/kg	C90781M	0.0819			0.00670			0.141		
	C90782M	0.405			0.0128			0.538		
	C90783M	1.52			0.0304			0.642		
	C90785M	0.0665			0.0217			0.189		
	C90787M	1.26			0.0125			0.289		
	C90788M	3.69			0.0395			0.84		
	C90791M	1.89			0.0139			0.81		
	C90794M	2.19			0.0374			0.670		
	C90799M	0.502			0.00900			0.212		
	C90804M	0.319			0.00728			0.214		
	C90809M	4.59			0.0339			0.94		
	C90810M	0.142			0.0213			0.764		
	C90811M	0.304			<LOQ (4.93 ng/mL)			0.105		
	C90812M	0.175			0.00590			0.617		
	C90815M	0.327			0.00515			0.278		
	C90823M	0.86			0.00667			0.174		
	C90829M	1.42			0.0227			0.475		
	C90830M	4.56			0.0370			0.579		
	C90832M	0.87			0.0314			1.56		
	C90835M	0.29			0.0196			0.684		
	C90838M	0.184		120	0.0115		64.0	0.769		62.4
	C90840M	0.215	1.18	1.41	0.00574	0.0187	0.0119	0.685	0.553	0.345
Group 2 Low Dose 3.00 mg/kg	C91197F	1.59			0.0170			0.327		
	C91201F	1.50			0.0133			0.368		
	C91207F	5.89			0.0492			0.63		
	C91216F	E			E			E		
	C91220F	4.82			0.0504			1.30		
	C91221F	0.96			0.00943			0.380		
	C91227F	4.12			0.0166			0.609		
	C91232F	4.62			0.253			3.60		
	C91235F	9.98			0.309			4.39		
	C91236F	3.70			0.129			2.72		
	C91242F	2.98		70.7	0.0621		122.5058	1.37		94
	C91243F	1.13	3.75	2.65	0.0220	0.0846	0.1037	0.896	1.51	1.42

A= CCVs analyzed bracketing these data were within criteria but had peak areas slightly above the valid linear range. LAC 05/03/01
E = Lost during extraction, no sample remaining to reextract.
PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuoroactane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/17/00, 5/16/00, 06/22/00, 06/23/00, 06/26/00, 09/05/00, 10/17/00, 04/13/01 LAC/CSH 4/17/01 mmh
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 SAL/RWW
Dates of Analysis/Analyst:	02/18/00, 03/15/00, 4/18/00, 4/19/00, 06/19/00, 06/20/00, 08/16/00, 08/23/00 MMH/IAS/CSH
Date of Data Reduction/Analyst:	02/21/00, 03/16/00, 4/19/00, 4/20/00, 06/20/00, 06/21/00, 08/18/00, 08/25/00, 04/11/01 MMH/IAS/HOJ
Sample Data	Box 00-009

WEEK 105 RAT SERA

Group Dose	Sample #	Concentration of EtFOSE-OH ug/mL or % Rec	Average EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of M556 ug/mL or % Rec	Average M556 ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSEA ug/mL or % Rec	Average PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	020700-H2O Blk-5	<LOQ (0.00977 ug/mL)			<LOQ (0.00977 ug/mL) A			<LOQ (0.00492 ug/mL)		
	020700-H2O Blk-6	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL) A	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020800-H2O Blk-1	<LOQ (0.00977 ug/mL)			<LOQ (0.00977 ug/mL) A			<LOQ (0.00492 ug/mL)		
	020800-H2O Blk-2	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020900-H2O Blk-5	<LOQ (0.0248 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	020900-H2O Blk-6	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
Matrix Blk	020700-RBS Blk-5	<LOQ (0.00977 ug/mL)			<LOQ (0.00977 ug/mL) A			<LOQ (0.00492 ug/mL)		
	020700-RBS Blk-6	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.00977 ug/mL) A	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020800-RBS Blk-1	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	020800-RBS-Blk-2	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020800-RTS Blk-1	<LOQ (0.00977 ug/mL)			<LOQ 0.00497 ug/mL)			<LOQ (0.00492 ug/mL)		
	020800-RTS-Blk-2	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ 0.00497 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020900-RBS Blk-5	<LOQ (0.0248 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	020900-RBS-Blk-6	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
	020900-RTS Blk-5	<LOQ (0.0248 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	020900-RTS-Blk-6	<LOQ (0.0248 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
QC-250 ppb	020700-RTS-MS-5	34%			153%			79%		
	020700-RTS-MSD-5	28%	31%	19%	153%	153%	0%	75%	77%	5%
	020700-RTS-MS-5	NA			139% 2nd			NA		
	020700-RTS-MSD-5	NA	NA	NA	145% 2nd	142%	4%	NA	NA	NA
	020700-RTS-MS-6	33%			146%			79%		
	020700-RTS-MSD-6	26%	30%	23%	142%	144%	3%	60%	70%	27%
	020700-RTS-MS-6	NA			143% 2nd			NA		
	020700-RTS-MSD-6	NA	NA	NA	141% 2nd	142%	1%	NA	NA	NA
	020800-RTS-MS-1	21%			100%			46%		
	020800-RTS-MSD-1	25%	23%	20%	102% A	101%	1%	65%	55%	34%
	020800-RTS-MS-1	32% 2nd			NA			42% 2nd		
	020800-RTS-MSD-1	NA	32%	NA	NA	NA	NA	NA	42%	NA
	020800-RTS-MS-2	21%			100%			46%		
	020800-RTS-MSD-2	25%	23%	17%	93% A	96%	7%	56%	51%	18%
	020800-RTS-MS-2	38% 2nd			NA			45% 2nd		
	020800-RTS-MSD-2	NA	38%	NA	NA	NA	NA	NA	45%	NA
	C90778M-MS-5	122%			93%			83%		
	C90778M-MSD-6	127%	125%	4%	86%	90%	8%	87%	85%	4%
	020900-RTS-MS-5	26%			137%			61%		
	020900-RTS-MSD-6	29%	27%	10%	139%	138%	1%	60%	60%	1%
	020900-RTS-MS-5	37% 2nd			131% 2nd			57% 2nd		
	020900-RTS-MSD-6	39% 2nd	38%	4%	127% 2nd	129%	3%	56% 2nd	57%	2%
Group 1 Control 0.0 mg/kg	C90712M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90716M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90722M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90724M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90726M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90728M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90735M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90742M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90743M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90747M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90750M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90758M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C90765M	<LOQ (0.00977 ug/mL)		NA	<LOQ (0.00494 ug/mL)		NA	<LOQ (0.00492 ug/mL)		NA
	C90778M	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91131F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91138F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91144F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91148F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91157F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91159F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91166F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91172F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91176F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91183F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91188F	<LOQ (0.00977 ug/mL)		NA	<LOQ (0.00494 ug/mL)		NA	<LOQ (0.00492 ug/mL)		NA
	C91190F	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.00 mg/kg	C90781M	<LOQ (0.00977 ug/mL)			0.0445			<LOQ (0.00492 ug/mL)		
	C90782M	<LOQ (0.00977 ug/mL)			0.123			<LOQ (0.00492 ug/mL)		
	C90783M	<LOQ (0.00977 ug/mL)			0.264			<LOQ (0.00492 ug/mL)		
	C90785M	<LOQ (0.00977 ug/mL)			0.0362			<LOQ (0.00492 ug/mL)		
	C90787M	<LOQ (0.00977 ug/mL)			0.0768			<LOQ (0.00492 ug/mL)		
	C90788M	<LOQ (0.00977 ug/mL)			0.534			<LOQ (0.00492 ug/mL)		
	C90791M	<LOQ (0.00977 ug/mL)			0.195			<LOQ (0.00492 ug/mL)		
	C90794M	<LOQ (0.00977 ug/mL)			0.217			<LOQ (0.00492 ug/mL)		
	C90799M	<LOQ (0.00977 ug/mL)			0.161			<LOQ (0.00492 ug/mL)		
	C90804M	<LOQ (0.00977 ug/mL)			0.0215			<LOQ (0.00492 ug/mL)		
	C90809M	<LOQ (0.00977 ug/mL)			0.255			<LOQ (0.00492 ug/mL)		
	C90810M	<LOQ (0.00977 ug/mL)			0.0558			<LOQ (0.00492 ug/mL)		
	C90811M	<LOQ (0.00977 ug/mL)			0.0916			<LOQ (0.00492 ug/mL)		
	C90812M	<LOQ (0.00977 ug/mL)			0.0610			<LOQ (0.00492 ug/mL)		
	C90815M	<LOQ (0.00977 ug/mL)			0.112			<LOQ (0.00492 ug/mL)		
	C90823M	<LOQ (0.00977 ug/mL)			0.175			<LOQ (0.00492 ug/mL)		
	C90829M	<LOQ (0.00977 ug/mL)			0.145			<LOQ (0.00492 ug/mL)		
	C90830M	<LOQ (0.00977 ug/mL)			0.276			<LOQ (0.00492 ug/mL)		
	C90832M	<LOQ (0.00977 ug/mL)			0.122			<LOQ (0.00492 ug/mL)		
	C90835M	<LOQ (0.00977 ug/mL)			0.0499			<LOQ (0.00492 ug/mL)		
	C90838M	<LOQ (0.00977 ug/mL)		NA	0.0950		79.4	<LOQ (0.00492 ug/mL)		NA
	C90840M	<LOQ (0.00977 ug/mL)	<LOQ	NA	0.0888	0.145	0.115	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 2 Low Dose 3.00 mg/kg	C91197F	<LOQ (0.00977 ug/mL)			0.285			<LOQ (0.00492 ug/mL)		
	C91201F	<LOQ (0.00977 ug/mL)			0.200			<LOQ (0.00492 ug/mL)		
	C91207F	<LOQ (0.00977 ug/mL)			0.300			<LOQ (0.00492 ug/mL)		
	C91216F	E			E			E		
	C91220F	<LOQ (0.00977 ug/mL)			0.355			<LOQ (0.00492 ug/mL)		
	C91221F	<LOQ (0.00977 ug/mL)			0.118			<LOQ (0.00492 ug/mL)		
	C91227F	<LOQ (0.00977 ug/mL)			0.164			<LOQ (0.00492 ug/mL)		
	C91232F	<LOQ (0.00977 ug/mL)			1.59			<LOQ (0.00492 ug/mL)		
	C91235F	<LOQ (0.00977 ug/mL)			1.90			<LOQ (0.00492 ug/mL)		
	C91236F	<LOQ (0.00977 ug/mL)			1.43			<LOQ (0.00492 ug/mL)		
	C91242F	<LOQ (0.00977 ug/mL)		NA	0.980		98	<LOQ (0.00492 ug/mL)		NA
	C91243F	<LOQ (0.00977 ug/mL)	<LOQ	NA	0.166	0.680	0.667	<LOQ (0.00492 ug/mL)	<LOQ	NA

A= CCVs analyzed bracketing these data were within criteria but had peak areas slightly above the valid linear range. LAC 05/03/01
E = Lost during extraction, no sample remaining to reextract.
PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.00492 ug/mL) to include std correction factors
new LOQ is 0.00394 ug/mL. LAC 02/19/01

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/17/00, 5/16/00, 06/22/00, 06/23/00, 06/26/00, 09/05/00, 10/17/00, 04/13/01 LAC/CSH 4/17/01 mmh
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00, 2/24/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 02/25/00, 03/27/00, 06/01/00, 06/19/00, 06/20/00, 08/02/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 02/29/00, 06/06/00, 06/02/00, 06/20/00, 06/26/00, 08/03/00, 08/18/00, 08/25/00 MMH/IAS/HOJ

Sample Data **Box** 00-009

WEEK 105 RAT SERA lot 171

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec	Average PFOS ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90842M	1	NA	0.9275	0.8640	500	102	A060100050	40.7		
Mid Dose	C90844M	1	NA	0.9275	0.8640	50	115	A040600039	4.61		
30.0 mg/kg	C90850M	1	NA	0.9275	0.8640	50	273	A040600040	11.0		
	C90851M	1	NA	0.9275	0.8640	50	224	A040600041	8.98		
	C90854M	1	NA	0.9275	0.8640	50	683	A040600042	27.3		
	C90855M	1	NA	0.9275	0.8640	50	483	A040600046	19.4		
	C90856M	1	NA	0.9275	0.8640	50	112	A040600047	4.49		
	C90858M	1	NA	0.9275	0.8640	50	670	A040600048	26.9		
	C90860M	1	NA	0.9275	0.8640	500	208	A060100102	83.2		
	C90864M	1	NA	0.9275	0.8640	50	667	A040600050	26.7		
	C90865M	1	NA	0.9275	0.8640	50	402	A040600054	16.1		
	C90867M	1	NA	0.9275	0.8640	500	122	A060100051	48.9		
	C90869M	1	NA	0.9275	0.8640	50	155	A040600056	6.22		
	C90871M	1	NA	0.9275	0.8640	50	420	A040600062	16.8		
	C90881M	1	NA	0.9275	0.8640	50	460	A040600063	18.4		
	C90882M	1	NA	0.9275	0.8640	50	592	A040600064	23.7		
	C90883M	1	NA	0.9275	0.8640	50	374	A040600057	15.0		
	C90884M	1	NA	0.9275	0.8640	50	391	A040600065	15.7		
	C90885M	1	NA	0.9275	0.8640	50	623	A040600058	24.9		
	C90889M	1	NA	0.9275	0.8640	50	159	A040600066	6.35		
	C90893M	1	NA	0.9275	0.8640	50	578	A040600071	23.2		80.0
	C90900M	1	NA	0.9275	0.8640	50	387	A040600070	15.5	22.0	17.6
Group 3	C91252F	1	NA	0.9275	0.8640	100	577	A040600014	46.2		
Mid Dose	C91253F	1	NA	0.9275	0.8640	100	674	A040600015	54.0		
30.0 mg/kg	C91254F	1	NA	0.9275	0.8640	1000	119	A060100049	95.5		
	C91256F	1	NA	0.9275	0.8640	100	823	A040600017	66.0		
	C91264F	1	NA	0.9275	0.8640	100	571	A040600018	45.7		
	C91278F	1	NA	0.9275	0.8640	1000	138	A060100100	110		
	C91279F	1	NA	0.9275	0.8640	100	139	A040600023	11.2		
	C91286F	1	NA	0.9275	0.8640	100	868	A040600024	69.5		
	C91292F	1	NA	0.9275	0.8640	100	210	A040600025	16.9		
	C91294F	1	NA	0.9275	0.8640	100	774	A040600026	62.0		
	C91295F	1	NA	0.9275	0.8640	100	185	A040600030	14.8		
	C91301F	1	NA	0.9275	0.8640	100	753	A040600031	60.4		
	C91303F	1	NA	0.9275	0.8640	500	128	A060100101	51.4		
	C91307F	1	NA	0.9275	0.8640	100	883	A040600033	70.8		49.7
	C91310F	1	NA	0.9275	0.8640	100	665	A040600034	53.3	55.2	27.4

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 04/16/00, 05/16/00, 06/12/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00, 2/24/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 02/25/00, 03/27/00, 06/01/00, 06/19/00, 06/20/00, 08/02/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 02/29/00, 06/06/00, 06/02/00, 06/20/00, 06/26/00, 08/03/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot L-15709

Group Dose	Sample #	PFOSA Purity Correction Factor	Surrogate Verified	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Average PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90842M	Unknown	NA	1	196	A060100052	0.196		
Mid Dose	C90844M	Unknown	NA	1	155	A060100053	0.155		
30.0 mg/kg	C90850M	Unknown	NA	1	187	A060100056	0.187		
	C90851M	Unknown	NA	1	83.7	A060100017	0.0837		
	C90854M	Unknown	NA	1	287	A060100057	0.287		
	C90855M	Unknown		10	34.6	A040600080	0.346		
	C90856M	Unknown	NA	1	223	A060100105	0.223		
	C90858M	Unknown	NA	1	217	A060100107	0.217		
	C90860M	Unknown	NA	1	338	A060100108	0.338		
	C90864M	Unknown	NA	1	272	A060100058	0.272		
	C90865M	Unknown	NA	1	238	D081600040	0.238		
	C90867M	Unknown	NA	1	430	A060100059	0.430		
	C90869M	Unknown	NA	1	258	A060100109	0.258		
	C90871M	Unknown	NA	1	343	A060100110	0.343		
	C90881M	Unknown	NA	1	536	A060100060	0.536		
	C90882M	Unknown	NA	1	482	A060100063	0.482		
	C90883M	Unknown	NA	1	436	A060100022	0.436		
	C90884M	Unknown	NA	1	232	A060100064	0.232		
	C90885M	Unknown	NA	1	414	D081600041	0.414		
	C90889M	Unknown	NA	1	315	A060100111	0.315		
	C90893M	Unknown	NA	1	459	A060100065	0.459		37.9
	C90900M	Unknown	NA	1	296	A060100066	0.296	0.306	0.116
Group 3	C91252F	Unknown	NA	1	369	A060100114	0.369		
Mid Dose	C91253F	Unknown	NA	1	484	A060100115	0.484		
30.0 mg/kg	C91254F	Unknown	NA	1	380	A060100116	0.380		
	C91256F	Unknown	NA	1	572	A060100117	0.572		
	C91264F	Unknown	NA	1	340	D081600044	0.340		
	C91278F	Unknown		10	71.7	A022700024	0.717		
	C91279F	Unknown	NA	1	206	A060100071	0.206		
	C91286F	Unknown	NA	1	497	A060100118	0.497		
	C91292F	Unknown	NA	1	447	A060100121	0.447		
	C91294F	Unknown	NA	1	542	A060100072	0.542		
	C91295F	Unknown	NA	1	306	A050100015	0.306		
	C91301F	Unknown	NA	1	263	A050100122	0.263		
	C91303F	Unknown	NA	1	368	A050100123	0.368		
	C91307F	Unknown	NA	1	536	A050100073	0.536		30.9
	C91310F	Unknown	NA	1	401	A050100016	0.401	0.429	0.133

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 04/16/00, 05/16/00, 06/12/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00, 2/24/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 02/25/00, 03/27/00, 06/01/00, 06/19/00, 06/20/00, 08/02/00, 08/16/00 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 02/29/00, 06/06/00, 06/02/00, 06/20/00, 06/26/00, 08/03/0(, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot T-7121.1

Group Dose	Sample #	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Dilution Factor	PFOSAA Conc. ng/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Average PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90842M	Unknown	Confirmed High	10	187	A040600072	1.87		
Mid Dose	C90844M	Unknown	NA	10	132	A040600073	1.32		
30.0 mg/kg	C90850M	Unknown	NA	10	308	A040600074	3.08		
	C90851M	Unknown	NA	10	98.4	A040600078	0.984		
	C90854M	Unknown	NA	10	715	A040600079	7.15		
	C90855M	Unknown	NA	10	828	A040600080	8.28		
	C90856M	Unknown	NA	10	509	A040600081	5.09		
	C90858M	Unknown	NA	10	379	A040600082	3.79		
	C90860M	Unknown	NA	10	376	A040600086	3.76		
	C90864M	Unknown	NA	10	355	A040600087	3.55		
	C90865M	Unknown	NA	10	166	A040600088	1.66		
	C90867M	Unknown	NA	10	287	A040600089	2.87		
	C90869M	Unknown	NA	10	455	A040600090	4.55		
	C90871M	Unknown	NA	10	330	A040600094	3.30		
	C90881M	Unknown	NA	10	472	A040600095	4.72		
	C90882M	Unknown	NA	10	390	A040600096	3.90		
	C90883M	Unknown	NA	10	435	A040600097	4.35		
	C90884M	Unknown	NA	10	296	A040600098	2.96		
	C90885M	Unknown	NA	10	605	A040600102	6.05		
	C90889M	Unknown	NA	10	652	A040600103	6.52		
	C90893M	Unknown	NA	10	481	A040600104	4.81		46.4
	C90900M	Unknown	NA	10	647	A040600105	6.47	4.14	1.92
Group 3	C91252F	Unknown	NA	10	373	A022700017	3.73		
Mid Dose	C91253F	Unknown	NA	10	733	A022700018	7.33		
30.0 mg/kg	C91254F	Unknown	NA	10	646	A022700019	6.46		
	C91256F	Unknown	NA	10	791	A022700020	7.91		
	C91264F	Unknown	NA	10	316	A022700021	3.16		
	C91278F	Unknown	NA	100	95.9	A060100103	9.59		
	C91279F	Unknown	NA	10	334	A022700025	3.34		
	C91286F	Unknown	NA	10	932	A022700026	9.32		
	C91292F	Unknown	NA	10	525	A022700027	5.25		
	C91294F	Unknown	NA	10	781	A022700028	7.81		
	C91295F	Unknown	NA	10	614	A022700031	6.14		
	C91301F	Unknown	NA	10	349	A022700032	3.49		
	C91303F	Unknown	NA	10	524	A022700033	5.24		
	C91307F	Unknown	NA	10	721	A022700034	7.21		34.2
	C91310F	Unknown	NA	10	614	A022700035	6.14	6.14	2.10

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 04/16/00, 05/16/00, 06/12/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00, 2/24/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 02/25/00, 03/27/00, 06/01/00, 06/19/00, 06/20/00, 08/02/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 02/29/00, 06/06/00, 06/02/00, 06/20/00, 06/26/00, 08/03/00, 08/18/00 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot Unknown/SD013

Group Dose	Sample #	EtFOSE-OH Purity Correction Factor	Surrogate Verified	EtFOSE-OH Dilution Factor	EtFOSE-OH Conc. ng/mL	Filename	Concentration of EtFOSE-OH ug/mL or % Rec	Average EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90842M	Unknown	NA	1	5.15	A080200017	0.00515		
Mid Dose	C90844M	Unknown	NA	1	1.89	A080200018	<LOQ (0.00493 ug/mL)		
30.0 mg/kg	C90850M	Unknown	NA	1	5.10	A080200019	0.00510		
	C90851M	Unknown	NA	1	3.82	A080200020	<LOQ (0.00493 ug/mL)		
	C90854M	Unknown	NA	1	1.90	A080200021	<LOQ (0.00493 ug/mL)		
	C90855M	Unknown	NA	1	3.37	A080200024	<LOQ (0.00493 ug/mL)		
	C90856M	Unknown	NA	1	3.76	A080200025	<LOQ (0.00493 ug/mL)		
	C90858M	Unknown	NA	1	3.30	A080200026	<LOQ (0.00493 ug/mL)		
	C90860M	Unknown	NA	1	4.90	A080200027	<LOQ (0.00493 ug/mL)		
	C90864M	Unknown	NA	1	10.3	A080200028	0.0103		
	C90865M	Unknown	NA	1	5.47	A080200031	0.00547		
	C90867M	Unknown	NA	1	6.66	A080200032	0.00666		
	C90869M	Unknown	NA	1	6.06	A080200033	0.00606		
	C90871M	Unknown	NA	1	2.60	A080200034	<LOQ (0.00493 ug/mL)		
	C90881M	Unknown	NA	1	9.89	A080200035	0.00989		
	C90882M	Unknown	NA	1	18.2	A080200038	0.0182		
	C90883M	Unknown	NA	1	1.86	A080200039	<LOQ (0.00493 ug/mL)		
	C90884M	Unknown	NA	1	3.89	A080200040	<LOQ (0.00493 ug/mL)		
	C90885M	Unknown	NA	1	15.2	A080200041	0.0152		
	C90889M	Unknown	NA	1	7.71	A080200042	0.00771		
	C90893M	Unknown	NA	1	10.6	A080200045	0.0106		47.2
	C90900M	Unknown	NA	1	4.68	A080200046	<LOQ (0.00493 ug/mL)	0.00912	0.00430
Group 3	C91252F	Unknown	NA	1	8.42	A080200047	0.00842		
Mid Dose	C91253F	Unknown	NA	1	8.56	A080200048	0.00856		
30.0 mg/kg	C91254F	Unknown	NA	1	21.3	A080200049	0.0213		
	C91256F	Unknown	NA	1	5.64	A080200052	0.00564		
	C91264F	Unknown	NA	1	5.17	A080200053	0.00517		
	C91278F	Unknown	NA	1	17.8	A080200054	0.0178		
	C91279F	Unknown	NA	1	1.90	A080200055	<LOQ (0.00493 ug/mL)		
	C91286F	Unknown	NA	1	6.46	A080200056	0.00646		
	C91292F	Unknown	NA	1	15.3	A080200059	0.0153		
	C91294F	Unknown	NA	1	12.8	A080200060	0.0128		
	C91295F	Unknown	NA	1	0.570	A080200061	<LOQ (0.00493 ug/mL)		
	C91301F	Unknown	NA	1	6.03	A080200062	0.00603		
	C91303F	Unknown	2nd Analysis OK	1	9.21	D081600046	0.00921		
	C91307F	Unknown	NA	1	12.8	A080200066	0.0128		48.4
	C91310F	Unknown	NA	1	1.36	A080200067	<LOQ (0.00493 ug/mL)	0.0108	0.00522

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 04/16/00, 05/16/00, 06/12/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00, 2/24/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 02/25/00, 03/27/00, 06/01/00, 06/19/00, 06/20/00, 08/02/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 02/29/00, 06/06/00, 06/02/00, 06/20/00, 06/26/00, 08/03/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot NB113047-80

Group Dose	Sample #	M556 Purity Correction Factor	Surrogate Verified	M556 Dilution Factor	M556 Conc. ng/mL	Filename	Concentration of M556 ug/mL or % Rec	Average M556 ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90842M	Unknown	Confirmed High	10	254	A022500074	2.54		
Mid Dose	C90844M	Unknown	NA	10	116.0	A022500075	1.160		
30.0 mg/kg	C90850M	Unknown	NA	10	214	A022500076	2.14		
	C90851M	Unknown	NA	10	169	A022500079	1.69		
	C90854M	Unknown	NA	10	187	A022500080	1.87		
	C90855M	Unknown	NA	10	162	A022500081	1.62		
	C90856M	Unknown	NA	10	278	A022500082	2.78		
	C90858M	Unknown	NA	10	610	A022500083	6.10		
	C90860M	Unknown	NA	10	264	A022500086	2.64		
	C90864M	Unknown	NA	10	228	A022500087	2.28		
	C90865M	Unknown	NA	10	337	A022500088	3.37		
	C90867M	Unknown	NA	10	208	A022500089	2.08		
	C90869M	Unknown	NA	10	270	A022500090	2.70		
	C90871M	Unknown	NA	10	192	A022500093	1.92		
	C90881M	Unknown	NA	10	378	A022500094	3.78		
	C90882M	Unknown	NA	10	294	A022500095	2.94		
	C90883M	Unknown	NA	10	534	A022500096	5.34		
	C90884M	Unknown	NA	10	334	A022500097	3.34		
	C90885M	Unknown	NA	10	284	A022500100	2.84		
	C90889M	Unknown	NA	10	440	A022500101	4.40		
	C90893M	Unknown	NA	10	252	A022500102	2.52		41.7
	C90900M	Unknown	NA	10	286	A022500103	2.86	2.86	1.191
Group 3	C91252F	Unknown	NA	10	385	A022700017	3.85		
Mid Dose	C91253F	Unknown	NA	10	474	A022700018	4.74		
30.0 mg/kg	C91254F	Unknown	NA	10	324	A022700019	3.24		
	C91256F	Unknown	NA	10	291	A022700020	2.91		
	C91264F	Unknown	NA	10	178	A022700021	1.78		
	C91278F	Unknown	NA	10	323	A022700024	3.23		
	C91279F	Unknown	NA	10	176	A022700025	1.76		
	C91286F	Unknown	NA	10	312	A022700026	3.12		
	C91292F	Unknown	NA	10	470	A022700027	4.70		
	C91294F	Unknown	NA	10	346	A022700028	3.46		
	C91295F	Unknown	NA	10	209	A022700031	2.09		
	C91301F	Unknown	NA	10	348	A022700032	3.48		
	C91303F	Unknown	NA	10	381	A022700033	3.81		
	C91307F	Unknown	NA	10	125	A022700034	1.25		35.7
	C91310F	Unknown	NA	10	181	A022700035	1.81	3.02	1.08

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 04/16/00, 05/16/00, 06/12/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00, 2/24/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 02/25/00, 03/27/00, 06/01/00, 06/19/00, 06/20/00, 08/02/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 02/29/00, 06/06/00, 06/02/00, 06/20/00, 06/26/00, 08/03/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot 529

Group Dose	Sample #	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Dilution Factor	PFOSEA Conc. ng/mL	Filename	Concentration of PFOSEA ug/mL or % Rec	Average PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90842M	Unknown	NA	1	0.00	A080200017	<LOQ (0.00492 ug/mL)		
Mid Dose	C90844M	Unknown	NA	1	0.00	A080200018	<LOQ (0.00492 ug/mL)		
30.0 mg/kg	C90850M	Unknown	NA	1	0.0300	A080200019	<LOQ (0.00492 ug/mL)		
	C90851M	Unknown	NA	1	0.00	A080200020	<LOQ (0.00492 ug/mL)		
	C90854M	Unknown	NA	1	1.04	A080200021	<LOQ (0.00492 ug/mL)		
	C90855M	Unknown	NA	1	0.210	A080200024	<LOQ (0.00492 ug/mL)		
	C90856M	Unknown	NA	1	0.00	A080200025	<LOQ (0.00492 ug/mL)		
	C90858M	Unknown	NA	1	1.44	A080200026	<LOQ (0.00492 ug/mL)		
	C90860M	Unknown	NA	1	0.00	A080200027	<LOQ (0.00492 ug/mL)		
	C90864M	Unknown	NA	1	0.00	A080200028	<LOQ (0.00492 ug/mL)		
	C90865M	Unknown	NA	1	0.00	A080200031	<LOQ (0.00492 ug/mL)		
	C90867M	Unknown	NA	1	0.00	A080200032	<LOQ (0.00492 ug/mL)		
	C90869M	Unknown	NA	1	0.00	A080200033	<LOQ (0.00492 ug/mL)		
	C90871M	Unknown	NA	1	0.00	A080200034	<LOQ (0.00492 ug/mL)		
	C90881M	Unknown	NA	1	0.00	A080200035	<LOQ (0.00492 ug/mL)		
	C90882M	Unknown	NA	1	2.88	A080200038	<LOQ (0.00492 ug/mL)		
	C90883M	Unknown	NA	1	0.00	A080200039	<LOQ (0.00492 ug/mL)		
	C90884M	Unknown	NA	1	0.00	A080200040	<LOQ (0.00492 ug/mL)		
	C90885M	Unknown	NA	1	0.0800	A080200041	<LOQ (0.00492 ug/mL)		
	C90889M	Unknown	NA	1	4.85	A080200042	<LOQ (0.00492 ug/mL)		
	C90893M	Unknown	NA	1	0.480	A080200045	<LOQ (0.00492 ug/mL)		NA
	C90900M	Unknown	NA	1	0.00	A080200046	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 3	C91252F	Unknown	NA	1	0.00	A080200047	<LOQ (0.00492 ug/mL)		
Mid Dose	C91253F	Unknown	NA	1	0.00	A080200048	<LOQ (0.00492 ug/mL)		
30.0 mg/kg	C91254F	Unknown	NA	1	0.00	A080200049	<LOQ (0.00492 ug/mL)		
	C91256F	Unknown	NA	1	0.00	A080200052	<LOQ (0.00492 ug/mL)		
	C91264F	Unknown	NA	1	0.710	A080200053	<LOQ (0.00492 ug/mL)		
	C91278F	Unknown	NA	1	0.320	A080200054	<LOQ (0.00492 ug/mL)		
	C91279F	Unknown	NA	1	0.00	A080200055	<LOQ (0.00492 ug/mL)		
	C91286F	Unknown	NA	1	0.640	A080200056	<LOQ (0.00492 ug/mL)		
	C91292F	Unknown	NA	1	0.190	A080200059	<LOQ (0.00492 ug/mL)		
	C91294F	Unknown	NA	1	0.00	A080200060	<LOQ (0.00492 ug/mL)		
	C91295F	Unknown	NA	1	0.00	A080200061	<LOQ (0.00492 ug/mL)		
	C91301F	Unknown	NA	1	0.00	A080200062	<LOQ (0.00492 ug/mL)		
	C91303F	Unknown	2nd Analysis OK	1	0.00	D081600046	<LOQ (0.00492 ug/mL)		
	C91307F	Unknown	NA	1	1.19	A080200066	<LOQ (0.00492 ug/mL)		NA
	C91310F	Unknown	NA	1	0.00	A080200067	<LOQ (0.00492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
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Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 04/16/00, 05/16/00, 06/12/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00, 2/24/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 02/25/00, 03/27/00, 06/01/00, 06/19/00, 06/20/00, 08/02/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 02/29/00, 06/06/00, 06/02/00, 06/20/00, 06/26/00, 08/03/00, 08/18/00, 08/25/00 MMH/IAS/HOJ

Sample Data

WEEK 105 RAT SERA

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Average PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Average PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Average PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90842M	40.7			0.196			1.87		
Mid Dose	C90844M	4.61			0.155			1.32		
30.0 mg/kg	C90850M	11.0			0.187			3.08		
	C90851M	8.98			0.0837			0.984		
	C90854M	27.3			0.287			7.15		
	C90855M	19.4			0.3459000			8.28		
	C90856M	4.49			0.223			5.09		
	C90858M	26.9			0.217			3.79		
	C90860M	83.2			0.338			3.76		
	C90864M	26.7			0.272			3.55		
	C90865M	16.1			0.238			1.66		
	C90867M	48.9			0.430			2.87		
	C90869M	6.22			0.258			4.55		
	C90871M	16.8			0.343			3.30		
	C90881M	18.4			0.536			4.72		
	C90882M	23.7			0.482			3.90		
	C90883M	15.0			0.436			4.35		
	C90884M	15.7			0.232			2.96		
	C90885M	24.9			0.414			6.05		
	C90889M	6.35			0.315			6.52		
	C90893M	23.2		80.0	0.459		37.9	4.81		46.4
	C90900M	15.5	22.0	17.6	0.296	0.306	0.116	6.47	4.14	1.92
Group 3	C91252F	46.2			0.369			3.73		
Mid Dose	C91253F	54.0			0.484			7.33		
30.0 mg/kg	C91254F	95.5			0.380			6.46		
	C91256F	66.0			0.572			7.91		
	C91264F	45.7			0.340			3.16		
	C91278F	110			0.7173000			9.59		
	C91279F	11.2			0.206			3.34		
	C91286F	69.5			0.497			9.32		
	C91292F	16.9			0.447			5.25		
	C91294F	62.0			0.542			7.81		
	C91295F	14.8			0.306			6.14		
	C91301F	60.4			0.263			3.49		
	C91303F	51.4			0.368			5.24		
	C91307F	70.8		49.7	0.536		30.9	7.21		34.2
	C91310F	53.3	55.2	27.4	0.401	0.429	0.133	6.14	6.14	2.10

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 04/16/00, 05/16/00, 06/12/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00, 2/24/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 02/25/00, 03/27/00, 06/01/00, 06/19/00, 06/20/00, 08/02/00, 08/16/00, 08/23/00 MMI-/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 02/29/00, 06/06/00, 06/02/00, 06/20/00, 06/26/00, 08/03/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009

WEEK 105 RAT SERA

Group Dose	Sample #	Concentration of EtFOSE-OH ug/mL or % Rec	Average EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of M556 ug/mL or % Rec	Average M556 ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSEA ug/mL or % Rec	Average PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 3	C90842M	0.00515			2.54			<LOQ (0.00492 ug/mL)		
Mid Dose	C90844M	<LOQ (0.00493 ug/mL)			1.160			<LOQ (0.00492 ug/mL)		
30.0 mg/kg	C90850M	0.00510			2.14			<LOQ (0.00492 ug/mL)		
	C90851M	<LOQ (0.00493 ug/mL)			1.69			<LOQ (0.00492 ug/mL)		
	C90854M	<LOQ (0.00493 ug/mL)			1.87			<LOQ (0.00492 ug/mL)		
	C90855M	<LOQ (0.00493 ug/mL)			1.62			<LOQ (0.00492 ug/mL)		
	C90856M	<LOQ (0.00493 ug/mL)			2.78			<LOQ (0.00492 ug/mL)		
	C90858M	<LOQ (0.00493 ug/mL)			6.10			<LOQ (0.00492 ug/mL)		
	C90860M	<LOQ (0.00493 ug/mL)			2.64			<LOQ (0.00492 ug/mL)		
	C90864M	0.0103			2.28			<LOQ (0.00492 ug/mL)		
	C90865M	0.00547			3.37			<LOQ (0.00492 ug/mL)		
	C90867M	0.00666			2.08			<LOQ (0.00492 ug/mL)		
	C90869M	0.00606			2.70			<LOQ (0.00492 ug/mL)		
	C90871M	<LOQ (0.00493 ug/mL)			1.92			<LOQ (0.00492 ug/mL)		
	C90881M	0.00989			3.78			<LOQ (0.00492 ug/mL)		
	C90882M	0.0182			2.94			<LOQ (0.00492 ug/mL)		
	C90883M	<LOQ (0.00493 ug/mL)			5.34			<LOQ (0.00492 ug/mL)		
	C90884M	<LOQ (0.00493 ug/mL)			3.34			<LOQ (0.00492 ug/mL)		
	C90885M	0.0152			2.84			<LOQ (0.00492 ug/mL)		
	C90889M	0.00771			4.40			<LOQ (0.00492 ug/mL)		
	C90893M	0.0106		47.2	2.52		41.7	<LOQ (0.00492 ug/mL)		NA
	C90900M	<LOQ (0.00493 ug/mL)	0.00912	0.00430	2.86	2.86	1.191	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 3	C91252F	0.00842			3.85			<LOQ (0.00492 ug/mL)		
Mid Dose	C91253F	0.00856			4.74			<LOQ (0.00492 ug/mL)		
30.0 mg/kg	C91254F	0.0213			3.24			<LOQ (0.00492 ug/mL)		
	C91256F	0.00564			2.91			<LOQ (0.00492 ug/mL)		
	C91264F	0.00517			1.78			<LOQ (0.00492 ug/mL)		
	C91278F	0.0178			3.23			<LOQ (0.00492 ug/mL)		
	C91279F	<LOQ (0.00493 ug/mL)			1.76			<LOQ (0.00492 ug/mL)		
	C91286F	0.00646			3.12			<LOQ (0.00492 ug/mL)		
	C91292F	0.0153			4.70			<LOQ (0.00492 ug/mL)		
	C91294F	0.0128			3.46			<LOQ (0.00492 ug/mL)		
	C91295F	<LOQ (0.00493 ug/mL)			2.09			<LOQ (0.00492 ug/mL)		
	C91301F	0.00603			3.48			<LOQ (0.00492 ug/mL)		
	C91303F	0.00921			3.81			<LOQ (0.00492 ug/mL)		
	C91307F	0.0128		48.4	1.25		35.7	<LOQ (0.00492 ug/mL)		NA
	C91310F	<LOQ (0.00493 ug/mL)	0.0108	0.00522	1.81	3.02	1.08	<LOQ (0.00492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 04/16/00, 05/16/00, 06/12/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	4/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Soup 020199, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 2/28/00, 03/27/00, 05/31/00, 06/19/00, 06/20/00, 08/03/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 3/02/00, 06/02/00, 06/20/00, 06/21/00, 08/07/00, 08/18/00, 08/25/00 MMH/IAS/HOJ

Sample Data **Box** 00-009

WEEK 105 RAT SERA lot 171

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec	Average PFOS ug/mL	RSD Std. Dev. MS/MSD RPD
Group 4	C90904M	1	NA	0.9275	0.8640	200	417	D062000016	66.9		
Mid-High Dose	C90908M	1	NA	0.9275	0.8640	200	443	D062000017	71.0		
100 mg/kg	C90910M	1	NA	0.9275	0.8640	50	786	A053100106	31.5		
	C90911M	1	NA	0.9275	0.8640	200	911	D062000018	146		
	C90916M	1	NA	0.9275	0.8640	200	551	D062000019	88.4		
	C90926M	1	NA	0.9275	0.8640	200	384	D062000020	61.6		
	C90927M	1	NA	0.9275	0.8640	50	548	A053100108	22.0		
	C90928M	1	NA	0.9275	0.8640	200	791	D062000023	127		
	C90933M	1	NA	0.9275	0.8640	200	286	D062000024	45.8		
	C90944M	1	NA	0.9275	0.8640	200	848	D062000025	136		
	C90947M	1	NA	0.9275	0.8640	200	899	D062000026	144		
	C90948M	1	NA	0.9275	0.8640	200	323	D062000027	51.7		
	C90953M	1	NA	0.9275	0.8640	50	324	A053100113	13.0		
	C90955M	1	NA	0.9275	0.8640	200	400	D062000030	64.1		
	C90956M	1	NA	0.9275	0.8640	200	822	D062000031	132		
	C90965M	1	NA	0.9275	0.8640	200	857	D062000032	137		53.6
	C90968M	1	NA	0.9275	0.8640	200	626	D062000033	100	84.6	45.4
Group 4	C91315F	1	NA	0.9275	0.8640	200	762	D062000034	122		
Mid-High Dose	C91324F	1	NA	0.9275	0.8640	200	829	D062000037	133		
100 mg/kg	C91327F	1	NA	0.9275	0.8640	20	612	D062000065	9.81		
	C91332F	1	NA	0.9275	0.8640	500	627	D081600030	251 A		
	C91335F	1	NA	0.9275	0.8640	200	621	D062000040	99.5		
	C91336F	1	NA	0.9275	0.8640	500	504	D081600031	202 A		
	C91338F	1	NA	0.9275	0.8640	200	875	D062000044	140		
	C91340F	1	NA	0.9275	0.8640	500	628	D081600032	252 A		
	C91341F	1	NA	0.9275	0.8640	200	474	D062000046	76.0		
	C91342F	1	NA	0.9275	0.8640	200	761	D062000047	122		
	C91356F	1	NA	0.9275	0.8640	20	386	D062000048	6.18		
	C91357F	1	NA	0.9275	0.8640	500	500	D081600033	200 A		
	C91360F	1	NA	0.9275	0.8640	200	881	D062000052	141		
	C91361F	1	NA	0.9275	0.8640	200	815	D062000053	131		
	C91362F	1	NA	0.9275	0.8640	200	158	D062000054	25.3		
	C91372F	1	NA	0.9275	0.8640	200	367	D062000055	58.8		
	C91376F	1	NA	0.9275	0.8640	200	831	D062000058	133		
	C91378F	1	NA	0.9275	0.8640	200	682	D062000059	109		57.9
	C91380F	1	NA	0.9275	0.8640	200	648	D062000060	104	122	70.5
Group 6	C91047M	1	NA	0.9275	0.8640	1	231	A053100082	0.185		
Mid-High Dose	C91054M	1	NA	0.9275	0.8640	1	27.7	A053100072	0.0222		
Recovery	C91056M	1	NA	0.9275	0.8640	5	293	A053100060	1.18		
100 mg/kg	C91060M	1	NA	0.9275	0.8640	1	177	A053100074	0.142		
	C91065M	1	NA	0.9275	0.8640	1	0.00	A053100075	<LOQ (0.0198 ug/mL)		
	C91070M	1	NA	0.9275	0.8640	1	64.1	A053100078	0.0514		
	C91071M	1	NA	0.9275	0.8640	1	4.80	A053100079	<LOQ (0.0198 ug/mL)		
	C91072M	1	NA	0.9275	0.8640	1	56.6	A053100080	0.0454		
	C91073M	1	NA	0.9275	0.8640	1	0.00	A053100081	<LOQ (0.0198 ug/mL)		166
	C91076M	1	NA	0.9275	0.8640	1	739	A053100085	0.592	0.227	0.377
Group 6	C91451F	1	NA	0.9275	0.8640	10	526	A053100040	5.02		
Mid-High Dose	C91453F	1	NA	0.9275	0.8640	10	210	A053100043	1.68		
Recovery	C91455F	1	NA	0.9275	0.8640	10	739	A053100044	5.92		
100 mg/kg	C91459F	1	NA	0.9275	0.8640	1	0.410	A053100089	<LOQ (0.0198 ug/mL)		
	C91462F	1	NA	0.9275	0.8640	10	576	A053100046	4.61		
	C91467F	1	NA	0.9275	0.8640	1	25.8	A053100093	0.0207		
	C91473F	1	NA	0.9275	0.8640	10	479	A053100050	3.84		
	C91479F	1	NA	0.9275	0.8640	1	0.00	A053100095	<LOQ (0.0198 ug/mL)		
	C91480F	1	NA	0.9275	0.8640	1	69.0	A053100096	0.0553		
	C91485F	1	NA	0.9275	0.8640	10	443	A053100051	3.55		
	C91487F	1	NA	0.9275	0.8640	1	8.20	A053100101	<LOQ (0.0198 ug/mL)		96.3
	C91488F	1	NA	0.9275	0.8640	10	519	A053100052	4.16	2.41	2.32

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.0247 ug/mL) to include std correction factors
new LOQ is 0.0198 ug/mL. LAC 02/19/01
A - data is above the upper limit of quantitation and is provided as an estimate.

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/15/00, 5/16/00, 06/13/00, 6/15/00, 6/19/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	04/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Soup 020199, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 2/28/00, 03/27/00, 05/31/00, 06/19/00, 06/20/00, 08/03/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 3/02/00, 06/02/00, 06/20/00, 06/21/00, 08/07/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot L-15709

Group Dose	Sample #	PFOSA Purity Correction Factor	Surrogate Verified	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Average PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 4	C90904M	Unknown	NA	10	56.8	A022700090	0.568		
Mid-High Dose	C90908M	Unknown	NA	10	60.6	A022700091	0.606		
100 mg/kg	C90910M	Unknown	NA	10	184	A022700094	1.84		
	C90911M	Unknown	NA	10	60.7	A022700054	0.607		
	C90916M	Unknown	NA	10	61.2	A022700055	0.612		
	C90926M	Unknown	NA	10	33.0	A022700095	0.330		
	C90927M	Unknown	NA	10	109	A022700096	1.09		
	C90928M	Unknown	NA	10	97.9	A022700097	0.979		
	C90933M	Unknown	NA	10	59.3	A022700098	0.593		
	C90944M	Unknown	NA	10	56.0	A022700056	0.560		
	C90947M	Unknown	NA	10	68.5	A022700059	0.685		
	C90948M	Unknown	NA	10	27.7	A022700060	0.277		
	C90953M	Unknown	NA	10	57.2	A022700101	0.572		
	C90955M	Unknown	NA	10	61.4	A022700102	0.614		
	C90956M	Unknown	NA	10	39.0	A022700061	0.390		
	C90965M	Unknown	NA	10	94.9	A022700062	0.949		51.9
	C90968M	Unknown	NA	10	63.8	A022700103	0.638	0.701	0.363
Group 4	C91315F	Unknown	NA	10	89.8	A022700077	0.898		
Mid-High Dose	C91324F	Unknown	NA	10	191	A022700080	1.91		
100 mg/kg	C91327F	Unknown	NA	10	102	A022700081	1.02		
	C91332F	Unknown	NA	10	136	A022700082	1.36		
	C91335F	Unknown	NA	10	205	A022700083	2.05		
	C91336F	Unknown	NA	10	107	A022700084	1.07		
	C91338F	Unknown	NA	10	171	A022700087	1.71		
	C91340F	Unknown	NA	10	72.1	A022700088	0.721		
	C91341F	Unknown	NA	10	138	A022700089	1.38		
	C91342F	Unknown	NA	10	82.6	A022700063	0.826		
	C91356F	Unknown	NA	10	34.5	A022700066	0.345		
	C91357F	Unknown	NA	10	201	A022700067	2.01		
	C91360F	Unknown	NA	10	185	A022700068	1.85		
	C91361F	Unknown	NA	10	116	A022700069	1.16		
	C91362F	Unknown	NA	10	72.0	A022700070	0.720		
	C91372F	Unknown	NA	10	161	A022700073	1.61		
	C91376F	Unknown	NA	10	193	A022700074	1.93		
	C91378F	Unknown	NA	10	117	A022700075	1.17		39.5
	C91380F	Unknown	NA	10	89.1	A022700076	0.891	1.30	0.512
Group 6	C91047M	Unknown	NA	1	0.00	A062000022	<LOQ (0.00493 ug/mL)		
Mid-High Dose	C91054M	Unknown	NA	1	0.00	A062000023	<LOQ (0.00493 ug/mL)		
Recovery	C91056M	Unknown	NA	1	0.00	A062000026	<LOQ (0.00493 ug/mL)		
100 mg/kg	C91060M	Unknown	NA	1	0.00	A062000027	<LOQ (0.00493 ug/mL)		
	C91065M	Unknown	NA	1	0.00	A062000028	<LOQ (0.00493 ug/mL)		
	C91070M	Unknown	NA	1	0.00	A062000029	<LOQ (0.00493 ug/mL)		
	C91071M	Unknown	NA	1	0.00	A062000030	<LOQ (0.00493 ug/mL)		
	C91072M	Unknown	NA	1	0.00	A062000033	<LOQ (0.00493 ug/mL)		
	C91073M	Unknown	NA	1	0.00	A062000034	<LOQ (0.00493 ug/mL)		NA
	C91076M	Unknown	NA	1	0.00	A062000035	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 6	C91451F	Unknown	NA	1	2.07	A062000036	<LOQ (0.00493 ug/mL)		
Mid-High Dose	C91453F	Unknown	NA	1	0.00	A062000037	<LOQ (0.00493 ug/mL)		
Recovery	C91455F	Unknown	NA	1	0.00	A062000040	<LOQ (0.00493 ug/mL)		
100 mg/kg	C91459F	Unknown	NA	1	0.00	A062000041	<LOQ (0.00493 ug/mL)		
	C91462F	Unknown	NA	1	0.00	A062000042	<LOQ (0.00493 ug/mL)		
	C91467F	Unknown	NA	1	0.00	A062000043	<LOQ (0.00493 ug/mL)		
	C91473F	Unknown	NA	1	0.00	A062000044	<LOQ (0.00493 ug/mL)		
	C91479F	Unknown	NA	1	0.00	A062000047	<LOQ (0.00493 ug/mL)		
	C91480F	Unknown	NA	1	0.00	A062000048	<LOQ (0.00493 ug/mL)		
	C91485F	Unknown	NA	1	0.00	A062000049	<LOQ (0.00493 ug/mL)		
	C91487F	Unknown	NA	1	0.00	A062000050	<LOQ (0.00493 ug/mL)		NA
	C91488F	Unknown	NA	1	0.00	A062000051	<LOQ (0.00493 ug/mL)	<LOQ	NA

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PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.0247 ug/mL) to include std correction factors
new LOQ is 0.0198 ug/mL. LAC 02/19/01
A - data is above the upper limit of quantitation and is provided as an estimate.

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/15/00, 5/16/00, 06/13/00, 6/15/00, 6/19/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	04/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamide Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Soup 020199, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 2/28/00, 03/27/00, 05/31/00, 06/19/00, 06/20/00, 08/03/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 3/02/00, 06/02/00, 06/20/00, 06/21/00, 08/07/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot T-7121.1

Group Dose	Sample #	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Dilution Factor	PFOSAA Conc. ug/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Average PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 4	C90904M	Unknown	2nd Analysis OK	50	132	D081600055	6.58		
Mid-High Dose	C90908M	Unknown	NA	50	154	A022800018	7.68		
100 mg/kg	C90910M	Unknown	NA	50	671	A022800019	33.6		
	C90911M	Unknown	NA	50	475	A022700038	23.7		
	C90916M	Unknown	2nd Analysis OK	50	177	D081600058	8.84		
	C90926M	Unknown	NA	10	119	D081600038	1.19		
	C90927M	Unknown	NA	50	193	A022800021	9.64		
	C90928M	Unknown	NA	50	329	A022800024	16.5		
	C90933M	Unknown	NA	50	134	A022800025	6.69		
	C90944M	Unknown	NA	50	279	A022700040	14.0		
	C90947M	Unknown	2nd Analysis OK	50	174	D081600059	8.71		
	C90948M	Unknown	2nd Analysis OK	50	84	D081600060	4.19		
	C90953M	Unknown	NA	50	112	A022800026	5.58		
	C90955M	Unknown	NA	50	207	A022800027	10.3		
	C90956M	Unknown	2nd Analysis OK	50	71.0	D081600061	3.55		
	C90965M	Unknown	2nd Analysis OK	50	251	D081600062	12.6		74.1
	C90968M	Unknown	NA	50	173	A022800028	8.63	10.7	7.93
Group 4	C91315F	Unknown	NA	20	510	D062000061	10.2		
Mid-High Dose	C91324F	Unknown	NA	20	570	D062000062	11.4		
100 mg/kg	C91327F	Unknown	NA	20	941	D062000065	18.8		
	C91332F	Unknown	NA	20	922	D062000066	18.4		
	C91335F	Unknown	NA	20	498	D062000067	10.0		
	C91336F	Unknown	NA	20	559	D062000068	11.2		
	C91338F	Unknown	NA	20	529	D062000069	10.6		
	C91340F	Unknown	NA	20	553	D062000072	11.1		
	C91341F	Unknown	NA	20	555	D062000073	11.1		
	C91342F	Unknown	NA	20	596	D062000074	11.9		
	C91356F	Unknown	NA	10	602	A05310029	6.02		
	C91357F	Unknown	NA	20	562	D062000076	11.2		
	C91360F	Unknown	NA	20	688	D062000079	13.8		
	C91361F	Unknown	NA	10	776	A05310032	7.76		
	C91362F	Unknown	NA	20	624	D062000081	12.5		
	C91372F	Unknown	NA	20	670	D062000082	13.4		
	C91376F	Unknown	NA	20	648	D062000083	13.0		
	C91378F	Unknown	NA	20	515	D062000086	10.3		25.4
	C91380F	Unknown	NA	20	646	D062000087	12.9	11.9	3.02
Group 6	C91047M	Unknown	NA	1	0.00	A062000022	<LOQ (0.0248 ug/mL)		
Mid-High Dose	C91054M	Unknown	NA	1	0.00	A062000023	<LOQ (0.0248 ug/mL)		
Recovery	C91056M	Unknown	NA	1	0.00	A062100026	<LOQ (0.0248 ug/mL)		
100 mg/kg	C91060M	Unknown	NA	1	0.00	A062100027	<LOQ (0.0248 ug/mL)		
	C91065M	Unknown	NA	1	0.00	A062100028	<LOQ (0.0248 ug/mL)		
	C91070M	Unknown	NA	1	0.00	A062100029	<LOQ (0.0248 ug/mL)		
	C91071M	Unknown	NA	1	0.00	A062100030	<LOQ (0.0248 ug/mL)		
	C91072M	Unknown	NA	1	0.00	A062100033	<LOQ (0.0248 ug/mL)		
	C91073M	Unknown	NA	1	0.00	A062100034	<LOQ (0.0248 ug/mL)		NA
	C91076M	Unknown	NA	1	0.00	A062100035	<LOQ (0.0248 ug/mL)	<LOQ	NA
Group 6	C91451F	Unknown	NA	1	26.1	A062100036	0.0261		
Mid-High Dose	C91453F	Unknown	NA	1	0.00	A062100037	<LOQ (0.0248 ug/mL)		
Recovery	C91455F	Unknown	NA	1	0.00	A062100040	<LOQ (0.0248 ug/mL)		
100 mg/kg	C91459F	Unknown	NA	1	0.00	A062100041	<LOQ (0.0248 ug/mL)		
	C91462F	Unknown	NA	1	0.00	A062100042	<LOQ (0.0248 ug/mL)		
	C91467F	Unknown	NA	1	0.00	A062100043	<LOQ (0.0248 ug/mL)		
	C91473F	Unknown	NA	1	0.00	A062100044	<LOQ (0.0248 ug/mL)		
	C91479F	Unknown	NA	1	0.00	A062100047	<LOQ (0.0248 ug/mL)		
	C91480F	Unknown	NA	1	0.00	A062100048	<LOQ (0.0248 ug/mL)		
	C91485F	Unknown	NA	1	0.00	A062100049	<LOQ (0.0248 ug/mL)		
	C91487F	Unknown	NA	1	0.00	A062100050	<LOQ (0.0248 ug/mL)		NA
	C91488F	Unknown	NA	1	0.00	A062100051	<LOQ (0.0248 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.0247 ug/mL) to include std correction factors
new LOQ is 0.0198 ug/mL. LAC 02/19/01
A - data is above the upper limit of quantitation and is provided as an estimate.

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/15/00, 5/16/00, 06/13/00, 6/15/00, 6/19/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	04/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Soup 020199, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 2/28/00, 03/27/00, 05/31/00, 06/19/00, 06/20/00, 08/03/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 3/02/00, 06/02/00, 06/20/00, 06/21/00, 08/07/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot Unknown/SD013

Group Dose	Sample #	EtFOSE-OH Purity Correction Factor	Surrogate Verified	EtFOSE-OH Dilution Factor	EtFOSE-OH Conc. ng/mL	Filename	Concentration of EtFOSE-OH ug/mL or % Rec	Average EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD
Group 4	C90904M	Unknown	NA	1	8.52	A080300017	<LOQ (0.00977 ug/mL)		
Mid-High Dose	C90908M	Unknown	NA	1	12.6	A080300018	0.0126		
100 mg/kg	C90910M	Unknown	NA	1	81.6	A080300019	0.0816		
	C90911M	Unknown	NA	1	4.19	A080300020	<LOQ (0.00977 ug/mL)		
	C90916M	Unknown	2nd Analysis OK	1	22.1	D081600053	0.0221		
	C90926M	Unknown	NA	1	0.440	A080300024	<LOQ (0.00977 ug/mL)		
	C90927M	Unknown	NA	1	16.4	A080300025	0.0164		
	C90928M	Unknown	2nd Analysis OK	1	22.7	D081600054	0.0227		
	C90933M	Unknown	NA	1	11.2	A080300027	0.0112		
	C90944M	Unknown	NA	1	11.6	A080300028	0.0116		
	C90947M	Unknown	NA	1	7.73	A080300031	<LOQ (0.00977 ug/mL)		
	C90948M	Unknown	NA	1	6.26	A080300032	<LOQ (0.00977 ug/mL)		
	C90953M	Unknown	NA	1	21.7	A080300033	0.0217		
	C90955M	Unknown	NA	1	15.4	A080300034	0.0154		
	C90956M	Unknown	NA	1	1.28	A080300035	<LOQ (0.00977 ug/mL)		
	C90965M	Unknown	NA	1	19.7	A080300038	0.0197		93.3
	C90968M	Unknown	NA	1	16.4	A080300039	0.0164	0.0182	0.0170
Group 4	C91315F	Unknown	2nd Analysis OK	1	67.2	D081600047	0.0672		
Mid-High Dose	C91324F	Unknown	NA	1	34.4	A080300041	0.0344		
100 mg/kg	C91327F	Unknown	NA	1	29.0	A080300042	0.0290		
	C91332F	Unknown	NA	1	31.2	A080300045	0.0312		
	C91335F	Unknown	NA	1	56.2	A080300046	0.0562		
	C91336F	Unknown	NA	1	30.0	A080300047	0.0300		
	C91338F	Unknown	NA	1	27.4	A080300048	0.0274		
	C91340F	Unknown	NA	1	10.1	A080300049	0.0101		
	C91341F	Unknown	NA	1	43.6	A080300052	0.0436		
	C91342F	Unknown	NA	1	33.7	A080300053	0.0337		
	C91356F	Unknown	NA	1	17.8	A080300054	0.0178		
	C91357F	Unknown	NA	1	33.5	A080300055	0.0335		
	C91360F	Unknown	NA	1	57.3	A080300056	0.0573		
	C91361F	Unknown	2nd Analysis OK	1	45.9	D081600048	0.0459*		
	C91362F	Unknown	NA	1	17.0	A080300060	0.0170		
	C91372F	Unknown	2nd Analysis OK	1	46.7	D081600051	0.0467		
	C91376F	Unknown	NA	1	40.8	A080300062	0.0408		
	C91378F	Unknown	2nd Analysis OK	1	42.4	D081600052	0.0424		40.0
	C91380F	Unknown	NA	1	27.0	A080300066	0.0270	0.0364	0.0145
Group 6	C91047M	Unknown	NA	1	0.00	A062100022	<LOQ (0.00977 ug/mL)		
Mid-High Dose	C91054M	Unknown	NA	1	0.00	A062100023	<LOQ (0.00977 ug/mL)		
Recovery	C91056M	Unknown	NA	1	0.00	A062100026	<LOQ (0.00977 ug/mL)		
100 mg/kg	C91060M	Unknown	NA	1	0.00	A062100027	<LOQ (0.00977 ug/mL)		
	C91065M	Unknown	NA	1	0.00	A062100028	<LOQ (0.00977 ug/mL)		
	C91070M	Unknown	NA	1	0.00	A062100029	<LOQ (0.00977 ug/mL)		
	C91071M	Unknown	NA	1	0.00	A062100030	<LOQ (0.00977 ug/mL)		
	C91072M	Unknown	NA	1	0.00	A062100033	<LOQ (0.00977 ug/mL)		
	C91073M	Unknown	NA	1	0.00	A062100034	<LOQ (0.00977 ug/mL)		NA
	C91076M	Unknown	NA	1	0.00	A062100035	<LOQ (0.00977 ug/mL)	<LOQ	NA
Group 6	C91451F	Unknown	NA	1	0.0800	A062100036	<LOQ (0.00977 ug/mL)		
Mid-High Dose	C91453F	Unknown	NA	1	0.00	A062100037	<LOQ (0.00977 ug/mL)		
Recovery	C91455F	Unknown	NA	1	0.00	A062100040	<LOQ (0.00977 ug/mL)		
100 mg/kg	C91459F	Unknown	NA	1	0.00	A062100041	<LOQ (0.00977 ug/mL)		
	C91462F	Unknown	NA	1	0.00	A062100042	<LOQ (0.00977 ug/mL)		
	C91467F	Unknown	NA	1	0.00	A062100043	<LOQ (0.00977 ug/mL)		
	C91473F	Unknown	NA	1	0.00	A062100044	<LOQ (0.00977 ug/mL)		
	C91479F	Unknown	NA	1	0.00	A062100047	<LOQ (0.00977 ug/mL)		
	C91480F	Unknown	NA	1	0.00	A062100048	<LOQ (0.00977 ug/mL)		
	C91485F	Unknown	NA	1	0.00	A062100049	<LOQ (0.00977 ug/mL)		
	C91487F	Unknown	NA	1	0.00	A062100050	<LOQ (0.00977 ug/mL)		NA
	C91488F	Unknown	NA	1	0.00	A062100051	<LOQ (0.00977 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.0247 ug/mL) to include std correction factors
new LOQ is 0.0198 ug/mL. LAC 02/19/01
A - data is above the upper limit of quantitation and is provided as an estimate.

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/15/00, 5/16/00, 06/13/00, 6/15/00, 6/19/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	04/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Soup 020199, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 2/28/00, 03/27/00, 05/31/00, 06/19/00, 06/20/00, 08/03/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 3/02/00, 06/02/00, 06/20/00, 06/21/00, 08/07/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot NB113047-80

Group Dose	Sample #	M556 Purity Correction Factor	Surrogate Verified	M556 Dilution Factor	M556 Conc. ng/mL	Filename	Concentration of M556 ug/mL or % Rec	Average M556 ug/mL	RSD Std. Dev. MS/MSD RPD
Group 4	C90904M	Unknown	NA	10	646	A022700090	6.46		
Mid-High Dose	C90908M	Unknown	NA	10	766	A022700091	7.66		
100 mg/kg	C90910M	Unknown	NA	50	295	A022800019	14.8		
	C90911M	Unknown	NA	10	610	A022700054	6.10		
	C90916M	Unknown	NA	10	599	A022700055	5.99		
	C90926M	Unknown	NA	10	641	A022700095	6.41		
	C90927M	Unknown	NA	10	824	A022700096	8.24		
	C90928M	Unknown	NA	10	694	A022700097	6.94		
	C90933M	Unknown	NA	10	899	A022700098	8.99		
	C90944M	Unknown	NA	10	410	A022700056	4.10		
	C90947M	Unknown	NA	10	416	A022700059	4.16		
	C90948M	Unknown	NA	10	538	A022700060	5.38		
	C90953M	Unknown	NA	10	401	A022700101	4.01		
	C90955M	Unknown	NA	10	871	A022700102	8.71		
	C90956M	Unknown	NA	10	345	A022700061	3.45		
	C90965M	Unknown	NA	10	421	A022700062	4.21		41.2
	C90968M	Unknown	NA	10	627	A022700103	6.27	6.58	2.71
Group 4	C91315F	Unknown	NA	10	635	A022700077	6.35		
Mid-High Dose	C91324F	Unknown	NA	10	421	A022700080	4.21		
100 mg/kg	C91327F	Unknown	NA	10	320	A022700081	3.20		
	C91332F	Unknown	NA	20	288	A022800048	5.76		
	C91335F	Unknown	NA	10	570	A022700083	5.70		
	C91336F	Unknown	NA	10	753	A022700084	7.53		
	C91338F	Unknown	NA	10	608	A022700087	6.08		
	C91340F	Unknown	NA	10	388	A022700088	3.88		
	C91341F	Unknown	NA	10	505	A022700089	5.05		
	C91342F	Unknown	NA	10	618	A022700063	6.18		
	C91356F	Unknown	NA	10	210	A022700066	2.10		
	C91357F	Unknown	NA	10	625	A022700067	6.25		
	C91360F	Unknown	NA	10	460	A022700068	4.60		
	C91361F	Unknown	NA	10	305	A022700069	3.05		
	C91362F	Unknown	NA	10	512	A022700070	5.12		
	C91372F	Unknown	NA	10	606	A022700073	6.06		
	C91376F	Unknown	NA	10	620	A022700074	6.20		
	C91378F	Unknown	NA	10	376	A022700075	3.76		27.5
	C91380F	Unknown	NA	10	512	A022700076	5.12	5.06	1.39
Group 6	C91047M	Unknown	NA	1	0.0600	A061900022	<LOQ (0.00494 ug/mL)		
Mid-High Dose	C91054M	Unknown	NA	1	0.00	A061900023	<LOQ (0.00494 ug/mL)		
Recovery	C91056M	Unknown	NA	1	0.00	A061900026	<LOQ (0.00494 ug/mL)		
100 mg/kg	C91060M	Unknown	NA	1	0.270	A061900027	<LOQ (0.00494 ug/mL)		
	C91065M	Unknown	NA	1	0.00	A061900028	<LOQ (0.00494 ug/mL)		
	C91070M	Unknown	NA	1	0.00	A061900029	<LOQ (0.00494 ug/mL)		
	C91071M	Unknown	NA	1	0.00	A061900030	<LOQ (0.00494 ug/mL)		
	C91072M	Unknown	NA	1	4.41	A061900033	<LOQ (0.00494 ug/mL)		
	C91073M	Unknown	NA	1	0.00	A061900034	<LOQ (0.00494 ug/mL)		NA
	C91076M	Unknown	NA	1	0.00	A061900035	<LOQ (0.00494 ug/mL)	<LOQ	NA
Group 6	C91451F	Unknown	NA	1	55.3	A061900036	<LOQ (0.00494 ug/mL)		
Mid-High Dose	C91453F	Unknown	NA	1	1.97	A061900037	<LOQ (0.00494 ug/mL)		
Recovery	C91455F	Unknown	NA	1	0.350	A061900040	<LOQ (0.00494 ug/mL)		
100 mg/kg	C91459F	Unknown	NA	1	0.00	A061900041	<LOQ (0.00494 ug/mL)		
	C91462F	Unknown	NA	1	1.87	A061900042	<LOQ (0.00494 ug/mL)		
	C91467F	Unknown	NA	1	0.00	A061900043	<LOQ (0.00494 ug/mL)		
	C91473F	Unknown	NA	1	0.350	A061900044	<LOQ (0.00494 ug/mL)		
	C91479F	Unknown	NA	1	0.00	A061900047	<LOQ (0.00494 ug/mL)		
	C91480F	Unknown	NA	1	0.00	A061900048	<LOQ (0.00494 ug/mL)		
	C91485F	Unknown	NA	1	2.75	A061900049	<LOQ (0.00494 ug/mL)		
	C91487F	Unknown	NA	1	0.00	A061900050	<LOQ (0.00494 ug/mL)		NA
	C91488F	Unknown	NA	1	0.00	A061900051	<LOQ (0.00494 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.0247 ug/mL) to include std correction factors
new LOQ is 0.0198 ug/mL. LAC 02/19/01
A - data is above the upper limit of quantitation and is provided as an estimate.

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/15/00, 5/16/00, 06/13/00, 6/15/00, 6/19/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	04/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Soup 020199, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 2/28/00, 03/27/00, 05/31/00, 06/19/00, 06/20/00, 08/03/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 3/02/00, 06/02/00, 06/20/00, 06/21/00, 08/07/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009
WEEK 105 RAT SERA	lot 529

Group Dose	Sample #	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Dilution Factor	PFOSEA Conc. ng/mL	Filename	Concentration of PFOSEA ug/mL or % Rec	Average PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 4	C90904M	Unknown	NA	1	0.00	A080300017	<LOQ (0.00492 ug/mL)		
Mid-High Dose	C90908M	Unknown	NA	1	0.00	A080300018	<LOQ (0.00492 ug/mL)		
100 mg/kg	C90910M	Unknown	NA	1	0.00	A080300019	<LOQ (0.00492 ug/mL)		
	C90911M	Unknown	NA	1	2.76	A080300020	<LOQ (0.00492 ug/mL)		
	C90916M	Unknown	2nd Analysis OK	1	0.00	D081600053	<LOQ (0.00492 ug/mL)		
	C90926M	Unknown	NA	1	0.00	A080300024	<LOQ (0.00492 ug/mL)		
	C90927M	Unknown	NA	1	0.00	A080300025	<LOQ (0.00492 ug/mL)		
	C90928M	Unknown	2nd Analysis OK	1	0.450	D081600054	<LOQ (0.00492 ug/mL)		
	C90933M	Unknown	NA	1	0.00	A080300027	<LOQ (0.00492 ug/mL)		
	C90944M	Unknown	NA	1	0.00	A080300028	<LOQ (0.00492 ug/mL)		
	C90947M	Unknown	NA	1	0.00	A080300031	<LOQ (0.00492 ug/mL)		
	C90948M	Unknown	NA	1	0.00	A080300032	<LOQ (0.00492 ug/mL)		
	C90953M	Unknown	NA	1	0.00	A080300033	<LOQ (0.00492 ug/mL)		
	C90955M	Unknown	NA	1	0.00	A080300034	<LOQ (0.00492 ug/mL)		
	C90956M	Unknown	NA	1	0.00	A080300035	<LOQ (0.00492 ug/mL)		
	C90965M	Unknown	NA	1	3.75	A080300038	<LOQ (0.00492 ug/mL)		NA
	C90968M	Unknown	NA	1	0.00	A080300039	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 4	C91315F	Unknown	2nd Analysis OK	1	0.00	D081600047	<LOQ (0.00492 ug/mL)		
Mid-High Dose	C91324F	Unknown	NA	1	4.02	A080300041	<LOQ (0.00492 ug/mL)		
100 mg/kg	C91327F	Unknown	NA	1	6.88	A080300042	0.00688		
	C91332F	Unknown	NA	1	0.00	A080300045	<LOQ (0.00492 ug/mL)		
	C91335F	Unknown	NA	1	1.85	A080300046	<LOQ (0.00492 ug/mL)		
	C91336F	Unknown	NA	1	0.320	A080300047	<LOQ (0.00492 ug/mL)		
	C91338F	Unknown	NA	1	0.00	A080300048	<LOQ (0.00492 ug/mL)		
	C91340F	Unknown	NA	1	0.00	A080300049	<LOQ (0.00492 ug/mL)		
	C91341F	Unknown	NA	1	0.00	A080300052	<LOQ (0.00492 ug/mL)		
	C91342F	Unknown	NA	1	0.00	A080300053	<LOQ (0.00492 ug/mL)		
	C91356F	Unknown	NA	1	1.72	A080300054	<LOQ (0.00492 ug/mL)		
	C91357F	Unknown	NA	1	0.00	A080300055	<LOQ (0.00492 ug/mL)		
	C91360F	Unknown	NA	1	2.40	A080300056	<LOQ (0.00492 ug/mL)		
	C91361F	Unknown	2nd Analysis OK	1	1.61	D081600048	<LOQ (0.00492 ug/mL)		
	C91362F	Unknown	NA	1	0.00	A080300060	<LOQ (0.00492 ug/mL)		
	C91372F	Unknown	2nd Analysis OK	1	0.610	D081600051	<LOQ (0.00492 ug/mL)		
	C91376F	Unknown	NA	1	6.20	A080300062	0.00620		
	C91378F	Unknown	2nd Analysis OK	1	1.04	D081600052	<LOQ (0.00492 ug/mL)		NA
	C91380F	Unknown	NA	1	0.00	A080300066	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 6	C91047M	Unknown	NA	1	0.00	A061900022	<LOQ (0.00492 ug/mL)		
Mid-High Dose	C91054M	Unknown	NA	1	0.00	A061900023	<LOQ (0.00492 ug/mL)		
Recovery	C91056M	Unknown	NA	1	0.00	A061900026	<LOQ (0.00492 ug/mL)		
100 mg/kg	C91060M	Unknown	NA	1	0.00	A061900027	<LOQ (0.00492 ug/mL)		
	C91065M	Unknown	NA	1	0.00	A061900028	<LOQ (0.00492 ug/mL)		
	C91070M	Unknown	NA	1	0.600	A061900029	<LOQ (0.00492 ug/mL)		
	C91071M	Unknown	NA	1	0.00	A061900030	<LOQ (0.00492 ug/mL)		
	C91072M	Unknown	NA	1	0.00	A061900033	<LOQ (0.00492 ug/mL)		
	C91073M	Unknown	NA	1	0.00	A061900034	<LOQ (0.00492 ug/mL)		NA
	C91076M	Unknown	NA	1	0.00	A061900035	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 6	C91451F	Unknown	NA	1	0.00	A061900036	<LOQ (0.00492 ug/mL)		
Mid-High Dose	C91453F	Unknown	NA	1	0.00	A061900037	<LOQ (0.00492 ug/mL)		
Recovery	C91455F	Unknown	NA	1	0.00	A061900040	<LOQ (0.00492 ug/mL)		
100 mg/kg	C91459F	Unknown	NA	1	0.00	A061900041	<LOQ (0.00492 ug/mL)		
	C91462F	Unknown	NA	1	0.00	A061900042	<LOQ (0.00492 ug/mL)		
	C91467F	Unknown	NA	1	0.00	A061900043	<LOQ (0.00492 ug/mL)		
	C91473F	Unknown	NA	1	0.00	A061900044	<LOQ (0.00492 ug/mL)		
	C91479F	Unknown	NA	1	0.00	A061900047	<LOQ (0.00492 ug/mL)		
	C91480F	Unknown	NA	1	0.00	A061900048	<LOQ (0.00492 ug/mL)		
	C91485F	Unknown	NA	1	0.00	A061900049	<LOQ (0.00492 ug/mL)		
	C91487F	Unknown	NA	1	0.00	A061900050	<LOQ (0.00492 ug/mL)		NA
	C91488F	Unknown	NA	1	0.00	A061900051	<LOQ (0.00492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Corrected PFOS LOQ (0.0247 ug/mL) to include std correction factors
new LOQ is 0.0198 ug/mL. LAC 02/19/01
A - data is above the upper limit of quantitation and is provided as an estimate.

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/15/00, 5/16/00, 06/13/00, 6/15/00, 6/19/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	04/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Soup 020199, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 2/28/00, 03/27/00, 05/31/00, 06/19/00, 06/20/00, 08/03/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 3/02/00, 06/02/00, 06/20/00, 06/21/00, 08/07/00, 08/18/00, 08/25/00 MMH/IAS/HOJ

Sample Data

WEEK 105 RAT SERA

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Average PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ug/mL or % Rec	Average PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSAA ug/mL or % Rec	Average PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 4	C90904M	66.9			0.568			6.58		
Mid-High Dose	C90908M	71.0			0.606			7.68		
100 mg/kg	C90910M	31.5			1.84			33.6		
	C90911M	146			0.607			23.7		
	C90916M	88.4			0.612			8.8		
	C90926M	61.61			0.330			1.19		
	C90927M	22.0			1.09			9.64		
	C90928M	127			0.979			16.5		
	C90933M	45.8			0.593			6.69		
	C90944M	136			0.560			14.0		
	C90947M	144			0.685			8.7		
	C90948M	51.7			0.277			4.19		
	C90953M	13.0			0.572			5.58		
	C90955M	64.1			0.614			10.3		
	C90956M	132			0.390			3.55		
	C90965M	137		53.6	0.949		51.9	12.6		74.1
	C90968M	100	84.6	45.4	0.638	0.701	0.363	8.63	10.7	7.93
Group 4	C91315F	122			0.898			10.2		
Mid-High Dose	C91324F	133			1.91			11.4		
100 mg/kg	C91327F	9.81			1.02			18.8		
	C91332F	251 A			1.36			18.4		
	C91335F	99.5			2.05			10.0		
	C91336F	202 A			1.07			11.2		
	C91338F	140			1.71			10.6		
	C91340F	252 A			0.721			11.1		
	C91341F	76.0			1.38			11.1		
	C91342F	122			0.826			11.9		
	C91356F	6.18			0.345			6.02		
	C91357F	200 A			2.01			11.2		
	C91360F	141			1.85			13.8		
	C91361F	131			1.16			7.76		
	C91362F	25.3			0.720			12.5		
	C91372F	58.8			1.61			13.4		
	C91376F	133			1.93			13.0		
	C91378F	109		57.9	1.17		39.5	10.3		25.4
	C91380F	104	122	70.5	0.891	1.30	0.512	12.9	11.9	3.02
Group 6	C91047M	0.185			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
Mid-High Dose	C91054M	0.0222			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
Recovery	C91056M	1.18			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
100 mg/kg	C91060M	0.142			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91065M	<LOQ (0.0198 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91070M	0.0514			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91071M	<LOQ (0.0198 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91072M	0.0454			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91073M	<LOQ (0.0198 ug/mL)		166	<LOQ (0.00493 ug/mL)		NA	<LOQ (0.0248 ug/mL)		NA
	C91076M	0.592	0.227	0.377	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA
Group 6	C91451F	5.02			<LOQ (0.00493 ug/mL)			0.03		
Mid-High Dose	C91453F	1.68			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
Recovery	C91455F	5.92			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
100 mg/kg	C91459F	<LOQ (0.0198 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91462F	4.61			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91467F	0.0207			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91473F	3.84			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91479F	<LOQ (0.0198 ug/mL)			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91480F	0.0553			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91485F	3.55			<LOQ (0.00493 ug/mL)			<LOQ (0.0248 ug/mL)		
	C91487F	<LOQ (0.0198 ug/mL)		96.3	<LOQ (0.00493 ug/mL)		NA	<LOQ (0.0248 ug/mL)		NA
	C91488F	4.16	2.41	2.32	<LOQ (0.00493 ug/mL)	<LOQ	NA	<LOQ (0.0248 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuoroocctane sulfonyl ethylamide

Corrected PFOS LOQ (0.0247 ug/mL) to include std correction factors
new LOQ is 0.0198 ug/mL. LAC 02/19/01
A - data is above the upper limit of quantitation and is provided as an estimate.

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/15/00, 5/16/00, 06/13/00, 6/15/00, 6/19/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	04/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Soup 020199, Davey 070799
Instrument Software/Version:	Masslynx 3.3 & 3.4
Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	02/07/00, 02/08/00, 02/09/00 RWW, SAL
Dates of Analysis/Analyst:	02/18/00, 2/28/00, 03/27/00, 05/31/00, 06/19/00, 06/20/00, 08/03/00, 08/16/00, 08/23/00 MMH/IAS
Date of Data Reduction/Analyst:	02/21/00, 02/29/00, 3/02/00, 06/02/00, 06/20/00, 06/21/00, 08/07/00, 08/18/00, 08/25/00 MMH/IAS/HOJ
Sample Data	Box 00-009

WEEK 105 RAT SERA

Group Dose	Sample #	Concentration of EtFOSE-OH ug/mL or % Rec	Average EtFOSE-OH ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of M556 ug/mL or % Rec	Average M556 ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSEA ug/mL or % Rec	Average PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Group 4	C90904M	<LOQ (0.00977 ug/mL)			6.46			<LOQ (0.00492 ug/mL)		
Mid-High Dose	C90908M	0.0126			7.66			<LOQ (0.00492 ug/mL)		
100 mg/kg	C90910M	0.0816			14.8			<LOQ (0.00492 ug/mL)		
	C90911M	<LOQ (0.00977 ug/mL)			6.10			<LOQ (0.00492 ug/mL)		
	C90916M	0.0221			5.99			<LOQ (0.00492 ug/mL)		
	C90926M	<LOQ (0.00977 ug/mL)			6.41			<LOQ (0.00492 ug/mL)		
	C90927M	0.0164			8.24			<LOQ (0.00492 ug/mL)		
	C90928M	0.0227			6.94			<LOQ (0.00492 ug/mL)		
	C90933M	0.0112			8.99			<LOQ (0.00492 ug/mL)		
	C90944M	0.0116			4.10			<LOQ (0.00492 ug/mL)		
	C90947M	<LOQ (0.00977 ug/mL)			4.16			<LOQ (0.00492 ug/mL)		
	C90948M	<LOQ (0.00977 ug/mL)			5.38			<LOQ (0.00492 ug/mL)		
	C90953M	0.0217			4.01			<LOQ (0.00492 ug/mL)		
	C90955M	0.0154			8.71			<LOQ (0.00492 ug/mL)		
	C90956M	<LOQ (0.00977 ug/mL)			3.45			<LOQ (0.00492 ug/mL)		
	C90965M	0.0197		93.3	4.21		41.18	<LOQ (0.00492 ug/mL)		NA
	C90968M	0.0164	0.0182	0.0170	6.27	6.58	2.71	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 4	C91315F	0.0672			6.35			<LOQ (0.00492 ug/mL)		
Mid-High Dose	C91324F	0.0344			4.21			<LOQ (0.00492 ug/mL)		
100 mg/kg	C91327F	0.0290			3.20			0.00688		
	C91332F	0.0312			5.76			<LOQ (0.00492 ug/mL)		
	C91335F	0.0562			5.70			<LOQ (0.00492 ug/mL)		
	C91336F	0.0300			7.53			<LOQ (0.00492 ug/mL)		
	C91338F	0.0274			6.08			<LOQ (0.00492 ug/mL)		
	C91340F	0.0101			3.88			<LOQ (0.00492 ug/mL)		
	C91341F	0.0436			5.05			<LOQ (0.00492 ug/mL)		
	C91342F	0.0337			6.18			<LOQ (0.00492 ug/mL)		
	C91356F	0.0178			2.10			<LOQ (0.00492 ug/mL)		
	C91357F	0.0335			6.25			<LOQ (0.00492 ug/mL)		
	C91360F	0.0573			4.60			<LOQ (0.00492 ug/mL)		
	C91361F	0.0459			3.05			<LOQ (0.00492 ug/mL)		
	C91362F	0.0170			5.12			<LOQ (0.00492 ug/mL)		
	C91372F	0.0467			6.06			<LOQ (0.00492 ug/mL)		
	C91376F	0.0408			6.20			0.00620		
	C91378F	0.0424		40.0	3.76		27.5	<LOQ (0.00492 ug/mL)		NA
	C91380F	0.0270	0.0364	0.0145	5.12	5.06	1.39	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 6	C91047M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
Mid-High Dose	C91054M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
Recovery	C91056M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
100 mg/kg	C91060M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91065M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91070M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91071M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91072M	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91073M	<LOQ (0.00977 ug/mL)		NA	<LOQ (0.00494 ug/mL)		NA	<LOQ (0.00492 ug/mL)		NA
	C91076M	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA
Group 6	C91451F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
Mid-High Dose	C91453F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
Recovery	C91455F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
100 mg/kg	C91459F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91462F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91467F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91473F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91479F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91480F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91485F	<LOQ (0.00977 ug/mL)			<LOQ (0.00494 ug/mL)			<LOQ (0.00492 ug/mL)		
	C91487F	<LOQ (0.00977 ug/mL)		NA	<LOQ (0.00494 ug/mL)		NA	<LOQ (0.00492 ug/mL)		NA
	C91488F	<LOQ (0.00977 ug/mL)	<LOQ	NA	<LOQ (0.00494 ug/mL)	<LOQ	NA	<LOQ (0.00492 ug/mL)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuoroocane sulfonyl ethylamide

Corrected PFOS LOQ (0.0247 ug/mL) to include std correction factors
new LOQ is 0.0198 ug/mL. LAC 02/19/01
A - data is above the upper limit of quantitation and is provided as an estimate.

Date Entered/By:	03/06/00, 03/10/00, 03/20/00, 03/21/00, 4/15/00, 5/16/00, 06/13/00, 6/15/00, 6/19/00, 06/22/00, 06/23/00, 06/26/00, 08/15/00, 09/05/00, 10/17/00 LAC/CSH
Date Verified/ By:	04/30/01 hoj / 04/30/01 LAC
Purity Entered/Verified:	02/19/01 LAC

Covance 6329-212

PFOS	PFOSA	PFOSAA	EtFOSE
06/03/98 Lot 193	06/03/98 Lot L-2353	06/03/98 Lot 617	06/03/98 Lot 936
06/05/98 Lot 193	06/05/98 Lot L-2353	06/05/98 Lot 617	06/05/98 Lot 936
06/12/98 Lot 193	06/12/98 Lot L-2353	06/12/98 Lot 617	06/12/98 Lot 936
06/14/98 Lot 193	07/09/98 Lot L-2353	07/09/98 Lot 617	07/09/98 Lot 936
	09/02/98 Lot L-2353	09/02/98 Lot 617	09/02/98 Lot 936

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Liver
Method/Revision:	FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0
Analytical Equipment System Number:	Chick 080697, Madeline 041098, and Amelia 062498
Instrument Software/Version:	MassLynx 2.3 and 3.1
Date of Extraction/Analyst:	05/29/98 and 06/01/98 RWW/OK/LAC
Date of Analysis/Analyst:	06/03/98, 06/05/98, 06/12/98, 06/14/98, 07/09/98, 09/02/98 HOJ/KJH/MEE
Date of Data Reduction/Analyst:	02/25/00, 03/14/00, 03/15/00, 03/16/00, 11/29/00, 12/10/00 MMH/IAS

Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments

Sample Data

WEEK 4 RAT LIVER REWORK

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Conc. ng/g	PFOS Dilution Factor	Filename (Lot 193)	PFOS Calc. Conc. ng/g	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor (Lot L-2353)	PFOSA Conc. ng/g	PFOSA Dilution Factor	Filename	PFOSA Calc. Conc. ng/g	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	1.0000	NA	0.9275	Unknown	12.4	1	60398014	11.5	<LOQ (0.0552 ug/g)			Unknown	0.00	1	60398014	0.00	<LOQ (0.0121 ug/g)		
	H2O Blk-2	1.0000	NA	0.9275	Unknown	0.00	1	61298056	0.00	<LOQ (0.0110 ug/g)	<LOQ	NA	Unknown	0.00	1	61298056	0.00	<LOQ (0.00604 ug/g)	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	1.0080	40.13	0.9275	Unknown	8.66	1	60398015	7.97	<LOQ (0.0552 ug/g)			Unknown	0.00	1	60398015	0.00	<LOQ (0.0121 ug/g)		
	Rabbit Liver Blk-2	1.0080	40.13	0.9275	Unknown	0.00	1	61298057	0.00	<LOQ (0.0110 ug/g)	<LOQ	NA	Unknown	0.00	1	61298057	0.00	<LOQ (0.00604 ug/g)	<LOQ	NA
QC - 100 ppb	C90719M-MS^	1.0248	NA	NA	NA	1043	1	60398042	233	196%			NA	130	1	60398042	121	100%		
	C90719M-MSD	1.0248	NA	NA	NA	1032	1	60398123	223	187%	192%	5%	NA	145	1	60398123	132	110%	105%	9%
Group 1	C90719M^	1.0248	NA	0.9275	Unknown	804	1	60398122	728	0.728			Unknown	9.19	1	60398122	8.97	0.00897		
Control	C90725M	1.0364	NA	0.9275	Unknown	723	1	60398043	647	0.647			Unknown	10.2	1	60398043	9.86	<LOQ (0.0121 ug/g)		
0.0 mg/kg	C90732M	1.0333	NA	0.9275	Unknown	1091	1	60398044	980	0.980			Unknown	18.4	1	60398044	17.8	0.0178		
	C90753M	0.9999	NA	0.9275	Unknown	723	1	60398045	670	0.670		17.8	Unknown	10.5	1	60398045	10.5	<LOQ (0.0121 ug/g)		49.9
	C90775M	1.0586	NA	0.9275	Unknown	963	1	60398046	844	0.844	0.774	0.138	Unknown	25.2	1	60398046	23.8	0.0238	0.0150	0.00746
	C91135F	1.0124	NA	0.9275	Unknown	221	1	60398050	203	0.203			Unknown	2.23	1	60398050	2.20	<LOQ (0.0121 ug/g)		
	C91143F	1.0380	NA	0.9275	Unknown	284	1	60398051	254	0.254			Unknown	23.0	1	60398051	22.1	0.0221		
	C91165F	1.0375	NA	0.9275	Unknown	631	1	60398052	564	0.564			Unknown	31.6	1	60398052	30.4	0.0304		
	C91169F	1.0477	NA	0.9275	Unknown	454	1	60398053	402	0.402		39.0	Unknown	14.4	1	60398053	13.8	0.0138		43.7
	C91171F	1.0064	NA	0.9275	Unknown	453	1	60398054	418	0.418	0.368	0.144	Unknown	13.6	1	60398054	13.5	0.0135	0.0184	0.00804
Group 2	C90789M	1.0591	NA	0.9275	Unknown	145	100	60598112	12658	12.7			Unknown	520	1	60598098	491	0.491		
Low Dose	C90814M	1.0572	NA	0.9275	Unknown	120	100	60598113	10487	10.5			Unknown	320	1	60598099	302	0.302		
3.0 mg/kg	C90817M	1.0460	NA	0.9275	Unknown	132	100	60598114	11723	11.7			Unknown	507	1	60598100	485	0.485		
	C90833M	1.0438	NA	0.9275	Unknown	169	100	60598115	15011	15.0		13.5	Unknown	900	1	60598101	863	0.863		37.8
	C90834M	1.0749	NA	0.9275	Unknown	140	100	60598116	12069	12.1	12.4	1.67	Unknown	721	1	60598102	670	0.670	0.562	0.212
	C91198F	1.0532	NA	0.9275	Unknown	125	100	60598119	11027	11.0			Unknown	670	1	60598105	636	0.636		
	C91205F	1.0651	NA	0.9275	Unknown	110	100	60598120	9588	9.59			Unknown	676	1	60598106	635	0.635		
	C91222F	1.0183	NA	0.9275	Unknown	146	100	60598121	13282	13.3			Unknown	663	1	60598107	651	0.651		
	C91230F	1.0433	NA	0.9275	Unknown	106	100	60598122	9412	9.41		17.1	Unknown	794	1	60598108	761	0.761		8.39
	C91237F	1.0467	NA	0.9275	Unknown	152	100	60598123	13426	13.4	11.3	1.94	Unknown	756	1	60598109	722	0.722	0.681	0.0572
Group 3	C90846M	1.0108	NA	0.9275	Unknown	141	500	61498027	64520	64.5			Unknown	80.5	100	M090298049	7961	7.96		
Mid Dose	C90853M	1.0440	NA	0.9275	Unknown	194	500	61498028	86362	86.4			Unknown	53.1	100	M090298050	5083	5.08		
30.0 mg/kg	C90866M	1.0389	NA	0.9275	Unknown	249	500	61498029	110931	111			Unknown	74.4	100	M090298051	7159	7.16		
	C90890M	1.0258	NA	0.9275	Unknown	243	500	61498030	109767	110		21.2	Unknown	74.6	100	M090298052	7276	7.28		15.9
	C90891M	1.0173	NA	0.9275	Unknown	239	500	61498031	108824	109	96.1	20.4	Unknown	67.1	100	M090298053	6598	6.60	6.82	1.08
	C91251F	1.0048	NA	0.9275	Unknown	215	500	61498034	99018	99.0			Unknown	59.6	100	M090298057	5934	5.93		
	C91272F	1.0564	NA	0.9275	Unknown	277	500	61498035	121385	121			Unknown	78.3	100	M090298058	7413	7.41		
	C91285F	1.0113	NA	0.9275	Unknown	200	500	61498036	91929	91.9			Unknown	48.2	100	M090298059	4761	4.76		
	C91287F	1.0695	NA	0.9275	Unknown	319	500	61498037	138167	138		21.0	Unknown	59.5	100	M090298060	5566	5.57		17.6
	C91291F	0.9961	NA	0.9275	Unknown	325	500	61498038	151495	151	120	25.3	Unknown	70.1	100	M090298061	7036	7.04	6.14	1.08
Group 4	C90935M	0.9608	NA	0.9275	Unknown	66.4	2000	M061298067	128159	128			Unknown	94.6	100	M07099813	9841	9.84		
Mid-High Dose	C90939M	1.0444	NA	0.9275	Unknown	94.2	2000	M061298068	167259	167			Unknown	69.3	100	M07099814	6639	6.64		
100 mg/kg	C90942M	1.0119	NA	0.9275	Unknown	111	2000	M061298069	202805	203			Unknown	88.3	100	M07099815	8722	8.72		
	C90961M	1.0621	NA	0.9275	Unknown	151	2000	M061298070	263867	264		26.8	Unknown	139	100	M07099816	13084	13.1		24.4
	C90966M	1.0166	NA	0.9275	Unknown	127	2000	M061298071	231647	232	199	53.2	Unknown	97.0	100	M07099817	9543	9.54	9.57	2.33
	C91316F	1.0324	NA	0.9275	Unknown	94.2	2000	M061298072	169257	169			Unknown	75.4	100	M07099820	7302	7.30		
	C91317F	0.9882	NA	0.9275	Unknown	223	2000	M061298074	419299	419			Unknown	107	100	M07099821	10851	10.9		
	C91333F	1.0133	NA	0.9275	Unknown	121	2000	M061298075	220960	221			Unknown	92.8	100	M07099822	9162	9.16		
	C91345F	1.0724	NA	0.9275	Unknown	141	2000	M061298076	243655	244		52.8	Unknown	95.3	100	M07099823	8889	8.89		26.5
	C91350F	1.0509	NA	0.9275	Unknown	52.5	2000	M061298077	92724	92.7	229	121	Unknown	53.0	100	M07099824	5043	5.04	8.25	2.19
Group 5	C90984M	1.0062	NA	0.9275	Unknown	41.6	5000	M061298078	191685	192			Unknown	1.37	1000	7099827	1362	1.36 *		
High Dose	C91005M	1.0225	NA	0.9275	Unknown	86.4	5000	M061298081	391682	392			Unknown	6.32	1000	7099828	6181	6.18 *		
300 mg/kg	C91024M	1.0121	NA	0.9275	Unknown	144	5000	M061298082	661557	662			Unknown	12.7	1000	7099829	12528	12.5 *		
	C91033M	1.0339	NA	0.9275	Unknown	154	5000	M061298083	691925	692		41.7	Unknown	13.6	1000	7099830	13154	13.2		56.5
	C91037M	1.0275	NA	0.9275	Unknown	124	5000	M061298084	559479	559	499	208	Unknown	11.8	1000	7099831	11465	11.5	8.94	5.05
	C91423F	1.0131	NA	0.9275	Unknown	122	5000	M061298085	557635	558			Unknown	15.7	1000	7099834	15517	15.5		
	C91427F	1.0217	NA	0.9275	Unknown	83.7	5000	M061298088	379688	380			Unknown	18.6	1000	7099835	18156	18.2		
	C91430F	1.0191	NA	0.9275	Unknown	107	5000	M061298089	488915	489			Unknown	16.5	1000	7099836	16152	16.2		
	C91443F	1.0106	NA	0.9275	Unknown	178	5000	M061298090	816358	816		30.1	Unknown	14.8	1000	7099837	14645	14.6		15.1
	C91448F	1.0130	NA	0.9275	Unknown	162	5000	M061298091	740764	741	597	180	Unknown	12.0	1000	7099838	11836	11.8	15.3	2.31

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
Date Entered/Analyst: 05/23/00, 06/20/00, 08/15/00 CSH/LAC 12/05/00 KJH
Date Verified/Analyst: 12/05/00 KJH, 12/06/00 HOJ
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0595 & 0.0119 ug/g) to include std correction factors
new LOQs are 0.0552 & 0.0110 ug/g. LAC 02/19/01

+ CCVs not within criteria due to carryover, estimated results.
* Below LOQ, tentative numbers.
++ Above the linear range of the curve, data are estimated.
^ Samples were mislabeled during the extraction or analysis. A deviation was written and sample data switched.

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Liver
Method/Revision:	FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0
Analytical Equipment System Number:	Chick 080697, Madeline 041098, and Amelia 062498
Instrument Software/Version:	MassLynx 2.3 and 3.1
Date of Extraction/Analyst:	05/29/98 and 06/01/98 RWW/OK/LAC
Date of Analysis/Analyst:	06/03/98, 06/05/98, 06/12/98, 06/14/98, 07/09/98, 09/02/98 HOJ/KJH/MEE
Date of Data Reduction/Analyst:	02/25/00, 03/14/00, 03/15/00, 03/16/00, 11/29/00, 12/10/00 MMH/IAS

Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments

Sample Data

WEEK 4 RAT LIVER REWORK

Group Dose	Sample #	PFOSAA Purity Correction Factor	PFOSAA Conc. ng/g	PFOSAA Dilution Factor	Filename (Lot 617)	PFOSAA Calc. Conc. ng/g	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD	EtFOSE Purity Correction Factor	EtFOSE Conc. ng/g	EtFOSE Dilution Factor	Filename (Lot 936)	EtFOSE Calc. Conc. ng/g	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	Unknown	3.85	1	60398014	3.85	<LOQ (0.0633 ug/g)			Unknown	32.5	1	60398014	32.5	<LOQ (0.0596 ug/g)		
	H2O Blk-2	Unknown	0.00	1	61298056	0.00	<LOQ (0.127 ug/g)	<LOQ	NA	Unknown	0.00	1	61298056	0.00	<LOQ (0.119 ug/g)	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	Unknown	0.00	1	60398015	0.00	<LOQ (0.0633 ug/g)			Unknown	0.00	1	60398015	0.00	<LOQ (0.0596 ug/g)		
	Rabbit Liver Blk-2	Unknown	0.00	1	61298057	0.00	<LOQ (0.127 ug/g)	<LOQ	NA	Unknown	0.00	1	61298057	0.00	<LOQ (0.119 ug/g)	<LOQ	NA
QC - 100 ppb	C90719M-MS^	NA	187	1	60398042	131	103%			NA	194	1	60398042	189	158%		
	C90719M-MSD	NA	191	1	60398123	134	106%	104%	3%	NA	133	1	60398123	129	109%	133%	37%
	C90719M^	Unknown	52.7	1	60398122	51.5	0.0515			Unknown	0.00	1	60398122	0.00	<LOQ (0.0596 ug/g)		
Control	C90725M	Unknown	109	1	60398043	105	0.105			Unknown	0.00	1	60398043	0.00	<LOQ (0.0596 ug/g)		
Group 1	C90732M	Unknown	147	1	60398044	143	0.143			Unknown	0.00	1	60398044	0.00	<LOQ (0.0596 ug/g)		
	C90753M	Unknown	151	1	60398045	151	0.151		46.1	Unknown	30.7	1	60398045	30.7	<LOQ (0.0596 ug/g)		NA
	C90775M	Unknown	232	1	60398046	219	0.219	0.134	0.0617	Unknown	0.00	1	60398046	0.00	<LOQ (0.0596 ug/g)	<LOQ	NA
	C91135F	Unknown	11.5	1	60398050	11.3	<LOQ (0.0633 ug/g)			Unknown	0.00	1	60398050	0.00	<LOQ (0.0596 ug/g)		
	C91143F	Unknown	80.9	1	60398051	77.9	0.0779			Unknown	0.00	1	60398051	0.00	<LOQ (0.0596 ug/g)		
	C91165F	Unknown	167	1	60398052	161	0.161			Unknown	0.00	1	60398052	0.00	<LOQ (0.0596 ug/g)		
	C91169F	Unknown	83.7	1	60398053	79.8	0.0798		39.2	Unknown	0.00	1	60398053	0.00	<LOQ (0.0596 ug/g)		NA
	C91171F	Unknown	116	1	60398054	115	0.115	0.0995	0.0390	Unknown	0.00	1	60398054	0.00	<LOQ (0.0596 ug/g)	<LOQ	NA
Group 2	C90789M	Unknown	221	10	60598140	2087	2.09			Unknown	23.5	1	60598098	22.2	<LOQ (0.119 ug/g)		
Low Dose	C90814M	Unknown	205	10	60598141	1940	1.94			Unknown	0.00	1	60598099	0.00	<LOQ (0.119 ug/g)		
3.0 mg/kg	C90817M	Unknown	193	10	60598142	1846	1.85			Unknown	0.00	1	60598100	0.00	<LOQ (0.119 ug/g)		
	C90833M	Unknown	203	50	60598129	9732	9.73		94.4	Unknown	0.00	1	60598101	0.00	<LOQ (0.119 ug/g)		NA
	C90834M	Unknown	272	10	60598144	2528	2.53	3.63	3.42	Unknown	0.00	1	60598102	0.00	<LOQ (0.119 ug/g)	<LOQ	NA
	C91198F	Unknown	809	10	60598147	7681	7.68			Unknown	0.00	1	60598105	0.00	<LOQ (0.119 ug/g)		
	C91205F	Unknown	293	10	60598148	2751	2.75			Unknown	0.00	1	60598106	0.00	<LOQ (0.119 ug/g)		
	C91222F	Unknown	396	10	60598149	3893	3.89			Unknown	69.2	1	60598107	68.0	<LOQ (0.119 ug/g)		
	C91230F	Unknown	221	10	60598150	2123	2.12		52.7	Unknown	0.00	1	60598108	0.00	<LOQ (0.119 ug/g)		NA
	C91237F	Unknown	421	10	60598151	4020	4.02	4.09	2.16	Unknown	0.00	1	60598109	0.00	<LOQ (0.119 ug/g)	<LOQ	NA
Group 3	C90846M	Unknown	989	100	90298049	97855	97.9 +			Unknown	128	1	90298064	128	0.128		
Mid Dose	C90853M	Unknown	525	100	90298050	50290	50.3 +			Unknown	45.7	1	90298065	45.7	0.0457		
30.0 mg/kg	C90866M	Unknown	529	100	90298051	50940	50.9 +			Unknown	64.0	1	90298066	64.0	0.0640		
	C90890M	Unknown	403	100	90298052	39263	39.3 +		37.9	Unknown	117	1	90298067	117	0.117		40.4
	C90891M	Unknown	608	100	90298053	59731	59.7 +	59.6	22.6	Unknown	79.6	1	90298068	79.6	0.0796	0.0870	0.0351
	C91251F	Unknown	462	100	90298057	46022	46.0 +			Unknown	51.3	1	90298072	51.3	0.0513		
	C91272F	Unknown	991	100	90298058	93774	93.8 +			Unknown	49.1	1	90298073	49.1	0.0491		
	C91285F	Unknown	260	100	90298059	25750	25.8 +			Unknown	44.8	1	90298074	44.8	0.0448		
	C91287F	Unknown	440	100	90298060	41154	41.2 +		55.1	Unknown	41.9	1	90298075	41.9	0.0419		15.4
	C91291F	Unknown	1020	100	90298061	102446	102 +	61.8	34.1	Unknown	33.9	1	90298076	33.9	0.0339	0.0442	0.00682
Group 4	C90935M	Unknown	486	100	M07099813	50589	50.6			Unknown	656	1	90298079	683	0.683		
Mid-High Dose	C90939M	Unknown	384	100	M07099814	36794	36.8			Unknown	167	1	90298080	NA	NA		
100 mg/kg	C90942M	Unknown	335	100	M07099815	33108	33.1			Unknown	328	1	90298081	324	0.324		
	C90961M	Unknown	936	100	M07099816	88123	88.1		41.2	Unknown	449	1	90298082	422	0.422		33.1
	C90966M	Unknown	579	100	M07099817	56911	56.9	53.1	21.9	Unknown	432	1	90298083	425	0.425	0.464	0.154
	C91316F	Unknown	509	100	M07099820	49348	49.3			Unknown	640	1	90298087	620	0.620		
	C91317F	Unknown	531	100	M07099821	53684	53.7			Unknown	267	1	90298088	270	0.270		
	C91333F	Unknown	543	100	M07099822	53610	53.6			Unknown	154	1	90298089	NA	NA		
	C91345F	Unknown	792	100	M07099823	73837	73.8		28.3	Unknown	124	1	90298090	NA	NA		56.7
	C91350F	Unknown	337	100	M07099824	32106	32.1	52.5	14.9	Unknown	247	1	90298091	235	0.235	0.375	0.213
Group 5	C90984M	Unknown	79.7	1000	M07099827	79219	79.2			Unknown	375	1	90298094	372	0.372		
High Dose	C91005M	Unknown	62.1	1000	M07099828	60714	60.7 *			Unknown	496	1	90298095	486	0.486		
300 mg/kg	C91024M	Unknown	94.6	1000	M07099829	93469	93.5			Unknown	1127	1	90298096	1113	1.11 ++		
	C91033M	Unknown	120	1000	M07099830	115688	116		23.8	Unknown	1045	1	90298097	1011	1.01 ++		47.5
	C91037M	Unknown	81.8	1000	M07099831	79582	79.6	85.7	20.4	Unknown	551	1	90298098	536	0.536	0.704	0.335
	C91423F	Unknown	85.0	1000	M07099834	83911	83.9			Unknown	926	1	90298102	914	0.914		
	C91427F	Unknown	32.3	1000	M07099835	31653	31.7 *			Unknown	1459	1	90298103	1428	1.43 ++		
	C91430F	Unknown	30.2	1000	M07099836	29614	29.6 *			Unknown	753	1	90298104	739	0.739		
	C91443F	Unknown	151	1000	M07099837	149030	149		63.5	Unknown	364	1	90298105	361	0.361		58.2
	C91448F	Unknown	108	1000	M07099838	106357	106	80.1	50.9	Unknown	371	1	90298106	366	0.366	0.762	0.443

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
Date Entered/Analyst: 05/23/00, 06/20/00, 08/15/00 CSH/LAC 12/05/00 KJH
Date Verified/Analyst: 12/05/00 KJH, 12/06/00 HOJ
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0595 & 0.0119 ug/g) to include std correction factors
new LOQs are 0.0552 & 0.0110 ug/g. LAC 02/19/01

+ CCVs not within criteria due to carryover, estimated results.
* Below LOQ, tentative numbers.
++ Above the linear range of the curve, data are estimated.
^ Samples were mislabeled during the extraction or analysis. A deviation was written and sample data switched.

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0
Analytical Equipment System Number: Chick 080697, Madeline 041098, and Amelia 062498
Instrument Software/Version: MassLynx 2.3 and 3.1
Date of Extraction/Analyst: 05/29/98 and 06/01/98 RWW/OK/LAC
Date of Analysis/Analyst: 06/03/98, 06/05/98, 06/12/98, 06/14/98, 07/09/98, 09/02/98 HOJ/KJH/MEE
Date of Data Reduction/Analyst: 02/25/00, 03/14/00, 03/15/00, 03/16/00, 11/29/00, 12/10/00 MMH/IAS

Filename: See Attachments
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 4 RAT LIVER REWORK

Group Dose	Sample #	PFOS Calc. Conc. ng/g	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	PFOSA Calc. Conc. ng/g	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	PFOSAA Calc. Conc. ng/g	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD	EtFOSE Calc. Conc. ng/g	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	11.5	<LOQ (0.0552 ug/g)			0.00	<LOQ (0.0121 ug/g)			3.85	<LOQ (0.0633 ug/g)			32.5	<LOQ (0.0596 ug/g)		
	H2O Blk-2	0.00	<LOQ (0.0110 ug/g)	<LOQ	NA	0.00	<LOQ (0.00604 ug/g)	<LOQ	NA	0.00	<LOQ (0.127 ug/g)	<LOQ	NA	0.00	<LOQ (0.119 ug/g)	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	7.97	<LOQ (0.0552 ug/g)			0.00	<LOQ (0.0121 ug/g)			0.00	<LOQ (0.0633 ug/g)			0.00	<LOQ (0.0596 ug/g)		
	Rabbit Liver Blk-2	0.00	<LOQ (0.0110 ug/g)	<LOQ	NA	0.00	<LOQ (0.00604 ug/g)	<LOQ	NA	0.00	<LOQ (0.127 ug/g)	<LOQ	NA	0.00	<LOQ (0.119 ug/g)	<LOQ	NA
QC - 100 ppb	C90719M-MS^	233	196%			121	100%			131	103%			189	158%		
	C90719M-MSD	223	187%	192%	5%	132	110%	105%	9%	134	106%	104%	3%	129	109%	133%	37%
	C90719M^	728	0.728			8.97	0.00897			51.5	0.0515			0.00	<LOQ (0.0596 ug/g)		
Control	C90725M	647	0.647			9.86	<LOQ (0.0121 ug/g)			105	0.105			0.00	<LOQ (0.0596 ug/g)		
Group 1	C90732M	980	0.980			17.8	0.0178			143	0.143			0.00	<LOQ (0.0596 ug/g)		
	C90753M	670	0.670		17.8	10.5	<LOQ (0.0121 ug/g)		49.9	151	0.151		46.1	30.7	<LOQ (0.0596 ug/g)		NA
	C90775M	844	0.844	0.774	0.138	23.8	0.0238	0.0150	0.00746	219	0.219	0.134	0.0617	0.00	<LOQ (0.0596 ug/g)	<LOQ	NA
	C91135F	203	0.203			2.20	<LOQ (0.0121 ug/g)			11.3	<LOQ (0.0633 ug/g)			0.00	<LOQ (0.0596 ug/g)		
	C91143F	254	0.254			22.1	0.0221			77.9	0.0779			0.00	<LOQ (0.0596 ug/g)		
	C91165F	564	0.564			30.4	0.0304			161	0.161			0.00	<LOQ (0.0596 ug/g)		
	C91169F	402	0.402		39.0	13.8	0.0138		43.7	79.8	0.0798		39.2	0.00	<LOQ (0.0596 ug/g)		NA
	C91171F	418	0.418	0.368	0.144	13.5	0.0135	0.0184	0.00804	115	0.115	0.0995	0.0390	0.00	<LOQ (0.0596 ug/g)	<LOQ	NA
Group 2	C90789M	12658	12.7			491	0.491			2087	2.09			22.2	<LOQ (0.119 ug/g)		
Low Dose	C90814M	10487	10.5			302	0.302			1940	1.94			0.00	<LOQ (0.119 ug/g)		
3.0 mg/kg	C90817M	11723	11.7			485	0.485			1846	1.85			0.00	<LOQ (0.119 ug/g)		
	C90833M	15011	15.0		13.5	863	0.863		37.8	9732	9.73		94.4	0.00	<LOQ (0.119 ug/g)		NA
	C90834M	12069	12.1	12.4	1.67	670	0.670	0.562	0.212	2528	2.53	3.63	3.42	0.00	<LOQ (0.119 ug/g)	<LOQ	NA
	C91198F	11027	11.0			636	0.636			7681	7.68			0.00	<LOQ (0.119 ug/g)		
	C91205F	9588	9.59			635	0.635			2751	2.75			0.00	<LOQ (0.119 ug/g)		
	C91222F	13282	13.3			651	0.651			3893	3.89			68.0	<LOQ (0.119 ug/g)		
	C91230F	9412	9.41		17.1	761	0.761		8.39	2123	2.12		52.7	0.00	<LOQ (0.119 ug/g)		NA
	C91237F	13426	13.4	11.3	1.94	722	0.722	0.681	0.0572	4020	4.02	4.09	2.16	0.00	<LOQ (0.119 ug/g)	<LOQ	NA
Group 3	C90846M	64520	64.5			7961	7.96			97855	97.9 +			128	0.128		
Mid Dose	C90853M	86362	86.4			5083	5.08			50290	50.3 +			45.7	0.0457		
30.0 mg/kg	C90866M	110931	111			7159	7.16			50940	50.9 +			64.0	0.0640		
	C90890M	109767	110		21.2	7276	7.28		15.9	39263	39.3 +		37.9	117	0.117		40.4
	C90891M	108824	109	96.1	20.4	6598	6.60	6.82	1.08	59731	59.7 +	59.6	22.6	79.6	0.0796	0.0870	0.0351
	C91251F	99018	99.0			5934	5.93			46022	46.0 +			51.3	0.0513		
	C91272F	121385	121			7413	7.41			93774	93.8 +			49.1	0.0491		
	C91285F	91929	91.9			4761	4.76			25750	25.8 +			44.8	0.0448		
	C91287F	138167	138		21.0	5566	5.57		17.6	41154	41.2 +		55.1	41.9	0.0419		15.4
	C91291F	151495	151	120	25.3	7036	7.04	6.14	1.08	102446	102 +	61.8	34.1	33.9	0.0339	0.0442	0.00682
Group 4	C90935M	128159	128			9841	9.84			50589	50.6			683	0.683		
Mid-High Dose	C90939M	167259	167			6639	6.64			36794	36.8			NA	NA		
100 mg/kg	C90942M	202805	203			8722	8.72			33108	33.1			324	0.324		
	C90961M	263867	264		26.8	13084	13.1		24.4	88123	88.1		41.2	422	0.422		33.1
	C90966M	231647	232	199	53.2	9543	9.54	9.57	2.33	56911	56.9	53.1	21.9	425	0.425	0.464	0.154
	C91316F	169257	169			7302	7.30			49348	49.3			620	0.620		
	C91317F	419299	419			10851	10.9			53684	53.7			270	0.270		
	C91333F	220960	221			9162	9.16			53610	53.6			NA	NA		
	C91345F	243655	244		52.8	8889	8.89		26.5	73837	73.8		28.3	NA	NA		56.7
	C91350F	92724	92.7	229	121	5043	5.04	8.25	2.19	32106	32.1	52.5	14.9	235	0.235	0.375	0.213
Group 5	C90984M	191685	192			1362	1.36 *			79219	79.2			372	0.372		
High Dose	C91005M	391682	392			6181	6.18 *			60714	60.7 *			486	0.486		
300 mg/kg	C91024M	661557	662			12528	12.5 *			93469	93.5			1113	1.11 ++		
	C91033M	691925	692		41.7	13154	13.2		56.5	115688	116		23.8	1011	1.01 ++		47.5
	C91037M	559479	559	499	208	11465	11.5	8.94	5.05	79582	79.6	85.7	20.4	536	0.536	0.704	0.335
	C91423F	557635	558			15517	15.5			83911	83.9			914	0.914		
	C91427F	379688	380			18156	18.2			31653	31.7 *			1428	1.43 ++		
	C91430F	488915	489			16152	16.2			29614	29.6 *			739	0.739		
	C91443F	816358	816		30.1	14645	14.6		15.1	149030	149		63.5	361	0.361		58.2
	C91448F	740764	741	597	180	11836	11.8	15.3	2.31	106357	106	80.1	50.9	366	0.366	0.762	0.443

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
Date Entered/Analyst: 05/23/00, 06/20/00, 08/15/00 CSH/LAC 12/05/00 KJH
Date Verified/Analyst: 12/05/00 KJH, 12/06/00 HOJ
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0595 & 0.0119 ug/g) to include std correction factors
new LOQs are 0.0552 & 0.0110 ug/g. LAC 02/19/01

+ CCVs not within criteria due to carryover, estimated results.
* Below LOQ, tentative numbers.
++ Above the linear range of the curve, data are estimated.
^ Samples were mislabeled during the extraction or analysis. A deviation was written and sample data switched.

AMDT# 092597.1
Covance# 6329-212

		06/14/98 Lot 193	06/12/98 Lot L-2353	06/12/98 Lot 617	06/12/98 Lot 936
		06/18/98,12/29/98	09/02/98 Lot L-2353	06/18/98 Lot 617	09/02/98 Lot 936

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0
Analytical Equipment System Number: Chick 080697 and Madeline 041098
Instrument Software/Version: MassLynx 2.3 and 3.1
Date of Extraction/Analyst: 06/01/98 RWW/OK
Date of Analysis/Analyst: 06/12/98, 06/14/98, 06/18/98, 09/02/98 HOJ/KJH/LAC
Date of Data Reduction/Analyst: 03/16/00, 11/29/00, 12/01/00, 12/10/00 IAS/MMH/KJH

Filename: See List to Right
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Filenames

	PFOS	PFOSA	PFOSAA	EtFOSE	Dilutions
Blks	M06189834-35 & 60-61	A90298002-3 & 116-117	M06189834-35 & 60-61	A90298002-3 &	1/1, 1/1, 1/1, 1/1
Grp 1	06189852-57, 122998069-	90298019-31	06189845-57	90298019-31	1/1, 1/1, 1/1, 1/1
Grp 5	61498013-24	122998041-52	122998055-66	need 1:10 dil.	1/10000, 1/100, 1/1000, 1/10
MS, MSD	61298127-128	61298127-128	61298127-128	90298110-111	need 1:5 dil, 1/1, 1/1, 1/1

Sample Data

WEEK 8 RAT LIVER REWORK Lot 193

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Conc. ng/g	PFOS Dilution Factor	PFOS Calc. Conc. ng/g	Filename	Concentration of PFOS ug/g or % Rec.		Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	1.0000	NA	0.9275	Unknown	0.00	1	0.00	61298056	<LOQ (0.0110 ug/g)			
	H2O Blk-2	1.0000	NA	0.9275	Unknown	47.4	1	43.9	122998002	<LOQ (0.112 ug/g)		<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	1.0080	40.13	0.9275	Unknown	0.00	1	0.00	61298057	<LOQ (0.0110 ug/g)			
	Rabbit Liver Blk-2	1.0080	40.13	0.9275	Unknown	33.1	1	30.4	122998003	<LOQ (0.112 ug/g)		< LOQ	NA
QC - 100 ppb	C90717M-MS	1.0248	NA	NA	NA	1153	1	-1584	61298127	-1331%	*		
	C90717M-MSD	1.0248	NA	NA	NA	959	1	-1773	61298128	-1490%	*	-1410%	11%
Group 1	C90717M	1.0455	NA	0.9275	Unknown	313	10	2776	122998073	2.78	+		
Control	C90718M	1.0268	NA	0.9275	Unknown	222	10	2004	122998072	2.00	+		
0.0 mg/kg	C90737M	1.0434	NA	0.9275	Unknown	307	10	2731	122998071	2.73	+		
	C90756M	1.0089	NA	0.9275	Unknown	218	10	2002	122998070	2.00	+		31.3
	C90769M	1.0083	NA	0.9275	Unknown	124	10	1144	122998069	1.14	+	2.13	0.668
	C91126F	1.0205	NA	0.9275	Unknown	270	1	245	6189852	0.245			
	C91140F	1.0265	NA	0.9275	Unknown	383	1	346	6189853	0.346			
	C91145F	1.0128	NA	0.9275	Unknown	558	1	511	6189854	0.511			
	C91162F	1.0160	NA	0.9275	Unknown	1198	1	1094	6189855	1.09	*		59.8
	C91163F	1.0162	NA	0.9275	Unknown	605	1	552	6189856	0.552		0.549	0.328
Group 5	C90971M	1.0129	NA	0.9275	Unknown	137	10000	1250280	61498013	1250			
High Dose	C90972M	1.0204	NA	0.9275	Unknown	122	10000	1111564	61498014	1112			
300 mg/kg	C90988M	1.0108	NA	0.9275	Unknown	136	10000	1249941	61498015	1250			
	C90996M	1.0128	NA	0.9275	Unknown	97.3	10000	890777	61498016	891			18.5
	C91017M	1.0116	NA	0.9275	Unknown	90.6	10000	830679	61498017	831		1067	197
	C91393F	1.0247	NA	0.9275	Unknown	96.5	10000	873644	61498020	874			
	C91394F	1.0126	NA	0.9275	Unknown	103	10000	945819	61498021	946			
	C91404F	1.0149	NA	0.9275	Unknown	112	10000	1027570	61498022	1028			
	C91415F	1.0244	NA	0.9275	Unknown	116	10000	1054710	61498023	1055			20.0
	C91438F	1.0230	NA	0.9275	Unknown	157	10000	1423345	61498024	1423		1065	213

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

* Data above the linear calibration range, estimated value.
\+ CCV's failed, data entered as estimated
**confirmed low on 90298
++ Qualitative data only.

Date Entered/Analyst: 4/13/00, 5/24/00 CSH 12/12/00 MMH
Date Verified/Analyst: 12/28/00 hoj (list a122998a)
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0119 & 0.0121 ug/g) to include std correction factors
new LOQs are 0.0110 & 0.112 ug/g. LAC 02/19/01

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0	Filename:	See List to Right
Analytical Equipment System Number:	Chick 080697 and Madeline 041098	R-Squared Value:	See Attachments
Instrument Software/Version:	MassLynx 2.3 and 3.1	Slope:	See Attachments
Date of Extraction/Analyst:	06/01/98 RWW/OK	Y-Intercept:	See Attachments
Date of Analysis/Analyst:	06/12/98, 06/14/98, 06/18/98, 09/02/98 HOJ/KJH/LAC		
Date of Data Reduction/Analyst:	03/16/00, 11/29/00, 12/01/00, 12/10/00 IAS/MMH/KJH		

Sample Data

WEEK 8 RAT LIVER REWORK Lot L-2353

Group Dose	Sample #	PFOSA Purity Correction Factor	PFOSA Conc. ng/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ng/g	Filename	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	Unknown	0.0	1	0.00	61298056	<LOQ (0.0119 ug/g)		
	H2O Blk-2	Unknown	66.3	1	66.3	12998002	0.0663 +	0.0391	NA
Matrix Blk	Rabbit Liver Blk-1	Unknown	0.0	1	0.00	61298057	<LOQ (0.0119 ug/g)		
	Rabbit Liver Blk-2	Unknown	41.8	1	41.4	12298003	0.0414 +	0.0267	NA
QC - 100 ppb	C90717M-MS	NA	83.4	1	48.4	61298127	40% **		
	C90717M-MSD	NA	65.4	1	30.8	61298128	26% **	33%	44%
Group 1	C90717M	Unknown	33.8	1	32.4	90298019	0.0324		
Control	C90718M	Unknown	19.6	1	<LOQ (0.0302 ug/g)	90298020	<LOQ (0.0302 ug/g)		
0.0 mg/kg	C90737M	Unknown	35.2	1	33.7	90298021	0.0337		
	C90756M	Unknown	31.1	1	<LOQ (0.0302 ug/g)	90298022	<LOQ (0.0302 ug/g)		5.21
	C90769M	Unknown	10.1	1	<LOQ (0.0302 ug/g)	90298023	<LOQ (0.0302 ug/g)	0.0313	0.00163
	C91126F	Unknown	10.4	1	<LOQ (0.0302 ug/g)	90298027	<LOQ (0.0302 ug/g)		
	C91140F	Unknown	10.4	1	<LOQ (0.0302 ug/g)	90298028	<LOQ (0.0302 ug/g)		
	C91145F	Unknown	12.7	1	<LOQ (0.0302 ug/g)	90298029	<LOQ (0.0302 ug/g)		
	C91162F	Unknown	35.0	1	34.4	90298030	0.0344		6.09
	C91163F	Unknown	15.2	1	<LOQ (0.0302 ug/g)	90298031	<LOQ (0.0302 ug/g)	0.0310	0.00189
Group 5	C90971M	Unknown	196	100	19353	12298045	19.4 +		
High Dose	C90972M	Unknown	191	100	18747	12298044	18.7 +		
300 mg/kg	C90988M	Unknown	191	100	18897	12298043	18.9 +		
	C90996M	Unknown	211	100	20877	12298042	20.9 +		5.13
	C91017M	Unknown	185	100	18323	12298041	18.3 +	19.2	0.987
	C91393F	Unknown	154	100	14999	12298052	15.0 +		
	C91394F	Unknown	142	100	14071	12298051	14.1 +		
	C91404F	Unknown	174	100	17148	12298050	17.1 +		
	C91415F	Unknown	142	100	13844	12298049	13.8 +		10.7
	C91438F	Unknown	177	100	17272	12298048	17.3 +	15.5	1.65

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

* Data above the linear calibration range, estimated value.
\+ CCV's failed, data entered as estimated
**confirmed low on 90298
++ Qualitative data only.

Date Entered/Analyst: 4/13/00, 5/24/00 CSH 12/12/00 MMH
Date Verified/Analyst: 12/28/00 hoj (list a122998a)
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0119 & 0.0121 ug/g) to include std correction factors
new LOQs are 0.0110 & 0.112 ug/g. LAC 02/19/01

AMDT# 092597.1
Covance# 6329-212

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0
Analytical Equipment System Number: Chick 080697 and Madeline 041098
Instrument Software/Version: MassLynx 2.3 and 3.1
Date of Extraction/Analyst: 06/01/98 RWW/OK
Date of Analysis/Analyst: 06/12/98, 06/14/98, 06/18/98, 09/02/98 HOJ/KJH/LAC
Date of Data Reduction/Analyst: 03/16/00, 11/29/00, 12/01/00, 12/10/00 IAS/MMH/KJH

Filename: See List to Right
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 8 RAT LIVER

Group Dose	Sample #	PFOSAA Purity Correction Factor	PFOSAA Conc. ng/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ng/g	Filename	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD	EtFOSE Purity Correction Factor	EtFOSE Conc. ng/g	EtFOSE Dilution Factor	EtFOSE Calc. Conc. ng/g	Filename	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD
		Lot 617								Lot 936							
Method Blk	H2O Blk-1	Unknown	27.2	1	27.2	6189834	<LOQ (0.633 ug/g)			Unknown	2.44	1	2.44	90298002	<LOQ (0.0298 ug/g) ++		
	H2O Blk-2	Unknown	0.00	1	0.00	s001206005	<LOQ (0.633 ug/g)	<LOQ	NA	Unknown		1	0.00		0.00 ++	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	Unknown	13.0	1	12.9	6189835	<LOQ (0.633 ug/g)			Unknown	0.00	1	0.00	90298003	<LOQ (0.0298 ug/g) ++		
	Rabbit Liver Blk-2	Unknown	0.00	1	0.00	s001206006	<LOQ (0.633 ug/g)	<LOQ	NA	Unknown		1	0.00		0.00 ++	<LOQ	NA
QC - 100 ppb	C90717M-MS	NA	88.6	1	12.3	61298127	10% +			NA	46.5	1	45.4	90298110	38% ++		
	C90717M-MSD	NA	97.6	1	21.0	61298128	17% +	13%	52%	NA	30.3	1	29.5	90298111	25% ++	31%	42%
Group 1	C90717M	Unknown	76.0	1	72.7	6189845	0.0727			Unknown	0.00	1	0.00	90298019	<LOQ (0.0298 ug/g) ++		
Control	C90718M	Unknown	90.4	1	88.1	6189846	0.0881			Unknown	0.00	1	0.00	90298020	<LOQ (0.0298 ug/g) ++		
0.0 mg/kg	C90737M	Unknown	156	1	149	6189847	0.149			Unknown	0.00	1	0.00	90298021	<LOQ (0.0298 ug/g) ++		
	C90756M	Unknown	118	1	117	6189848	0.117		35.8	Unknown	0.00	1	0.00	90298022	<LOQ (0.0298 ug/g) ++		12.7
	C90769M	Unknown	3.13	1	3.10	6189849	<LOQ (0.633 ug/g)	0.0980	0.0351	Unknown	39.2	1	38.8	90298023	0.0388 ++	0.0316	0.00402
	C91126F	Unknown	15.6	1	15.2	6189852	<LOQ (0.633 ug/g)			Unknown	0.00	1	0.00	90298027	<LOQ (0.0298 ug/g) ++		
	C91140F	Unknown	1.74	1	1.70	6189853	<LOQ (0.633 ug/g)			Unknown	0.00	1	0.00	90298028	<LOQ (0.0298 ug/g) ++		
	C91145F	Unknown	40.6	1	40.1	6189854	<LOQ (0.633 ug/g)			Unknown	0.00	1	0.00	90298029	<LOQ (0.0298 ug/g) ++		
	C91162F	Unknown	195	1	192	6189855	0.192		61.1	Unknown	0.00	1	0.00	90298030	<LOQ (0.0298 ug/g) ++		NA
	C91163F	Unknown	79.4	1	78.1	6189856	0.0781	0.0920	0.0561	Unknown	0.00	1	0.00	90298031	<LOQ (0.0298 ug/g) ++	<LOQ	NA
Group 5	C90971M	Unknown	512	1000	505667	122998059	506 +			Unknown		10	0.00		0.00 ++		
High Dose	C90972M	Unknown	594	1000	581791	122998058	582 +			Unknown		10	0.00		0.00 ++		
300 mg/kg	C90988M	Unknown	478	1000	473002	122998057	473 +			Unknown	892	1	883	90298036	0.883 ++		
	C90996M	Unknown	480	1000	473865	122998056	474 +		15.1	Unknown		10	0.00		0.00 ++		137
	C91017M	Unknown	384	1000	379557	122998055	380 +	483	72.7	Unknown	816	1	807	90298038	0.807 ++	0.338	0.463
	C91393F	Unknown	306	1000	298858	122998066	299 +			Unknown	805	1	786	90298042	0.786 ++		
	C91394F	Unknown	272	1000	268201	122998065	268 +			Unknown		10	0.00		0.00 ++		
	C91404F	Unknown	375	1000	369672	122998064	370 +			Unknown		10	0.00		0.00 ++		
	C91415F	Unknown	293	1000	286207	122998063	286 +		20.3	Unknown	347	1	339	90298045	0.339 ++		102
	C91438F	Unknown	439	1000	429003	122998062	429 +	330	67.2	Unknown	650	1	636	90298046	0.636 ++	0.352	0.359

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

* Data above the linear calibration range, estimated value.
\+ CCV's failed, data entered as estimated
**confirmed low on 90298
++ Qualitative data only.

Date Entered/Analyst: 4/13/00, 5/24/00 CSH 12/12/00 MMH
Date Verified/Analyst:
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0119 & 0.0121 ug/g) to include std correction factors
new LOQs are 0.0110 & 0.112 ug/g. LAC 02/19/01

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0		
Analytical Equipment System Number:	Chick 080697 and Madeline 041098	Filename:	See Attachments
Instrument Software/Version:	MassLynx 2.3 and 3.1	R-Squared Value:	See Attachments
Date of Extraction/Analyst:	06/01/98 RWW/OK	Slope:	See Attachments
Date of Analysis/Analyst:	06/12/98, 06/14/98, 06/18/98, 09/02/98 HOJ/KJH/LAC	Y-Intercept:	See Attachments
Date of Data Reduction/Analyst:	03/16/00, 11/29/00, 12/01/00, 12/10/00 IAS/MMH/KJH		

Sample Data

WEEK 8 RAT LIVER REWORK

Group Dose	Sample #	PFOS Calc. Conc. ng/g	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	PFOSA Calc. Conc. ng/g	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	PFOSAA Calc. Conc. ng/g	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD	EtFOSE Calc. Conc. ng/g	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	0.00	<LOQ (0.0110 ug/g)			0.00	<LOQ (0.0119 ug/g)			27.2	<LOQ (0.633 ug/g)			2.44	<LOQ (0.0298 ug/g) ++		
	H2O Blk-2	43.9	<LOQ (0.112 ug/g)	<LOQ	NA	66.3	0.0663 +	0.0391	NA	0.00	<LOQ (0.633 ug/g)	<LOQ	NA	0.00	0.00 ++	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	0.00	<LOQ (0.0110 ug/g)			0.00	<LOQ (0.0119 ug/g)			12.9	<LOQ (0.633 ug/g)			0.00	<LOQ (0.0298 ug/g) ++		
	Rabbit Liver Blk-2	30.4	<LOQ (0.112 ug/g)	< LOQ	NA	41.4	0.0414 +	0.0267	NA	0.00	<LOQ (0.633 ug/g)	<LOQ	NA	0.00	0.00 ++	<LOQ	NA
QC - 100 ppb	C90717M-MS	-1584	-1331% *			48.4	40% **			12.3	10% +			45.4	38% ++		
	C90717M-MSD	-1773	-1490% *	-1410%	11%	30.8	26% **	33%	44%	21.0	17% +	13%	52%	29.5	25% ++	31%	42%
Group 1	C90717M	2776	2.78 +			32.4	0.0324			72.7	0.0727			0.00	<LOQ (0.0298 ug/g) ++		
Control	C90718M	2004	2.00 +			<LOQ (0.0302 ug/g)	<LOQ (0.0302 ug/g)			88.1	0.088			0.00	<LOQ (0.0298 ug/g) ++		
0.0 mg/kg	C90737M	2731	2.73 +			33.7	0.0337			149	0.149			0.00	<LOQ (0.0298 ug/g) ++		
	C90756M	2002	2.00 +		31.3	<LOQ (0.0302 ug/g)	<LOQ (0.0302 ug/g)		5.21	117	0.117		35.8	0.00	<LOQ (0.0298 ug/g) ++		12.7
	C90769M	1144	1.14 +	2.13	0.668	<LOQ (0.0302 ug/g)	<LOQ (0.0302 ug/g)	0.0313	0.00163	3.10	<LOQ (0.633 ug/g)	0.0980	0.0351	38.8	0.0388 ++	0.0316	0.00402
	C91126F	245	0.245			<LOQ (0.0302 ug/g)	<LOQ (0.0302 ug/g)			15.2	<LOQ (0.633 ug/g)			0.00	<LOQ (0.0298 ug/g) ++		
	C91140F	346	0.346			<LOQ (0.0302 ug/g)	<LOQ (0.0302 ug/g)			1.70	<LOQ (0.633 ug/g)			0.00	<LOQ (0.0298 ug/g) ++		
	C91145F	511	0.511			<LOQ (0.0302 ug/g)	<LOQ (0.0302 ug/g)			40.1	<LOQ (0.633 ug/g)			0.00	<LOQ (0.0298 ug/g) ++		
	C91162F	1094	1.09 *		59.8	34.4	0.0344		6.09	192	0.192		61.1	0.00	<LOQ (0.0298 ug/g) ++		NA
	C91163F	552	0.552	0.549	0.328	<LOQ (0.0302 ug/g)	<LOQ (0.0302 ug/g)	0.0310	0.00189	78.1	0.0781	0.0920	0.0561	0.00	<LOQ (0.0298 ug/g) ++	<LOQ	NA
Group 5	C90971M	1250280	1250			19353	19.4 +			505667	506 +			0.00	0.00 ++		
High Dose	C90972M	1111564	1112			18747	18.7 +			581791	582 +			0.00	0.00 ++		
300 mg/kg	C90988M	1249941	1250			18897	18.9 +			473002	473 +			883	0.883 ++		
	C90996M	890777	891		18.5	20877	20.9 +		5.13	473865	474 +		15.1	0.00	0.00 ++		137
	C91017M	830679	831	1067	197	18323	18.3 +	19.2	0.99	379557	380 +	483	72.7	807	0.807 ++	0.338	0.463
	C91393F	873644	874			14999	15.0 +			298858	299 +			786	0.786 ++		
	C91394F	945819	946			14071	14.1 +			268201	268 +			0.00	0.00 ++		
	C91404F	1027570	1028			17148	17.1 +			369672	370 +			0.00	0.00 ++		
	C91415F	1054710	1055		20.0	13844	13.8 +		10.7	286207	286 +		20.3	339	0.339 ++		102
	C91438F	1423345	1423	1065	213	17272	17.3 +	15.5	1.65	429003	429 +	330	67.2	636	0.636 ++	0.352	0.359

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

* Data above the linear calibration range, estimated value.
\+ CCV's failed, data entered as estimated
**confirmed low on 90298
++ Qualitative data only.

Date Entered/Analyst:	4/13/00, 5/24/00 CSH 12/12/00 MMH
Date Verified/Analyst:	12/28/00 hoj (list a122998a)
Purity Entered/Verified:	12/26/00 LAC

Corrected PFOS LOQs (0.0119 & 0.0121 ug/g) to include std correction factors
new LOQs are 0.0110 & 0.112 ug/g. LAC 02/19/01

PFOS	PFOSA	PFOSAA	EtFOSE
12/14/98 Lot 193	06/26/98 Lot L-2353	06/26/98 Lot 617	06/25/98 Lot 936
01/18/99 Lot 171	12/14/98 Lot L-2353	12/14/98 Lot 617	02/18/99 Lot Unknown
02/16/99 Lot 171	02/18/99 Lot Unknown	02/18/99 Lot Unknown	
02/18/99 Lot 171			

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98 1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0
Analytical Equipment System Number: Chick 080698, Amelia 062498, Madeline 041098, and Soup 020199
Instrument Software/Version: MassLynx 2.3 and 3.1
Date of Extraction/Analyst: 06/02/98, 01/14/99, 01/17/99 RWW/IAS/SAH
Date of Analysis/Analyst: 06/25/98, 06/26/98, 12/14/98, 01/18/99, 02/16/99, 02/18/99 HOJ/KJH/MEE/DRB
Date of Data Reduction/Analyst: 04/05/00, 11/30/00, 12/01/00, 12/05/00, 12/07/00, 01/02/01 CSH/MMH/KJH

Filename: See List to Right
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 14 RAT LIVER REWORK

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Conc. ng/g	PFOS Dilution Factor	Filename	PFOS Calc. Conc. ng/g	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD	PFOSA Purity Correction Factor	PFOSA Conc. ng/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ng/g	Filename	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD
					Lot 171								Lot L-2353 or Unknown							
Method Blk	H2O Blk-1	1.0000	NA	0.9275	0.8640	0.00	1	M011899013	0.00	<LOQ (0.0496 ug/g)			Unknown	0.00	1	0.00	M062698057	<LOQ (0.00604 ug/g)		
	H2O Blk-2	1.0000	NA	0.9275	0.8640	0.00	1	S021899003	0.00	<LOQ (0.0248 ug/g)	<LOQ	NA	Unknown	NA	1	NA	NA	NA	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	1.0080	40.13	0.9275	0.8640	0.00	1	M011899014	0.00	<LOQ (0.0496 ug/g)			Unknown	0.00	1	0.00	M062698058	<LOQ (0.00604 ug/g)		
	Rabbit Liver Blk-2	1.0080	40.13	0.9275	0.8640	0.00	1	S021899004	0.00	<LOQ (0.0248 ug/g)	<LOQ	NA	Unknown	NA	1	NA	NA	NA	<LOQ	NA
QC - 100 ppb	C90768M-MS	1.0214	NA	NA	NA	867	1	M121498109	-351	-295%			NA	102	1	97.3	M121498109	81%		
	C90768M-MSD	1.0214	NA	NA	NA	1023	1	M121498110	-199	-167%	-231%	55%	NA	90.3	1	85.8	M121498110	71%	76%	13%
QC - 250 ppb	C90780M-MS	1.0166	NA	NA	NA	88.9	10	M021699036	-27.0	-9%			NA	NA	1	NA	NA	NA		
	C90780M-MSD	1.0166	NA	NA	NA	98.3	10	M021699037	65.3	22%	6%	481%	NA	NA	1	NA	NA	NA	NA	NA
QC - 750 ppb	C91174F-MS	1.0123	NA	NA	NA	140	10	S021899015	1136	126%			NA	NA	1	NA	NA	NA		
	C91174F-MSD	1.0123	NA	NA	NA	151	10	S021899016	1245	138%	132%	9%	NA	NA	1	NA	NA	NA	NA	NA
Group 1	C90731M	1.0133	NA	0.9275	0.8640	348	4	M011899025	1100	1.10			Unknown	54.4	1	53.7	M121498014	0.0537		
Control	C90746M	1.0226	NA	0.9275	0.8640	425	4	M011899026	1332	1.33			Unknown	0.00	1	0.00	M121498015	<LOQ (0.0121 ug/g)		
0.0 mg/kg	C90748M	1.0072	NA	0.9275	0.8640	504	4	M011899027	1603	1.60			Unknown	E	1	E	E	E		
	C90768M	1.0214	NA	0.9275	0.8640	383	4	M011899028	1200	1.20		21.4	Unknown	2.74	1	2.68	M121498016	<LOQ (0.0121 ug/g)		71.1
	C90780M	1.0166	NA	0.9275	0.8640	286	4	M011899029	901	0.901	1.23	0.262	Unknown	36.5	1	35.9	M121498017	0.0359	0.0284	0.0202
	C91129F	1.0264	NA	0.9275	0.8640	133	4	M011899032	417	0.417			Unknown	6.60	1	6.43	M121498021	<LOQ (0.0121 ug/g)		
	C91155F	1.0156	NA	0.9275	0.8640	155	4	M011899033	489	0.489			Unknown	9.75	1	9.60	M121498022	<LOQ (0.0121 ug/g)		
	C91160F	1.0103	NA	0.9275	0.8640	175	4	M011899034	555	0.555			Unknown	29.9	1	29.6	M121498023	0.0296		
	C91174F	1.0123	NA	0.9275	0.8640	76.6	4	M011899035	243	0.243		32.1	Unknown	2.44	1	2.41	M121498024	<LOQ (0.0121 ug/g)		50.2
	C91181F	1.0251	NA	0.9275	0.8640	97.0	4	M011899036	303	0.303	0.402	0.129	Unknown	0.00	1	0.00	M121498025	<LOQ (0.0121 ug/g)	0.0156	0.00784
Group 2	C90797M	1.0060	NA	0.9275	0.8640	230	125	M011899039	22950	23.0			Unknown	133	10	1319	M121498060	1.32		
Low Dose	C90807M	1.0258	NA	0.9275	0.8640	231	125	M011899040	22597	22.6			Unknown	111	10	1084	M121498061	1.08		
3.0 mg/kg	C90818M	1.0085	NA	0.9275	0.8640	247	125	M011899041	24494	24.5			Unknown	89.7	10	889	M121498062	0.889		
	C90831M	1.0231	NA	0.9275	0.8640	210	125	M011899042	20514	20.5		9.16	Unknown	134	10	1305	M121498063	1.31		17.5
	C90836M	1.0155	NA	0.9275	0.8640	266	125	M011899043	26220	26.2	23.4	2.14	Unknown	98.3	10	968	M121498064	0.968	1.11	0.194
	C91192F	1.0047	NA	0.9275	0.8640	214	125	M011899046	21300	21.3			Unknown	194	10	1927	M121498068	1.93		
	C91223F	1.0062	NA	0.9275	0.8640	235	125	M011899047	23437	23.4			Unknown	171	10	1697	M121498069	1.70		
	C91233F	1.0257	NA	0.9275	0.8640	289	125	M011899048	28206	28.2			Unknown	183	10	1786	M121498070	1.79		
	C91241F	1.0093	NA	0.9275	0.8640	195	125	M011899049	19334	19.3		14.8	Unknown	180	10	1781	M121498071	1.78		4.61
	C91248F	1.0031	NA	0.9275	0.8640	256	125	M011899050	25542	25.5	23.6	3.48	Unknown	182	10	1815	M121498072	1.82	1.80	0.0831
Group 3	C90843M	1.0214	NA	0.9275	0.8640	26.3	2000	M011899053	41221	41.2 *			Unknown	68.2	100	6672	M062698067	6.67		
Mid Dose	C90852M	1.0105	NA	0.9275	0.8640	72.0	2000	M011899054	114260	114			Unknown	91.3	100	9036	M062698068	9.04		
30.0 mg/kg	C90863M	1.0349	NA	0.9275	0.8640	26.1	2000	M011899055	40451	40.5 *			Unknown	58.2	100	5626	M062698069	5.63		
	C90877M	1.0019	NA	0.9275	0.8640	96.2	2000	M011899056	153905	154		63.0	Unknown	101	100	10096	M062698070	10.1		24.2
	C90880M	1.0018	NA	0.9275	0.8640	33.9	2000	M011899057	54251	54.3 *	80.8	50.9	Unknown	98.6	100	9840	M062698071	9.84	8.25	2.00
	C91281F	1.0201	NA	0.9275	0.8640	87.2	2000	M011899060	137003	137 +			Unknown	87.6	100	8589	M062698074	8.59		
	C91288F	1.0210	NA	0.9275	0.8640	85.8	2000	M011899061	134654	135 +			Unknown	66.2	100	6480	M062698075	6.48		
	C91293F	1.0016	NA	0.9275	0.8640	70.9	2000	M011899062	113467	113 +			Unknown	73.7	100	7359	M062698076	7.36		
	C91299F	1.0173	NA	0.9275	0.8640	148	2000	M011899063	233295	233 +		39.8	Unknown	90.9	100	8935	M062698077	8.94		12.9
	C91304F	1.0277	NA	0.9275	0.8640	54.1	2000	M011899064	84401	84.4 +	141	56.0	Unknown	87.5	100	8512	M062698078	8.51	7.98	1.03
Group 4	C90905M	1.0099	NA	0.9275	0.8640	523	1000	M021699022	415193	415			Unknown	183	100	18099	M121498092	18.1		
Mid-High Dose	C90907M	1.0123	NA	0.9275	0.8640	686	1000	M021699023	543101	543			Unknown	158	100	15579	M121498093	15.6		
100 mg/kg	C90917M	1.0104	NA	0.9275	0.8640	469	1000	M021699024	372033	372			Unknown	153	100	15150	M121498094	15.2		
	C90921M	1.0378	NA	0.9275	0.8640	407	1000	M021699025	314243	314		24.0	Unknown	172	100	16600	M121498095	16.6		7.28
	C90960M	1.0243	NA	0.9275	0.8640	405	1000	M021699026	316914	317	392	94.1	Unknown	177	100	17258	M121498096	17.3	16.5	1.20
	C91329F	1.0016	NA	0.9275	0.8640	328	1000	M021699029	262314	262			Unknown	127	100	12659	M121498100	12.7		
	C91337F	1.0180	NA	0.9275	0.8640	377	1000	M021699030	296535	297			Unknown	160	100	15721	M121498101	15.7		
	C91355F	1.0081	NA	0.9275	0.8640	313	1000	M021699031	248810	249			Unknown	142	100	14058	M121498102	14.1		
	C91370F	1.0150	NA	0.9275	0.8640	823	1000	M021699032	649710	650		53.3	Unknown	143	100	14066	M121498103	14.1		10.4
	C91379F	1.0090	NA	0.9275	0.8640	273	1000	M021699033	216709	217	335	178	Unknown	167	100	16522	M121498104	16.5	14.6	1.52

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

Note: PFOS data may be biased high based on the MS/MSD recoveries.
+ Bracketed by a CCV at -30% difference, data may be biased low. LAC 02/19/01
* Reportd below the LOQ
++ PFOSAA MS/MSD not fully bracketed. LAC 12/14/00
+++ EtFOSE MS/MSD reported, only a 4 point curve could be generated. LAC 12/14/00
E = Sample went to dryness after the initial analysis. No extract remaining for analysis.
# Qualitative data only, based on MS/MSD results.

Date Entered/Analyst: 5/24/00, 12/07/00, 12/14/00 CSH/KJH/LAC
Date Verified/Analyst: 12/28/00 HOJ
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0619 & 0.0309 ug/g) to include std correction factors
new LOQs are 0.0496 & 0.0248 ug/g. LAC 02/19/01

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0
Analytical Equipment System Number: Chick 080698, Amelia 062498, Madeline 041098, and Soup 020199
Instrument Software/Version: MassLynx 2.3 and 3.1
Date of Extraction/Analyst: 5/11/98, 6/2/98, 01/14/99 RWW/IAS/SAH
Date of Analysis/Analyst: 6/5/98, 12/14/98, 01/18/99, 02/16/99, 02/18/99 HOJ/KJH/MEE/DRB
Date of Data Reduction/Analyst: 12/22/98, 01/20/99, 02/22/99, 3/23/00 HOJ/KJH/DRB/MMH

Filename: See Attachments
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 14 RAT LIVER

Group Dose	Sample #	PFOSAA Purity Correction Factor (Lot 617 or Unknown)	PFOSAA Conc. ng/g	PFOSAA Dilution Factor	Filename	PFOSAA Calc. Conc. ug/g	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD	EtFOSE Purity Correction Factor (Lot 936 or Unknown)	EtFOSE Conc. ng/g	EtFOSE Dilution Factor	Filename	EtFOSE Calc. Conc. ng/g	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD
Method Blk	H2O Blk-1	Unknown	0.00	1	M121498003	0.00	<LOQ (0.0633 ug/g)			Unknown	0.00	1	A062598002	0.00	<LOQ (0.0596 ug/g) #		
	H2O Blk-2	Unknown	NA	1	NA	NA	NA	<LOQ	NA	Unknown	NA	1	NA	NA	NA #	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	Unknown	0.00	1	M121498004	0.00	<LOQ (0.0633 ug/g)			Unknown	0.00	1	A062598003	0.00	<LOQ (0.0596 ug/g) #		
	Rabbit Liver Blk-2	Unknown	NA	1	NA	NA	NA	<LOQ	NA	Unknown	NA	1	NA	NA	NA #	<LOQ	NA
QC - 100 ppb	C90768M-MS	NA	115	1	M121498109	-93.9	-74% ++			NA	NA	1	NA	NA	NA #		
	C90768M-MSD	NA	153	1	M121498110	-56.6	-45% ++	-59%	50%	NA	NA	1	NA	NA	NA #	NA	NA
QC - 250 ppb	C90780M-MS	NA	NA	1	NA	NA	NA			NA	NA	1	NA	NA	NA #		
	C90780M-MSD	NA	NA	1	NA	NA	NA	NA	NA	NA	NA	1	NA	NA	NA #	NA	NA
QC - 750 ppb	C91174F-MS	NA	905	1	S021899017	665	74%			NA	516	10	S021899015	5102	567% +++		
	C91174F-MSD	NA	1315	1	S021899018	1070	119%	97%	47%	NA	480	10	S021899016	4739	527% +++	547%	7%
Group 1 Control 0.0 mg/kg	C90731M	Unknown	174	1	M121498014	172	0.172			Unknown	0.00	1	A062598034	0.00	<LOQ (0.0596 ug/g) #		
	C90746M	Unknown	143	1	M121498015	140	0.140			Unknown	0.00	1	A062598035	0.00	<LOQ (0.0596 ug/g) #		
	C90748M	Unknown	E	1	E	E	E			Unknown	0.00	1	A062598036	0.00	<LOQ (0.0596 ug/g) #		
	C90768M	Unknown	211	1	M121498016	206	0.206		42.0	Unknown	0.00	1	A062598037	0.00	<LOQ (0.0596 ug/g) #		NA
	C90780M	Unknown	29.0	1	M121498017	28.5	<LOQ (0.0633 ug/g)	0.145	0.0611	Unknown	0.00	1	A062598038	0.00	<LOQ (0.0596 ug/g) #	<LOQ	NA
	C91129F	Unknown	76.4	1	M121498021	74.5	0.0745			Unknown	0.00	1	A062598041	0.00	<LOQ (0.0596 ug/g) #		
	C91155F	Unknown	94.9	1	M121498022	93.4	0.0934			Unknown	0.00	1	A062598042	0.00	<LOQ (0.0596 ug/g) #		
	C91160F	Unknown	69.1	1	M121498023	68.4	0.0684			Unknown	0.00	1	A062598043	0.00	<LOQ (0.0596 ug/g) #		
	C91174F	Unknown	232	1	M121498024	229	0.229		66.1	Unknown	0.00	1	A062598044	0.00	<LOQ (0.0596 ug/g) #		NA
	C91181F	Unknown	21.8	1	M121498025	21.3	<LOQ (0.0633 ug/g)	0.106	0.0699	Unknown	0.00	1	A062598045	0.00	<LOQ (0.0596 ug/g) #	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90797M	Unknown	238	10	M121498060	2363	2.36			Unknown	NA	1	NA	NA	NA #		
	C90807M	Unknown	145	10	M121498061	1414	1.41			Unknown	NA	1	NA	NA	NA #		
	C90818M	Unknown	180	10	M121498062	1786	1.79			Unknown	NA	1	NA	NA	NA #		
	C90831M	Unknown	203	10	M121498063	1984	1.98		18.9	Unknown	NA	1	NA	NA	NA #		NA
	C90836M	Unknown	175	10	M121498064	1719	1.72	1.85	0.351	Unknown	NA	1	NA	NA	NA #	NA	NA
	C91192F	Unknown	218	10	M121498068	2168	2.17			Unknown	NA	1	NA	NA	NA #		
	C91223F	Unknown	326	10	M121498069	3240	3.24			Unknown	NA	1	NA	NA	NA #		
	C91233F	Unknown	262	10	M121498070	2556	2.56			Unknown	NA	1	NA	NA	NA #		
	C91241F	Unknown	232	10	M121498071	2295	2.29		17.4	Unknown	NA	1	NA	NA	NA #		NA
	C91248F	Unknown	303	10	M121498072	3023	3.02	2.66	0.462	Unknown	NA	1	NA	NA	NA #	NA	NA
Group 3 Mid Dose 30.0 mg/kg	C90843M	Unknown	7.58	100	M062698067	742	0.742 *			Unknown	234	1	A062598076	229	0.229 #		
	C90852M	Unknown	202	100	M062698068	20013	20.0			Unknown	177	1	A062598077	175	0.175 #		
	C90863M	Unknown	116	100	M062698069	11226	11.2			Unknown	325	1	A062598078	314	0.314 #		
	C90877M	Unknown	358	100	M062698070	35706	35.7		72.8	Unknown	146	1	A062598079	146	0.146 #		32.8
	C90880M	Unknown	326	100	M062698071	32557	32.6	20.0	14.6	Unknown	168	1	A062598080	167	0.167 #	0.206	0.0677
	C91281F	Unknown	53.8	100	M062698074	5269	5.27 *			Unknown	152	1	A062598083	149	0.149 #		
	C91288F	Unknown	140	100	M062698075	13748	13.7			Unknown	142	1	A062598084	139	0.139 #		
	C91293F	Unknown	128	100	M062698076	12775	12.8			Unknown	171	1	A062598085	171	0.171 #		
	C91299F	Unknown	302	100	M062698077	29678	29.7		60.0	Unknown	117	1	A062598086	115	0.115 #		15.1
	C91304F	Unknown	134	100	M062698078	13073	13.1	14.9	8.95	Unknown	170	1	A062598087	165	0.165 #	0.148	0.0224
Group 4 Mid-High Dose 100 mg/kg	C90905M	Unknown	953	100	M121498092	94336	94.3			Unknown	NA	1	NA	NA	NA #		
	C90907M	Unknown	749	100	M121498093	73968	74.0			Unknown	NA	1	NA	NA	NA #		
	C90917M	Unknown	619	100	M121498094	61232	61.2			Unknown	NA	1	NA	NA	NA #		
	C90921M	Unknown	1209	100	M121498095	116449	116.4		29.7	Unknown	NA	1	NA	NA	NA #		NA
	C90960M	Unknown	612	100	M121498096	59712	59.7	81.1	24.1	Unknown	NA	1	NA	NA	NA #	NA	NA
	C91329F	Unknown	404	100	M121498100	40356	40.4			Unknown	NA	1	NA	NA	NA #		
	C91337F	Unknown	800	100	M121498101	78625	78.6			Unknown	NA	1	NA	NA	NA #		
	C91355F	Unknown	641	100	M121498102	63619	63.6			Unknown	NA	1	NA	NA	NA #		
	C91370F	Unknown	510	100	M121498103	50197	50.2		24.6	Unknown	NA	1	NA	NA	NA #		NA
	C91379F	Unknown	640	100	M121498104	63386	63.4	59.2	14.6	Unknown	NA	1	NA	NA	NA #	NA	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

Note: PFOS data may be biased high based on the MS/MSD recoveries.
+ Bracketed by a CCV at -30% difference, data may be biased low. LAC 02/19/01
* Reportd below the LOQ
++ PFOSAA MS/MSD not fully bracketed. LAC 12/14/00
+++ EtFOSE MS/MSD reported, only a 4 point curve could be generated. LAC 12/14/00
E = Sample went to dryness after the initial analysis. No extract remaining for analysis.
# Qualitative data only, based on MS/MSD results.

Date Entered/Analyst: 5/24/00, 12/07/00, 12/14/00 CSH/KJH/LAC
Date Verified/Analyst: 12/28/00 HOJ
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0619 & 0.0309 ug/g) to include std correction factors
new LOQs are 0.0496 & 0.0248 ug/g. LAC 02/19/01

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0 reworked using ETS-8-7.0
Analytical Equipment System Number: Chick 080698, Amelia 062498, Madeline 041098, and Soup 020199
Instrument Software/Version: MassLynx 2.3 and 3.1
Date of Extraction/Analyst: 06/02/98, 01/14/99, 01/17/99 RWW/IAS/SAH
Date of Analysis/Analyst: 06/25/98, 06/26/98, 12/14/98, 01/18/99, 02/16/99, 02/18/99 HOJ/KJH/MEE/DRB
Date of Data Reduction/Analyst: 04/05/00, 11/30/00, 12/01/00, 12/05/00, 12/07/00, 01/02/01 CSH/MMH/KJH

Filename: See List to Right
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 14 RAT LIVER REWORK

Group Dose	Sample #	PFOS Calc. Conc. ug/g	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD	PFOSA Calc. Conc. ug/g	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD	PFOSAA Calc. Conc. ug/g	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD	EtFOSE Calc. Conc. ug/g	Concentration of EtFOSE ug/g or % Rec.		Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD
Method Blk	H2O Blk-1	0.00	<LOQ (0.0496 ug/g)			0.00	<LOQ (0.00604 ug/g)			0.00	<LOQ (0.0633 ug/g)			0.00	<LOQ (0.0596 ug/g)	#		
	H2O Blk-2	0.00	<LOQ (0.0248 ug/g)	<LOQ	NA	NA	NA	<LOQ	NA	NA	NA	<LOQ	NA	NA	NA	#	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	0.00	<LOQ (0.0496 ug/g)			0.00	<LOQ (0.00604 ug/g)			0.00	<LOQ (0.0633 ug/g)			0.00	<LOQ (0.0596 ug/g)	#		
	Rabbit Liver Blk-2	0.00	<LOQ (0.0248 ug/g)	<LOQ	NA	NA	NA	<LOQ	NA	NA	NA	<LOQ	NA	NA	NA	#	<LOQ	NA
QC - 100 ppb	C90768M-MS	-351	-295%			97.3	81%			-94	-74% ++			NA	NA	#		
	C90768M-MSD	-199	-167%	-231%	55%	85.8	71%	76%	13%	-57	-45% ++	-59%	50%	NA	NA	#	NA	NA
QC - 250 ppb	C90780M-MS	-27	-9%			NA	NA			NA	NA			NA	NA	#		
	C90780M-MSD	65	22%	6%	481%	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	#	NA	NA
QC - 750 ppb	C91174F-MS	1136	126%			NA	NA			665	74%			5102	567%	+++		
	C91174F-MSD	1245	138%	132%	9%	NA	NA	NA	NA	1070	119%	97%	47%	4739	527%	+++	547%	7%
Group 1	C90731M	1100	1.10			53.7	0.0537			172	0.172			0.00	<LOQ (0.0596 ug/g)	#		
Control	C90746M	1332	1.33			0.00	<LOQ (0.0121 ug/g)			140	0.140			0.00	<LOQ (0.0596 ug/g)	#		
0.0 mg/kg	C90748M	1603	1.60			E	E			E	E			0.00	<LOQ (0.0596 ug/g)	#		
	C90768M	1200	1.20		21.4	2.68	<LOQ (0.0121 ug/g)		71.1	206	0.206		42.0	0.00	<LOQ (0.0596 ug/g)	#		NA
	C90780M	901	0.90	1.23	0.262	35.9	0.0359	0.0284	0.0202	28.5	<LOQ (0.0633 ug/g)	0.145	0.0611	0.00	<LOQ (0.0596 ug/g)	#	<LOQ	NA
	C91129F	417	0.417			6.43	<LOQ (0.0121 ug/g)			74.5	0.0745			0.00	<LOQ (0.0596 ug/g)	#		
	C91155F	489	0.489			9.60	<LOQ (0.0121 ug/g)			93.4	0.0934			0.00	<LOQ (0.0596 ug/g)	#		
	C91160F	555	0.555			29.6	0.0296			68.4	0.0684			0.00	<LOQ (0.0596 ug/g)	#		
	C91174F	243	0.243		32.1	2.41	<LOQ (0.0121 ug/g)		50.2	229	0.229		66.1	0.00	<LOQ (0.0596 ug/g)	#		NA
	C91181F	303	0.303	0.402	0.129	0.00	<LOQ (0.0121 ug/g)	0.0156	0.00784	21.3	<LOQ (0.0633 ug/g)	0.106	0.0699	0.00	<LOQ (0.0596 ug/g)	#	<LOQ	NA
Group 2	C90797M	22950	23.0			1319	1.32			2363	2.36			NA	NA	#		
Low Dose	C90807M	22597	22.6			1084	1.08			1414	1.41			NA	NA	#		
3.0 mg/kg	C90818M	24494	24.5			889	0.889			1786	1.79			NA	NA	#		
	C90831M	20514	20.5		9.16	1305	1.31		17.5	1984	1.98		18.9	NA	NA	#		NA
	C90836M	26220	26.2	23.4	2.14	968	0.968	1.11	0.194	1719	1.72	1.85	0.351	NA	NA	#	NA	NA
	C91192F	21300	21.3			1927	1.93			2168	2.17			NA	NA	#		
	C91223F	23437	23.4			1697	1.70			3240	3.24			NA	NA	#		
	C91233F	28206	28.2			1786	1.79			2556	2.56			NA	NA	#		
	C91241F	19334	19.3		14.8	1781	1.78		4.61	2295	2.29		17.4	NA	NA	#		NA
	C91248F	25542	25.5	23.6	3.48	1815	1.82	1.80	0.0831	3023	3.02	2.66	0.462	NA	NA	#	NA	NA
Group 3	C90843M	41221	41.2 *			6672	6.67			742	0.742 *			229	0.229	#		
Mid Dose	C90852M	114260	114			9036	9.04			20013	20.0			175	0.175	#		
30.0 mg/kg	C90863M	40451	40.5 *			5626	5.63			11226	11.2			314	0.314	#		
	C90877M	153905	154		63.0	10096	10.1		24.2	35706	35.7		72.8	146	0.146	#		32.8
	C90880M	54251	54 *	80.8	50.9	9840	9.84	8.25	2.00	32557	32.6	20.0	14.6	167	0.167	#	0.206	0.0677
	C91281F	137003	137 +			8589	8.59			5269	5.27 *			149	0.149	#		
	C91288F	134654	135 +			6480	6.48			13748	13.7			139	0.139	#		
	C91293F	113467	113 +			7359	7.36			12775	12.8			171	0.171	#		
	C91299F	233295	233 +		39.8	8935	8.94		12.9	29678	29.7		60.0	115	0.115	#		15.1
	C91304F	84401	84 +	141	56.0	8512	8.51	7.98	1.03	13073	13.1	14.9	8.95	165	0.165	#	0.148	0.0224
Group 4	C90905M	415193	415			18099	18.1			94336	94.3			NA	NA	#		
Mid-High Dose	C90907M	543101	543			15579	15.6			73968	74.0			NA	NA	#		
100 mg/kg	C90917M	372033	372			15150	15.2			61232	61.2			NA	NA	#		
	C90921M	314243	314		24.0	16600	16.6		7.28	116449	116		29.7	NA	NA	#		NA
	C90960M	316914	317	392	94.1	17258	17.3	16.5	1.20	59712	59.7	81.1	24.1	NA	NA	#	NA	NA
	C91329F	262314	262			12659	12.7			40356	40.4			NA	NA	#		
	C91337F	296535	297			15721	15.7			78625	78.6			NA	NA	#		
	C91355F	248810	249			14058	14.1			63619	63.6			NA	NA	#		
	C91370F	649710	650		53.3	14066	14.1		10.4	50197	50.2		24.6	NA	NA	#		NA
	C91379F	216709	217	335	178	16522	16.5	14.6	1.52	63386	63.4	59.2	14.6	NA	NA	#	NA	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

Note: PFOS data may be biased high based on the MS/MSD recoveries.
+ Bracketed by a CCV at -30% difference, data may be biased low. LAC 02/19/01
* Reportd below the LOQ
++ PFOSAA MS/MSD not fully bracketed. LAC 12/14/00
+++ EtFOSE MS/MSD reported, only a 4 point curve could be generated. LAC 12/14/00
E = Sample went to dryness after the initial analysis. No extract remaining for analysis.
# Qualitative data only, based on MS/MSD results.

Date Entered/Analyst: 5/24/00, 12/07/00, 12/14/00 CSH/KJH/LAC
Date Verified/Analyst: 12/28/00 HOJ
Purity Entered/Verified: 12/26/00 LAC

Corrected PFOS LOQs (0.0619 & 0.0309 ug/g) to include std correction factors
new LOQs are 0.0496 & 0.0248 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename:	See Below
Analytical Equipment System Number:	Davey 070799	R-Squared Value:	See Attachments
Instrument Software/Version:	MassLynx 3.3, 3.4	Slope:	See Attachments
Date of Extraction/Analyst:	2/29/00 SAL	Y-Intercept:	See Attachments
Date of Analysis/Analyst:	05/08/00, 05/24/00, 08/21/00, 08/23/00 IAS/HOJ/MMH		
Date of Data Reduction/Analyst:	05/09/00, 05/25/00, 08/22/00, 08/28/00 IAS/MMH/HOJ		

Sample Data

WEEK 53 RAT LIVER Lot 171

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor	Surrogate Verified	PFOS Conc. ng/g	PFOS Dilution Factor	PFOS Calc. Conc. ng/g	Filename	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL02290-H2O Blk-5	1.0000	NA	0.9275	0.8640	NA	2.15	1	1.72	A082100016	<LOQ (0.00491 ug/g)		NA
	RBL02290-H2O Blk-6	1.0000	NA	0.9275	0.8640	NA	0.00	1	0.00	A082100017	<LOQ (0.00491 ug/g)	<LOQ	NA
Matrix Blk	RBL02290-Liver Blk-5	1.0000	40.13	0.9275	0.8640	NA	0.00	1	0.00	A082100018	<LOQ (0.00491 ug/g)		NA
	RBL02290-Liver Blk-6	1.0000	40.13	0.9275	0.8640	NA	3.16	1	2.53	A082100019	<LOQ (0.00491 ug/g)	<LOQ	NA
QC - 250 ppb	C90714M-250 ppb-MS-5-1	1.0093	NA	NA	NA	Confirmed low	1239	1	528	D050800019	177% *		
	C90714M-250 ppb-MS-5-2	1.0093	NA	NA	NA	Confirmed low	1006	1	298	D050800020	100%	138%	56%
Group 1	C90714M	1.0093	NA	0.9275	0.8640	NA	706	1	560	D050800023	0.560		
Control	C90751M	1.0033	NA	0.9275	0.8640	NA	473	1	378	D050800024	0.378		
0.0 mg/kg	C90752M	0.9994	NA	0.9275	0.8640	NA	993	1	796	D050800025	0.796		
	C90754M	0.9912	NA	0.9275	0.8640	NA	857	1	693	D050800026	0.693		30.7
	C90767M	0.9845	NA	0.9275	0.8640	NA	526	1	428	D050800027	0.428	0.571	0.175
Group 1	C91121F	0.9972	NA	0.9275	0.8640	NA	374	1	301	D050800030	0.301		
Control	C91122F	1.0073	NA	0.9275	0.8640	NA	383	1	305	D050800031	0.305		
0.0 mg/kg	C91124F	1.0079	NA	0.9275	0.8640	NA	539	1	429	D050800032	0.429		
	C91133F	0.9918	NA	0.9275	0.8640	NA	668	1	540	D050800033	0.540		25.5
	C91152F	1.0162	NA	0.9275	0.8640	NA	474	1	374	D050800034	0.374	0.390	0.0993
Group 4	C90902M	0.9925	NA	0.9275	0.8640	NA	739	1000	596825	D050800037	597		
Low Dose	C90918M	1.0022	NA	0.9275	0.8640	NA	548	1000	437941	D050800038	438		
3.0 mg/kg	C90950M	0.9878	NA	0.9275	0.8640	NA	1138	1000	923568	D050800039	924		
	C90951M	1.0037	NA	0.9275	0.8640	NA	875	1000	698501	D050800040	699		27.2
	C90957M	0.9880	NA	0.9275	0.8640	NA	1008	1000	817501	D050800041	818	695	189
Group 4	C91323F	0.9939	NA	0.9275	0.8640	NA	820	1000	661438	D050800044	661		
Low Dose	C91347F	0.9917	NA	0.9275	0.8640	NA	1065	1000	860688	D050800045	861		
3.0 mg/kg	C91352F	0.9983	NA	0.9275	0.8640	NA	959	1000	770046	D050800046	770		
	C91367F	0.9908	NA	0.9275	0.8640	NA	776	1000	627330	D050800047	627		17.7
	C91371F	0.9903	NA	0.9275	0.8640	NA	678	1000	548628	D050800048	549	694	123

* High recovery was confirmed on 8/21/00, data was not entered because it was outside the calibration range. 4/20/01 mmh

PFOS – Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Date Entered/Analyst:	05/15/00, 06/12/00, 09/03/00 CSH/LAC
Date Verified/Analyst:	06/05/00. 06/06/00 CSH: 02/26/01 hoj
Purity Entered/Verified:	02/16/01 LAC

Corrected PFOS LOQ (0.0613 ug/g) to include std correction factors
new LOQ is 0.0491 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename:	See Below
Analytical Equipment System Number:	Davey 070799	R-Squared Value:	See Attachments
Instrument Software/Version:	MassLynx 3.3, 3.4	Slope:	See Attachments
Date of Extraction/Analyst:	2/29/00 SAL	Y-Intercept:	See Attachments
Date of Analysis/Analyst:	05/08/00, 05/24/00, 08/21/00, 08/23/00 IAS/HOJ/MMH		
Date of Data Reduction/Analyst:	05/09/00, 05/25/00, 08/22/00, 08/28/00 IAS/MMH/HOJ		

Sample Data

WEEK 53 RAT LIVER Lot L-15709

Group Dose	Sample #	PFOSA Purity Correction Factor	Surrogate Verified	PFOSA Conc. ng/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ng/g	Filename	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL02290-H2O Blk-5	Unknown	NA	0.00	1	0.00	D050800004	<LOQ (0.0123 ug/g)		NA
	RBL02290-H2O Blk-6	Unknown	NA	0.00	1	0.00	D050800017	<LOQ (0.0123 ug/g)	<LOQ	NA
Matrix Blk	RBL02290-Liver Blk-5	Unknown	NA	2.58	1	2.58	D050800005	<LOQ (0.0123 ug/g)		NA
	RBL02290-Liver Blk-6	Unknown	NA	0.00	1	0.00	D050800018	<LOQ (0.0123 ug/g)	<LOQ	NA
QC - 250 ppb	C90714M-250 ppb-MS-5-1	NA	Confirmed Low	233	1	230	D050800019	77%		
	C90714M-250 ppb-MS-5-2	NA	Confirmed Low	288	1	285	D050800020	95%	86%	21%
Group 1	C90714M	Unknown	NA	0.710	1	0.703	A082100024	<LOQ (0.00614 ug/g)		
Control	C90751M	Unknown	NA	35.5	1	35.4	A082100025	0.0354		
0.0 mg/kg	C90752M	Unknown	NA	11.7	1	11.7	A082100026	0.0117		
	C90754M	Unknown	NA	18.1	1	18.3	A082100027	0.0183		55.1
	C90767M	Unknown	NA	19.3	1	19.6	A082100028	0.0196	0.0182	0.0100
Group 1	C91121F	Unknown	NA	12.5	1	12.5	A082100031	0.0125		
Control	C91122F	Unknown	NA	5.41	1	5.37	A082100032	<LOQ (0.00614 ug/g)		
0.0 mg/kg	C91124F	Unknown	NA	25.5	1	25.3	A082100033	0.0253		
	C91133F	Unknown	NA	15.8	1	15.9	A082100034	0.0159		48.7
	C91152F	Unknown	NA	9.87	1	9.71	A082100035	0.00971	0.0139	0.00677
Group 4	C90902M	Unknown	NA	296	100	29822	D052400040	29.8		
Low Dose	C90918M	Unknown	NA	196	100	19579	D052400041	19.6		
3.0 mg/kg	C90950M	Unknown	NA	289	100	29280	D052400042	29.3		
	C90951M	Unknown	NA	216	100	21481	D052400043	21.5		29.9
	C90957M	Unknown	2nd Analysis OK	404	100	40923	D052400047	40.9	28.2	8.44
Group 4	C91323F	Unknown	NA	236	100	23731	D052400048	23.7		
Low Dose	C91347F	Unknown	2nd Analysis OK	216	100	21786	D052400049	21.8		
3.0 mg/kg	C91352F	Unknown	Confirmed High	219	100	21911	D052400050	21.9		
	C91367F	Unknown	High, Not Confirmed	264	100	26678	A082300016	26.7		17.3
	C91371F	Unknown	High, Not Confirmed	319	100	32243	A082300017	32.2	25.3	4.37

PFOS – Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Date Entered/Analyst:	05/15/00, 06/12/00, 09/03/00 CSH/LAC
Date Verified/Analyst:	06/05/00. 06/06/00 CSH: 02/26/01 hoj
Purity Entered/Verified:	02/16/01 LAC

Corrected PFOS LOQ (0.0613 ug/g) to include std correction factors
new LOQ is 0.0491 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename:	See Below
Analytical Equipment System Number:	Davey 070799	R-Squared Value:	See Attachments
Instrument Software/Version:	MassLynx 3.3, 3.4	Slope:	See Attachments
Date of Extraction/Analyst:	2/29/00 SAL	Y-Intercept:	See Attachments
Date of Analysis/Analyst:	05/08/00, 05/24/00, 08/21/00, 08/23/00 IAS/HOJ/MMH		
Date of Data Reduction/Analyst:	05/09/00, 05/25/00, 08/22/00, 08/28/00 IAS/MMH/HOJ		

Sample Data

WEEK 53 RAT LIVER Lot T-7121.1

Group Dose	Sample #	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Conc. ng/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ng/g	Filename	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL02290-H2O Blk-5	Unknown	NA	0.00	1	0.00	D050800004	<LOQ (0.0307 ug/g)		NA
	RBL02290-H2O Blk-6	Unknown	NA	0.00	1	0.00	D050800017	<LOQ (0.0307 ug/g)	<LOQ	NA
Matrix Blk	RBL02290-Liver Blk-5	Unknown	NA	0.00	1	0.00	D050800005	<LOQ (0.0307 ug/g)		NA
	RBL02290-Liver Blk-6	Unknown	NA	0.00	1	0.00	D050800018	<LOQ (0.0307 ug/g)	<LOQ	NA
QC - 250 ppb	C90714M-250 ppb-MS-5-1	NA	Confirmed Low	339	1	327	D050800019	109%		
	C90714M-250 ppb-MS-5-2	NA	Confirmed Low	370	1	359	D050800020	120%	114%	9%
Group 1	C90714M	Unknown	NA	8.54	1	8.46	D050800023	<LOQ (0.0307 ug/g)		
Control	C90751M	Unknown	NA	48.7	1	48.6	D050800024	0.0486		
0.0 mg/kg	C90752M	Unknown	NA	8.27	1	8.27	D050800025	<LOQ (0.0307 ug/g)		
	C90754M	Unknown	NA	44.0	1	44.4	D050800026	0.0444		NA
	C90767M	Unknown	NA	4.96	1	5.04	D050800027	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 1	C91121F	Unknown	NA	8.95	1	8.98	D050800030	<LOQ (0.0307 ug/g)		
Control	C91122F	Unknown	NA	4.04	1	4.01	D050800031	<LOQ (0.0307 ug/g)		
0.0 mg/kg	C91124F	Unknown	NA	61.6	1	61.1	D050800032	<LOQ (0.0307 ug/g)		
	C91133F	Unknown	NA	25.5	1	25.7	D050800033	<LOQ (0.0307 ug/g)		NA
	C91152F	Unknown	NA	16.0	1	15.7	D050800034	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 4	C90902M	Unknown	NA	803	100	80884	D050800051	80.9		
Low Dose	C90918M	Unknown	NA	402	100	40068	D050800052	40.1		
3.0 mg/kg	C90950M	Unknown	NA	677	100	68551	D050800053	68.6		
	C90951M	Unknown	NA	286	100	28475	D050800054	28.5		38.2
	C90957M	Unknown	NA	600	100	60700	D050800055	60.7	55.7	21.3
Group 4	C91323F	Unknown	NA	491	100	49362	D050800058	49.4		
Low Dose	C91347F	Unknown	NA	409	100	41192	D050800059	41.2		
3.0 mg/kg	C91352F	Unknown	NA	451	100	45161	D050800060	45.2		
	C91367F	Unknown	NA	457	100	46121	D050800061	46.1		11.9
	C91371F	Unknown	NA	558	100	56329	D050800062	56.3	47.6	5.67

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide
Date Entered/Analyst: 05/15/00, 06/12/00, 09/03/00 CSH/LAC
Date Verified/Analyst: 06/05/00. 06/06/00 CSH: 02/26/01 hoj
Purity Entered/Verified: 02/16/01 LAC

Corrected PFOS LOQ (0.0613 ug/g) to include std correction factors
new LOQ is 0.0491 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename:	See Below
Analytical Equipment System Number:	Davey 070799	R-Squared Value:	See Attachments
Instrument Software/Version:	MassLynx 3.3, 3.4	Slope:	See Attachments
Date of Extraction/Analyst:	2/29/00 SAL	Y-Intercept:	See Attachments
Date of Analysis/Analyst:	05/08/00, 05/24/00, 08/21/00, 08/23/00 IAS/HOJ/MMH		
Date of Data Reduction/Analyst:	05/09/00, 05/25/00, 08/22/00, 08/28/00 IAS/MMH/HOJ		

Sample Data

WEEK 53 RAT LIVER lot Unknown

Group Dose	Sample #	EtFOSE Purity Correction Factor	Surrogate Verified	EtFOSE Conc. ng/g	EtFOSE Dilution Factor	EtFOSE Calc. Conc. ng/g	Filename	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL02290-H2O Blk-5	Unknown	NA	NR	1	NR	NA	NR		NA
	RBL02290-H2O Blk-6	Unknown	NA	NR	1	NR	NA	NR	NR	NA
Matrix Blk	RBL02290-Liver Blk-5	Unknown	NA	NR	1	NR	NA	NR		NA
	RBL02290-Liver Blk-6	Unknown	NA	NR	1	NR	NA	NR	NR	NA
QC - 250 ppb	C90714M-250 ppb-MS-5-1	NA	NA	NR	1	NR	NA	NR		
	C90714M-250 ppb-MS-5-2	NA	NA	NR	1	NR	NA	NR	NR	NA
Group 1	C90714M	Unknown	NA	NR	1	NR	NA	NR		
Control	C90751M	Unknown	NA	NR	1	NR	NA	NR		
0.0 mg/kg	C90752M	Unknown	NA	NR	1	NR	NA	NR		
	C90754M	Unknown	NA	NR	1	NR	NA	NR		NA
	C90767M	Unknown	NA	NR	1	NR	NA	NR	NR	NA
Group 1	C91121F	Unknown	NA	NR	1	NR	NA	NR		
Control	C91122F	Unknown	NA	NR	1	NR	NA	NR		
0.0 mg/kg	C91124F	Unknown	NA	NR	1	NR	NA	NR		
	C91133F	Unknown	NA	NR	1	NR	NA	NR		NA
	C91152F	Unknown	NA	NR	1	NR	NA	NR	NR	NA
Group 4	C90902M	Unknown	NA	NR	1	NR	NA	NR		
Low Dose	C90918M	Unknown	NA	NR	1	NR	NA	NR		
3.0 mg/kg	C90950M	Unknown	NA	NR	1	NR	NA	NR		
	C90951M	Unknown	NA	NR	1	NR	NA	NR		NA
	C90957M	Unknown	NA	NR	1	NR	NA	NR	NR	NA
Group 4	C91323F	Unknown	NA	NR	1	NR	NA	NR		
Low Dose	C91347F	Unknown	NA	NR	1	NR	NA	NR		
3.0 mg/kg	C91352F	Unknown	NA	NR	1	NR	NA	NR		
	C91367F	Unknown	NA	NR	1	NR	NA	NR		NA
	C91371F	Unknown	NA	NR	1	NR	NA	NR	NR	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Date Entered/Analyst: 05/15/00, 06/12/00, 09/03/00 CSH/LAC
Date Verified/Analyst: 06/05/00. 06/06/00 CSH: 02/26/01 hoj
Purity Entered/Verified: 02/16/01 LAC

Corrected PFOS LOQ (0.0613 ug/g) to include std correction factors
new LOQ is 0.0491 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename:	See Below
Analytical Equipment System Number:	Davey 070799	R-Squared Va	See Attachments
Instrument Software/Version:	MassLynx 3.3, 3.4	Slope:	See Attachments
Date of Extraction/Analyst:	2/29/00 SAL	Y-Intercept:	See Attachments
Date of Analysis/Analyst:	05/08/00, 05/24/00, 08/21/00, 08/23/00 IAS/HOJ/MMH		
Date of Data Reduction/Analyst:	05/09/00, 05/25/00, 08/22/00, 08/28/00 IAS/MMH/HOJ		

Sample Data

WEEK 53 RAT LIVER Lot NB113047-80

Group Dose	Sample #	M556 Purity Correction Factor	Surrogate Verified	M556 Conc. ng/g	M556 Dilution Factor	M556 Calc. Conc. ng/g	Filename	Concentration of M556 ug/g or % Rec.	Mean M556 ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL02290-H2O Blk-5	Unknown	NA	0.00	1	0.00	D050800004	<LOQ (0.0308 ug/g)		NA
	RBL02290-H2O Blk-6	Unknown	NA	0.00	1	0.00	D050800017	<LOQ (0.0308 ug/g)	<LOQ	NA
Matrix Blk	RBL02290-Liver Blk-5	Unknown	NA	0.00	1	0.00	D050800005	<LOQ (0.0308 ug/g)		NA
	RBL02290-Liver Blk-6	Unknown	NA	0.00	1	0.00	D050800018	<LOQ (0.0308 ug/g)	<LOQ	NA
QC - 250 ppb	C90714M-250 ppb-MS-5-1	NA	Confirmed Low	296	1	294	D050800019	98%		
	C90714M-250 ppb-MS-5-2	NA	Confirmed Low	336	1	333	D050800020	112%	105%	13%
Group 1	C90714M	Unknown	NA	0.00	1	0.00	D050800023	<LOQ (0.0308 ug/g)		
Control	C90751M	Unknown	NA	37.4	1	37.3	D050800024	0.0373		
0.0 mg/kg	C90752M	Unknown	NA	0.00	1	0.00	D050800025	<LOQ (0.0308 ug/g)		
	C90754M	Unknown	NA	29.0	1	29.3	D050800026	<LOQ (0.0308 ug/g)		NA
	C90767M	Unknown	NA	8.62	1	8.76	D050800027	<LOQ (0.0308 ug/g)	<LOQ	NA
Group 1	C91121F	Unknown	NA	5.14	1	5.15	D050800030	<LOQ (0.0308 ug/g)		
Control	C91122F	Unknown	NA	0.00	1	0.00	D050800031	<LOQ (0.0308 ug/g)		
0.0 mg/kg	C91124F	Unknown	NA	10.9	1	10.8	D050800032	<LOQ (0.0308 ug/g)		
	C91133F	Unknown	NA	8.22	1	8.29	D050800033	<LOQ (0.0308 ug/g)		NA
	C91152F	Unknown	NA	7.79	1	7.67	D050800034	<LOQ (0.0308 ug/g)	<LOQ	NA
Group 4	C90902M	Unknown	NA	860	100	86649	D050800051	86.6		
Low Dose	C90918M	Unknown	NA	591	100	58978	D050800052	59.0		
3.0 mg/kg	C90950M	Unknown	NA	546	100	55321	D050800053	55.3		
	C90951M	Unknown	NA	409	100	40710	D050800054	40.7		28.8
	C90957M	Unknown	NA	522	100	52862	D050800055	52.9	58.9	17.0
Group 4	C91323F	Unknown	NA	395	100	39739	D050800058	39.7		
Low Dose	C91347F	Unknown	NA	297	100	29927	D050800059	29.9		
3.0 mg/kg	C91352F	Unknown	NA	292	100	29267	D050800060	29.3		
	C91367F	Unknown	NA	307	100	30992	D050800061	31.0		26.2
	C91371F	Unknown	NA	510	100	51513	D050800062	51.5	36.3	9.51

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Date Entered/Analyst:	05/15/00, 06/12/00, 09/03/00 CSH/LAC
Date Verified/Analyst:	06/05/00. 06/06/00 CSH: 02/26/01 hoj
Purity Entered/Verified:	02/16/01 LAC

Corrected PFOS LOQ (0.0613 ug/g) to include std correction factors
new LOQ is 0.0491 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename:	See Below
Analytical Equipment System Number:	Davey 070799	R-Squared Value:	See Attachments
Instrument Software/Version:	MassLynx 3.3, 3.4	Slope:	See Attachments
Date of Extraction/Analyst:	2/29/00 SAL	Y-Intercept:	See Attachments
Date of Analysis/Analyst:	05/08/00, 05/24/00, 08/21/00, 08/23/00 IAS/HOJ/MMH		
Date of Data Reduction/Analyst:	05/09/00, 05/25/00, 08/22/00, 08/28/00 IAS/MMH/HOJ		

Sample Data

WEEK 53 RAT LIVER lot 529

Group Dose	Sample #	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Conc. ng/g	PFOSEA Dilution Factor	PFOSEA Calc. Conc. ng/g	Filename	Concentration of PFOSEA ug/g or % Rec.	Mean PFOSEA ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL02290-H2O Blk-5	Unknown	NA	NR	1	NR	NA	NR		
	RBL02290-H2O Blk-6	Unknown	NA	NR	1	NR	NA	NR	NR	NA
Matrix Blk	RBL02290-Liver Blk-5	Unknown	NA	NR	1	NR	NA	NR		
	RBL02290-Liver Blk-6	Unknown	NA	NR	1	NR	NA	NR	NR	NA
QC - 250 ppb	C90714M-250 ppb-MS-5-1	NA	NA	NR	1	NR	NA	NR		
	C90714M-250 ppb-MS-5-2	NA	NA	NR	1	NR	NA	NR	NR	NA
Group 1	C90714M	Unknown	NA	NR	1	NR	NA	NR		
Control	C90751M	Unknown	NA	NR	1	NR	NA	NR		
0.0 mg/kg	C90752M	Unknown	NA	NR	1	NR	NA	NR		
	C90754M	Unknown	NA	NR	1	NR	NA	NR		NA
	C90767M	Unknown	NA	NR	1	NR	NA	NR	NR	NA
Group 1	C91121F	Unknown	NA	NR	1	NR	NA	NR		
Control	C91122F	Unknown	NA	NR	1	NR	NA	NR		
0.0 mg/kg	C91124F	Unknown	NA	NR	1	NR	NA	NR		
	C91133F	Unknown	NA	NR	1	NR	NA	NR		NA
	C91152F	Unknown	NA	NR	1	NR	NA	NR	NR	NA
Group 4	C90902M	Unknown	NA	NR	1	NR	NA	NR		
Low Dose	C90918M	Unknown	NA	NR	1	NR	NA	NR		
3.0 mg/kg	C90950M	Unknown	NA	NR	1	NR	NA	NR		
	C90951M	Unknown	NA	NR	1	NR	NA	NR		NA
	C90957M	Unknown	NA	NR	1	NR	NA	NR	NR	NA
Group 4	C91323F	Unknown	NA	NR	1	NR	NA	NR		
Low Dose	C91347F	Unknown	NA	NR	1	NR	NA	NR		
3.0 mg/kg	C91352F	Unknown	NA	NR	1	NR	NA	NR		
	C91367F	Unknown	NA	NR	1	NR	NA	NR		NA
	C91371F	Unknown	NA	NR	1	NR	NA	NR	NR	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Date Entered/Analyst: 05/15/00, 06/12/00, 09/03/00 CSH/LAC
Date Verified/Analyst: 06/05/00. 06/06/00 CSH: 02/26/01 hoj
Purity Entered/Verified: 02/16/01 LAC

Corrected PFOS LOQ (0.0613 ug/g) to include std correction factors
new LOQ is 0.0491 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename:	See Below
Analytical Equipment System Number:	Davey 070799	R-Squared	See Attachments
Instrument Software/Version:	MassLynx 3.3, 3.4	Slope:	See Attachments
Date of Extraction/Analyst:	2/29/00 SAL	Y-Intercept	See Attachments
Date of Analysis/Analyst:	05/08/00, 05/24/00, 08/21/00, 08/23/00 IAS/HOJ/MMH		
Date of Data Reduction/Analyst:	05/09/00, 05/25/00, 08/22/00, 08/28/00 IAS/MMH/HOJ		

Sample Data

WEEK 53 RAT LIVER

Group Dose	Sample #	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL02290-H2O Blk-5	<LOQ (0.00491 ug/g)		NA	<LOQ (0.0123 ug/g)		NA	<LOQ (0.0307 ug/g)		NA
	RBL02290-H2O Blk-6	<LOQ (0.00491 ug/g)	<LOQ	NA	<LOQ (0.0123 ug/g)	<LOQ	NA	<LOQ (0.0307 ug/g)	<LOQ	NA
Matrix Blk	RBL02290-Liver Blk-5	<LOQ (0.00491 ug/g)		NA	<LOQ (0.0123 ug/g)		NA	<LOQ (0.0307 ug/g)		NA
	RBL02290-Liver Blk-6	<LOQ (0.00491 ug/g)	<LOQ	NA	<LOQ (0.0123 ug/g)	<LOQ	NA	<LOQ (0.0307 ug/g)	<LOQ	NA
QC - 250 ppb	C90714M-250 ppb-MS-5-1	177% *			77%			109%		
	C90714M-250 ppb-MS-5-2	100%	138%	56%	95%	86%	21%	120%	114%	9%
Group 1	C90714M	0.560			<LOQ (0.00614 ug/g)			<LOQ (0.0307 ug/g)		
Control	C90751M	0.378			0.0354			0.05		
0.0 mg/kg	C90752M	0.796			0.0117			<LOQ (0.0307 ug/g)		
	C90754M	0.693		30.7	0.0183		55.1	0.04		NA
	C90767M	0.428	0.571	0.175	0.0196	0.0182	0.0100	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 1	C91121F	0.301			0.0125			<LOQ (0.0307 ug/g)		
Control	C91122F	0.305			<LOQ (0.00614 ug/g)			<LOQ (0.0307 ug/g)		
0.0 mg/kg	C91124F	0.429			0.0253			<LOQ (0.0307 ug/g)		
	C91133F	0.540		25.5	0.0159		48.7	<LOQ (0.0307 ug/g)		NA
	C91152F	0.374	0.390	0.0993	0.00971	0.0139	0.00677	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 4	C90902M	597			29.8			80.9		
Low Dose	C90918M	438			19.6			40.1		
3.0 mg/kg	C90950M	924			29.3			68.6		
	C90951M	699		27.2	21.5		29.9	28.5		38.2
	C90957M	818	695	189	40.9	28.2	8.44	60.7	55.7	21.3
Group 4	C91323F	661			23.7			49.4		
Low Dose	C91347F	861			21.8			41.2		
3.0 mg/kg	C91352F	770			21.9			45.2		
	C91367F	627		17.7	26.7		17.3	46.1		11.9
	C91371F	549	694	123	32.2	25.3	4.37	56.3	47.6	5.67

* High recovery was confirmed on 8/21/00, data was not entered because it was outside the calibration range. 4/20/01 mmh

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PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Date Entered/Analyst:	05/15/00, 06/12/00, 09/03/00 CSH/LAC
Date Verified/Analyst:	06/05/00. 06/06/00 CSH: 02/26/01 hoj
Purity Entered/Verified:	02/16/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename:	See Below
Analytical Equipment System Number:	Davey 070799	R-Squared Value:	See Attachments
Instrument Software/Version:	MassLynx 3.3, 3.4	Slope:	See Attachments
Date of Extraction/Analyst:	2/29/00 SAL	Y-Intercept:	See Attachments
Date of Analysis/Analyst:	05/08/00, 05/24/00, 08/21/00, 08/23/00 IAS/HOJ/MMH		
Date of Data Reduction/Analyst:	05/09/00, 05/25/00, 08/22/00, 08/28/00 IAS/MMH/HOJ		

Sample Data

WEEK 53 RAT LIVER

Group Dose	Sample #	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD	Concentration of M556 ug/g or % Rec.	Mean M556 ug/g	RSD Std Dev MS/MSD RPD	Concentration of PFOSEA ug/g or % Rec.	Mean PFOSEA ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL02290-H2O Blk-5	NR			<LOQ (0.0308 ug/g)		NA	NR		
	RBL02290-H2O Blk-6	NR	NR	NA	<LOQ (0.0308 ug/g)	<LOQ	NA	NR	NR	NA
Matrix Blk	RBL02290-Liver Blk-5	NR			<LOQ (0.0308 ug/g)		NA	NR		
	RBL02290-Liver Blk-6	NR	NR	NA	<LOQ (0.0308 ug/g)	<LOQ	NA	NR	NR	NA
QC - 250 ppb	C90714M-250 ppb-MS-5-1	NR			98%			NR		
	C90714M-250 ppb-MS-5-2	NR	NR	NA	112%	105%	13%	NR	NR	NA
Group 1	C90714M	NR			<LOQ (0.0308 ug/g)			NR		
Control	C90751M	NR			0.0373			NR		
0.0 mg/kg	C90752M	NR			<LOQ (0.0308 ug/g)			NR		
	C90754M	NR		NA	<LOQ (0.0308 ug/g)		NA	NR		NA
	C90767M	NR	NR	NA	<LOQ (0.0308 ug/g)	<LOQ	NA	NR	NR	NA
Group 1	C91121F	NR			<LOQ (0.0308 ug/g)			NR		
Control	C91122F	NR			<LOQ (0.0308 ug/g)			NR		
0.0 mg/kg	C91124F	NR			<LOQ (0.0308 ug/g)			NR		
	C91133F	NR		NA	<LOQ (0.0308 ug/g)		NA	NR		NA
	C91152F	NR	NR	NA	<LOQ (0.0308 ug/g)	<LOQ	NA	NR	NR	NA
Group 4	C90902M	NR			86.6			NR		
Low Dose	C90918M	NR			59.0			NR		
3.0 mg/kg	C90950M	NR			55.3			NR		
	C90951M	NR		NA	40.7		28.8	NR		NA
	C90957M	NR	NR	NA	52.9	58.9	17.0	NR	NR	NA
Group 4	C91323F	NR			39.7			NR		
Low Dose	C91347F	NR			29.9			NR		
3.0 mg/kg	C91352F	NR			29.3			NR		
	C91367F	NR		NA	31.0		26.2	NR		NA
	C91371F	NR	NR	NA	51.5	36.3	9.51	NR	NR	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide

Date Entered/Analyst: 05/15/00, 06/12/00, 09/03/00 CSH/LAC
Date Verified/Analyst: 06/05/00. 06/06/00 CSH: 02/26/01 hoj
Purity Entered/Verified: 02/16/01 LAC

Corrected PFOS LOQ (0.0613 ug/g) to include std correction factors
new LOQ is 0.0491 ug/g. LAC 02/19/01

AMDT# 092597.1
Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver		
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	Filename	See Below
Analytical Equipment System Number:	Amelia 062498, Davey 070799	R-Squared Value	See Attachments
Instrument Software/Version:	MassLynx 3.3	Slope	See Attachments
Date of Extraction/Analyst:	02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS	Y-Intercept	See Attachments
Date of Analysis/Analyst:	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 03/27/00, 04/24/00, 04/25/00, 04/26/00, 08/15/00, 03/22/01 MMH/MEE/IAS/SAH		Box 00-012, 00-013
Date of Data Reduction/Analyst:	02/28/00, 03/01/00, 03/03/00, 03/06/00, 04/17/00, 04/24/00, 04/25/00, 04/26/00, 04/27/00, 08/17/00, 03/23/01 IAS/MEE/MMH/ADV		

Sample Data

WEEK 105 RAT LIVER

Lot 171

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor	Surrogate Verified	PFOS Conc. ng/g	PFOS Dilution Factor	PFOS Calc. Conc. ng/g	Filename	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL021400-WBlk-1	1.0000	NA	0.9275	0.8640	NA	0.00	1	0.00	D042600015	<LOQ (0.0245 ug/g)		
	RBL021400-WBlk-2	1.0000	NA	0.9275	0.8640	NA	0.00	1	0.00	D042600016	<LOQ (0.0245 ug/g)	<LOQ	NA
	RBL021600-WBlk-5	1.0000	NA	0.9275	0.8640	NA	0.00	1	0.00	A030100003	<LOQ (0.0245 ug/g)		
	RBL021600-WBlk-6	1.0000	NA	0.9275	0.8640	NA	0.00	1	0.00	D042600018	<LOQ (0.0245 ug/g)	<LOQ	NA
	RBL031501-WBlk-1	1.0000	NA	1.0000	1.0000	NA	0.00	1	0.00	A010322005	<LOQ (0.0613 ug/g)		
	RBL031501-WBlk-2	1.0000	NA	1.0000	1.0000	High - Not Confirmed	0.00	1	0.00	A010322019	<LOQ (0.0613 ug/g)	<LOQ	NA
Matrix Blk	RBL021400-LBlk-1	1.0000	40.13	0.9275	0.8640	NA	0.00	1	0.00	D042600019	<LOQ (0.0245 ug/g)		
	RBL021400-LBlk-2	1.0000	40.13	0.9275	0.8640	NA	0.00	1	0.00	D042600022	<LOQ (0.0245 ug/g)	<LOQ	NA
	RBL021600-LBlk-5	1.0000	40.13	0.9275	0.8640	NA	0.00	1	0.00	A030100004	<LOQ (0.0245 ug/g)		
	RBL021600-LBlk-6	1.0000	40.13	0.9275	0.8640	NA	0.00	1	0.00	D042600024	<LOQ (0.0245 ug/g)	<LOQ	NA
	RBL031501-LBlk-1	1.0000	NA	1.0000	1.0000	NA	0.00	1	0.00	A010322006	<LOQ (0.0613 ug/g)		
	RBL031501-LBlk-2	1.0000	NA	1.0000	1.0000	NA	0.00	1	0.00	A010322020	<LOQ (0.0613 ug/g)	<LOQ	NA
	RTL031501-LBlk-1	1.0000	NA	1.0000	1.0000	NA	12.1	1	12.1	A010322007	<LOQ (0.0613 ug/g)		
	RTL031501-LBlk-2	1.0000	NA	1.0000	1.0000	NA	16.3	1	16.3	A010322021	0.0163	<LOQ	NA
QC - 250 ppb	C90716M-MS-1	1.0187	NA	NA	NA	NA	291	1	262	A022100016	89%		
	C90716M-MS-2	1.0187	NA	NA	NA	NA	356	1	327	A022100017	111%	100%	22%
	C90722M-MSD-1	1.0324	NA	NA	NA	NA	395	1	206	A022100018	71%		
	C90722M-MSD-2	1.0324	NA	NA	NA	NA	469	1	278	A022100019	95%	83%	30%
	C90712M-MS-5	1.0080	NA	NA	NA	NA	312	1	287	A030400016	96%		
	C90712M-MSD-5	1.0080	NA	NA	NA	NA	332	1	307	A030400017	103%	100%	7%
	C90726M-MS-6	1.0035	NA	NA	NA	NA	250	1	180	A030400018	60%		
	C90726M-MSD-6	1.0035	NA	NA	NA	NA	356	1	285	A030400019	95%	78%	45%
	C91157F-MS-5	0.9882	NA	NA	NA	NA	659	1	441	A081500017	146%		
	C91157F-MSD-5	0.9882	NA	NA	NA	NA	628	1	410	A081500018	136%	141%	7%
	RTL031501-Liver MS	1.0000	NA	NA	NA	NA	374	1	360	A010322022	114%		
	RTL031501-Liver MSD	1.0000	NA	NA	NA	NA	316	1	301	A010322023	96%	105%	18%
Group 1 Control 0.0 mg/kg	C90712M-Blk-5	1.0080	NA	0.9275	0.8640	NA	23.2	1	18.5	D042600025	<LOQ (0.0245 ug/g)		
	C90712M-Blk-6	1.0080	NA	0.9275	0.8640	NA	30.1	1	23.9	D042600026	<LOQ (0.0245 ug/g)		
	C90716M-1	1.0187	NA	0.9275	0.8640	NA	29.9	1	23.5	D042600029	<LOQ (0.0245 ug/g)		
	C90716M-2	1.0187	NA	0.9275	0.8640	NA	50.6	1	39.8	D042600030	0.0398		
	C90722M-1	1.0324	NA	0.9275	0.8640	NA	182	1	141	A032700024	0.141		
	C90722M-2	1.0324	NA	0.9275	0.8640	NA	180	1	140	A032700025	0.140		
	C90724M	1.0503	NA	0.9275	0.8640	NA	380	1	290	A032700026	0.290		
	C90726M-Blk-5	1.0035	NA	0.9275	0.8640	NA	69.3	1	55.3	D042600036	0.0553		
	C90726M-Blk-6	1.0035	NA	0.9275	0.8640	NA	72.1	1	57.6	D042600037	0.0576		
	C90728M	1.0033	NA	0.9275	0.8640	NA	261	1	208	A032700029	0.208		
	C90735M	1.0631	NA	0.9275	0.8640	NA	172	1	130	A032700030	0.130		
	C90742M	1.0954	NA	0.9275	0.8640	NA	431	1	315	A032700031	0.315		
	C90743M	0.9865	NA	0.9275	0.8640	NA	27.1	1	22.0	D042800043	<LOQ (0.0245 ug/g)		
	C90747M	0.9921	NA	0.9275	0.8640	NA	480	1	387	A032700032	0.387		
	C90750M	1.0012	NA	0.9275	0.8640	NA	520	1	417	A032700054	0.417		
	C90758M	0.9884	NA	0.9275	0.8640	NA	53.1	1	43.0	D042800046	0.0430		
	C90765M	1.0717	NA	0.9275	0.8640	NA	26.5	1	19.8	D042800047	<LOQ (0.0245 ug/g)		93.5
	C90778M	0.9860	NA	0.9275	0.8640	NA	276	1	224	D042800050	0.224	0.143	0.133
Group 1 Control 0.0 mg/kg	C91127F	1.0644	NA	0.9275	0.8640	NA	235	1	177	A032700036	0.177		
	C91131F	1.0303	NA	0.9275	0.8640	NA	549	1	427	A032700037	0.427		
	C91138F	1.0158	NA	0.9275	0.8640	NA	69.8	1	55.1	A032700064	0.0551		
	C91144F	1.0169	NA	0.9275	0.8640	NA	273	1	215	A032700056	0.215		
	C91148F	1.0069	NA	0.9275	0.8640	NA	147	1	117	A032700038	0.117		
	C91157F-Blk-5	0.9882	NA	0.9275	0.8640	NA	223	1	181	A032700059	0.181		
	C91157F-Blk-6	0.9882	NA	0.9275	0.8640	NA	224	1	181	A032700060	0.181		
	C91159F	0.9986	NA	0.9275	0.8640	NA	394	1	317	A032700039	0.317		
	C91166F	0.9455	NA	0.9275	0.8640	NA	64.1	1	54.3	A032700061	0.0543		
	C91172F	1.0766	NA	0.9275	0.8640	NA	300	1	223	A032700040	0.223		
	C91176F	1.0829	NA	0.9275	0.8640	NA	76.5	1	5.66	D042800065	<LOQ (0.0245 ug/g)		
	C91183F	1.0424	NA	0.9275	0.8640	NA	267	1	205	A032700044	0.205		
	C91188F	1.0142	NA	0.9275	0.8640	NA	308	1	243	A032700062	0.243		62.5
	C91190F	1.0073	NA	0.9275	0.8640	NA	81.7	1	65.0	D042800068	0.0650	0.178	0.111
Group 2 Low Dose 3.0 mg/kg	C90781M	1.0802	NA	0.9275	0.8640	Low, Not Confirmed	201	20	2977	A022900032	2.98		
	C90782M	0.9809	NA	0.9275	0.8640	2nd Analysis OK	368	5	1504	A022900065	1.50		
	C90783M	0.9895	NA	0.9275	0.8640	Low, Not Confirmed	488	20	7907	A022900034	7.91		
	C90785M	1.0134	NA	0.9275	0.8640	NA	471	1	373	A032700068	0.373		
	C90787M	0.9997	NA	0.9275	0.8640	2nd Analysis OK	124	5	497	A022900068	0.497		
	C90788M	0.9836	NA	0.9275	0.8640	Low, Not Confirmed	746	40	24309	A081500053	24.3		
	C90791M	0.9904	NA	0.9275	0.8640	Low, Not Confirmed	153	100	12394	A081500056	12.4		
	C90794M	1.0030	NA	0.9275	0.8640	Low, Not Confirmed	258	40	8255	A081500057	8.26		
	C90799M	1.0431	NA	0.9275	0.8640	Low, Not Confirmed	912	20	14019	A022900044	14.0		
	C90804M	0.9900	NA	0.9275	0.8640	Low, Not Confirmed	876	20	14183	A022900045	14.2		
	C90809M	1.0083	NA	0.9275	0.8640	Low, Not Confirmed	371	20	5897	A022900046	5.90		
	C90810M	1.0099	NA	1.0000	1.0000	High - Not confirmed	54.4	20	1077	A010322027	1.08	*	
	C90811M	1.0613	NA	0.9275	0.8640	2nd Analysis OK	360	5	1358	A022900080	1.36		
	C90812M	1.0107	NA	0.9275	0.8640	Low, Not Confirmed	94.2	40	2987	A081500065	2.99		
	C90815M	1.1100	NA	0.9275	0.8640	Low, Not Confirmed	394	20	5689	A022900052	5.69		
	C90823M	1.1067	NA	0.9275	0.8640	Low, Not Confirmed	237	100	17157	A081500067	17.2		
	C90829M	0.9971	NA	0.9275	0.8640	Low, Not Confirmed	640	20	10279	A022900053	10.3		
	C90830M	1.0073	NA	0.9275	0.8640	Low, Not Confirmed	670	40	21330	A081500071	21.3		
	C90832M	0.9861	NA	0.9275	0.8640	2nd Analysis OK	140	1	114	A032700076	0.114		
	C90835M	1.0032	NA	0.9275	0.8640	2nd Analysis OK	589	20	9405	A022900059	9.41		
	[illegible]	0.9891	NA	0.9275	0.8640	Low, Not Confirmed	108	20	2721	A022900060	2.72		93.9
	C90840M	0.9871	NA	0.9275	0.8640	Low, Not Confirmed	123	20	1990	A022900061	1.99	7.56	7.10
Group 2 Low Dose 3.0 mg/kg	C91197F	1.0489	NA	0.9275	0.8640	2nd Analysis OK	337	100	25746	A022900016	25.7		
	C91201F	1.0319	NA	0.9275	0.8640	Low, Not Confirmed	335	100	26030	A022900017	26.0		
	C91207F	1.0045	NA	0.9275	0.8640	Low, Not Confirmed	674	100	53763	A022900018	53.8		
	C91211F	0.9796	NA	0.9275	0.8640	Low, Not Confirmed	398	100	32550	A022900019	32.6		
	C91216F	0.9828	NA	0.9275	0.8640	Low, Not Confirmed	873	100	71153	A022900020	71.2		
	C91220F	0.9870	NA	0.9275	0.8640	NA	257	100	20899	A030400038	20.9		
	C91221F	1.0088	NA	0.9275	0.8640	Low, Not Confirmed	488	100	38750	A022900023	38.8		
	C91227F	1.0088	NA	0.9275	0.8640	Low, Not Confirmed	158	100	12540	A022900024	12.5		
	C91232F	0.9945	NA	0.9275	0.8640	Low, Not Confirmed	408	100	32853	A022900025	32.9		
	C91235F	1.0062	NA	0.9275	0.8640	Low, Not Confirmed	307	100	24425	A022900026	24.4		
	C91236F	1.0914	NA	0.9275	0.8640	Low, Not Confirmed	141	200	20754	A081500044	20.8		
	C91242F	1.0070	NA	0.9275	0.8640	Low, Not Confirmed	687	100	54632	A022900030	54.6		56.9
	C91243F	1.0080	NA	0.9275	0.8640	Low, Not Confirmed	1092	100	86844	A022900031	86.8	38.5	21.9

* Sample was reextracted 03/15/01, analyzed 03/22/01, and the PFOS concentration reported from 03/15/01 extraction. LAC 03/26/01

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 04/27/00, 05/17/00, 05/18/00, 09/03/00, 03/26/01, 04/19/01 LAC/CSH
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0.0245 ug/g LAC 02/19/01

ETS-8-7.0
Excel 97

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98 1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number/Test Substance) T-6316 (EPOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6 0 & ETS-8-7 0
Analytical Equipment System Number: Amelia 062498, Davey 070799
Instrument Software/Version: MassLynx 3 3
Date of Extraction/Analyst: 02/14/00, 02-16/00, 03/15-01 RWW SAL/KJS
Date of Analysis/Analyst: 02/21 00, 02-29/00, 03/01 00, 03/03/00, 03/04/00, 03/27/00, 04/24/00, 04/25/00, 04/26/00, 05/15/00, 03-22/01 MMH/MEE/IAS/SAH
Date of Data Reduction/Analyst: 02/28 00, 03/01 00, 03-03/00, 03/06/00, 04/17/00, 04/24/00, 04/25/00, 04/26/00, 04/27/00, 05/17/00, 03/23/01 IAS/MEE/MMH/ADV
Sample Data

WEEK 105 RAT LIVER

Group Dose	Sample #	PFOSA Purity Correction Factor	Surrogate Verified	PFOSA Conc. ug/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ug/g	Filename	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD	PFOSAA Purity Correction Factor	Surrogate Verified	PFOSAA Conc. ug/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ug/g	Filename	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD
	lot L-15709										lot T-7121 1								
Method Blk	RBL021400-WBlk-1	Unknown	NA	0 00	1	0.00	D042600015	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0.00	A022100003	<LOQ (0.0307 ug/g)		
	RBL021400-WBlk-2	Unknown	NA	0.00	1	0 00	D042600016	<LOQ (0 0123 ug/g)	<LOQ	NA	Unknown	NA	0 00	1	0 00	D042400015	<LOQ (0 00615 ug/g)	<LOQ	NA
	RBL021600-WBlk-5	Unknown	NA	4 17	1	4 17	A030100003	<LOQ (0.0123 ug/g)			Unknown	NA	0 00	1	0 00	A030100003	<LOQ (0 0123 ug/g)		
	RBL021600-WBlk-6	Unknown	NA	1 16	1	1.16	A030400003	<LOQ (0 00614 ug/g)	<LOQ	NA	Unknown	NA	0 00	1	0.00	A030400003	<LOQ (0 0307 ug/g)	<LOQ	NA
	RBL031501-WBlk-1	NA	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA	NA		
	RBL031501-WBlk-2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Matrix Blk	RBL021400-LBlk-1	Unknown	NA	0 00	1	0.00	D042600019	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0 00	A022100004	<LOQ (0.0307 ug/g)		
	RBL021400-LBlk-2	Unknown	NA	0 00	1	0.00	D042600022	<LOQ (0 0123 ug/g)	<LOQ	NA	Unknown	NA	0 00	1	0 00	D042400023	<LOQ (0.00615 ug/g)	<LOQ	NA
	RBL021600-LBlk-5	Unknown	NA	1 89	1	1.89	A030100004	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0 00	A030100004	<LOQ (0.0123 ug/g)		
	RBL021600-LBlk-6	Unknown	NA	0.670	1	0.670	A030400004	<LOQ (0 00614 ug/g)	<LOQ	NA	Unknown	NA	0 00	1	0.00	A030400004	<LOQ (0 0307 ug/g)	<LOQ	NA
	RBL031501-LBlk-1	NA	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA	NA		
	RBL031501-LBlk-2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	RTL031501-LBlk-1	NA	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA	NA		
	RTL031501-LBlk-2	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
QC - 250 ppb	C90716M-MS-1	NA	NA	333	1	328	A022100016	111%			NA	NA	281	1	250	A022100016	84%		
	C90716M-MS-2	NA	NA	279	1	274	A022100017	92%	102%	18%	NA	NA	380	1	356	A022100017	120%	102%	35%
	C90722M-MSD-1	NA	NA	315	1	303	A022100018	104%			NA	NA	328	1	264	A022100018	90%		
	C90722M-MSD-2	NA	NA	326	1	313	A022100019	107%	105%	3%	NA	NA	343	1	281	A022100019	96%	93%	6%
	C90712M-MS-5	NA	NA	374	1	371	A030400016	124%			NA	NA	303	1	275	A030400016	92%		
	C90712M-MSD-5	NA	NA	349	1	346	A030400017	116%	120%	7%	NA	NA	311	1	282	A030400017	94%	93%	3%
	C90726M-MS-6	NA	NA	385	1	382	A030400018	128%			NA	NA	276	1	275	A030400018	92%		
	C90726M-MSD-6	NA	NA	260	1	257	A030400019	86%	107%	39%	NA	NA	328	1	327	A030400019	109%	100%	17%
	C91157F-MS-5	NA	2nd Analysis OK	334	1	316	A081500017	111%			NA	2nd Analysis OK	442	1	447	A081500017	147%		
	C91157F-MSD-5	NA	2nd Analysis OK	353	1	333	A081500018	117%	114%	5%	NA	2nd Analysis OK	387	1	392	A081500018	129%	138%	13%
	RTL031501-Liver MS	NA	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA	NA		
	RTL031501-Liver MSD	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 1 Control 0 0 mg/kg	C90712M-Blk-5	Unknown	NA	0.460	1	0.458	D042800025	<LOQ (0.0123 ug/g)			Unknown	NA	26.7	1	26 5	A032700047	<LOQ (0.0615 ug/g)		
	C90712M-Blk-6	Unknown	NA	4.11	1	4 08	D042800026	<LOQ (0 0123 ug/g)			Unknown	NA	0.00	1	0.00	A032700048	<LOQ (0.0615 ug/g)		
	C90716M-1	Unknown	NA	2 46	1	2 41	D042800029	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0 00	A022100022	<LOQ (0.0307 ug/g)		
	C90716M-2	Unknown	NA	5 05	1	4.96	D042800030	<LOQ (0.0123 ug/g)			Unknown	NA	31 8	1	31 2	A022100023	0 0312		
	C90722M-1	Unknown	NA	2 76	1	2.67	D042800031	<LOQ (0 0123 ug/g)			Unknown	NA	53 0	1	51.4	A022100024	0 0514		
	C90722M-2	Unknown	NA	2 48	1	2 40	D042800032	<LOQ (0 0123 ug/g)			Unknown	NA	56 7	1	55 0	A022100025	0.0550		
	C90724M	Unknown	NA	0.630	1	0 600	D042800033	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0 00	A022100026	<LOQ (0 0307 ug/g)		
	C90726M-Blk-5	Unknown	NA	1 46	1	1.45	D042800036	<LOQ (0 0123 ug/g)			Unknown	NA	0.00	1	0 00	A032700049	<LOQ (0.0615 ug/g)		
	C90726M-Blk-6	Unknown	NA	3 05	1	3 04	D042800037	<LOQ (0.0123 ug/g)			Unknown	NA	0.00	1	0 00	A032700050	<LOQ (0.0615 ug/g)		
	C90728M	Unknown	NA	4.60	1	4.54	D042800038	<LOQ (0 0123 ug/g)			Unknown	NA	0.00	1	0 00	A022100029	<LOQ (0.0307 ug/g)		
	C90735M	Unknown	NA	0.00	1	0 00	D042800039	<LOQ (0.0123 ug/g)			Unknown	NA	0.00	1	0 00	A022100030	<LOQ (0 0307 ug/g)		
	C90742M	Unknown	NA	1 52	1	1.48	D042800040	<LOQ (0.0123 ug/g)			Unknown	NA	41 4	1	37 8	A022100031	0.0378		
	C90743M	Unknown	NA	0.00	1	0 00	D042800043	<LOQ (0 0123 ug/g)			Unknown	NA	40 7	1	41 2	A032700053	<LOQ (0.0615 ug/g)		
	C90747M	Unknown	NA	76 1	1	76 7	A022100032	0 0767			Unknown	NA	63 7	1	64.2	A022100032	0 0642		
	C90750M	Unknown	NA	1.69	1	1.69	D042800045	<LOQ (0.0123 ug/g)			Unknown	NA	21 3	1	21.3	A032700054	<LOQ (0.0615 ug/g)		
	C90758M	Unknown	NA	3 14	1	3.18	D042800046	<LOQ (0 0123 ug/g)			Unknown	NA	51 5	1	52.1	A032700055	<LOQ (0.0615 ug/g)		
	C90765M	Unknown	NA	0.560	1	0.523	D042800047	<LOQ (0 0123 ug/g)		NA	Unknown	NA	0.00	1	0 00	A022100033	<LOQ (0.0307 ug/g)		37 8
	C90776M	Unknown	NA	0.00	1	0 00	D042800050	<LOQ (0 0123 ug/g)	<LOQ	NA	Unknown	NA	0 00	1	0 00	D042500055	<LOQ (0 00615 ug/g)	0 0461	0 0174
Group 1 Control 0 0 mg/kg	C91127F	Unknown	NA	0 00	1	0.00	D042800051	<LOQ (0.0123 ug/g)			Unknown	NA	0 00	1	0.00	A022100036	<LOQ (0.0307 ug/g)		
	C91131F	Unknown	NA	0.820	1	0 80	D042800052	<LOQ (0.0123 ug/g)			Unknown	NA	0 00	1	0 00	A022100037	<LOQ (0.0307 ug/g)		
	C91134F	Unknown	NA	9.11	1	8.97	D042800053	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0.00	D042500058	<LOQ (0 00615 ug/g)		
	C91144F	Unknown	NA	9 03	1	8 88	D042800054	<LOQ (0 0123 ug/g)			Unknown	NA	50.7	1	49 9	A032700056	<LOQ (0.0615 ug/g)		
	C91148F	Unknown	NA	0.00	1	0.00	D042800057	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0 00	A022100038	<LOQ (0 0307 ug/g)		
	C91157F-Blk-5	Unknown	NA	1 79	1	1 81	D042800058	<LOQ (0.0123 ug/g)			Unknown	NA	0 00	1	0.00	A032700059	<LOQ (0.0615 ug/g)		
	C91157F-Blk-6	Unknown	NA	4 01	1	4 06	D042800059	<LOQ (0 0123 ug/g)			Unknown	NA	25.5	1	25 8	A032700060	<LOQ (0.0615 ug/g)		
	C91158F	Unknown	NA	4 38	1	4.39	D042800060	<LOQ (0 0123 ug/g)			Unknown	NA	41.3	1	41.4	A022100039	0.0414		
	C91166F	Unknown	NA	0 00	1	0 00	D042800061	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0.00	A032700061	<LOQ (0.0615 ug/g)		
	C91172F	Unknown	NA	13 7	1	12.8	D042800064	0.0128			Unknown	NA	0.00	1	0.00	A022100040	<LOQ (0.0307 ug/g)		
	C91176F	Unknown	NA	0 00	1	0.00	D042800065	<LOQ (0 0123 ug/g)			Unknown	NA	0 00	1	0.00	A032700043	<LOQ (0.0615 ug/g)		
	C91183F	Unknown	NA	2.02	1	1 94	D042800066	<LOQ (0.0123 ug/g)			Unknown	NA	19 4	1	18 6	A032700044	0.0186		
	C91188F	Unknown	NA	0 990	1	0.976	D042800067	<LOQ (0 0123 ug/g)		NA	Unknown	NA	0 00	1	0.00	A032700062	<LOQ (0 0615 ug/g)		45 6
	C91190F	Unknown	NA	13.9	1	13 8	D042800068	0 0138	<LOQ	NA	Unknown	NA	22 6	1	22.4	A032700063	<LOQ (0 0615 ug/g)	0 0354	0 0162
Group 2 Low Dose 3 0 mg/kg	C90781M	Unknown	NA	1003	1	929	A022100064	0 929			Unknown	2nd Analysis OK	288	5	1333	A081500078	1.335		
	C90782M	Unknown	NA	175	5	894	A022900065	0 894			Unknown	2nd Analysis OK	352	5	1796	A081500050	1 796		
	C90783M	Unknown	NA	875	1	885	A032700067	0.885			Unknown	NA	192	5	970	A022900066	0 970		
	C90785M	Unknown	NA	558	1	550	A032700068	0 550			Unknown	NA	315	1	310	A032700068	0 310		
	C90787M	Unknown	NA	378	1	378	A032700069	0.378			Unknown	NA	52 0	5	260	A022900068	0.260		
	C90788M	Unknown	NA	519	5	426	A022900069	0.426			Unknown	NA	[illegible]	[illegible]	[illegible]	[illegible]	0.088		
	C90791M	Unknown	NA	587	1	593	A032700090	0 593			Unknown	NA	210	5	1060	A042400045	1.06		
	C90794M	Unknown	NA	950	1	957	A032700070	0.957			Unknown	NA	187	5	931	A042400046	0 931		
	C90799M	Unknown	NA	752	1	721	A022100065	0 721			Unknown	NA	218	5	1043	A042400047	1.04		
	C90804M	Unknown	NA	244	1	250	A032700071	0 250			Unknown	NA	473	1	478	A032700071	0.478		
	C90809M	Unknown	NA	211	5	1045	A042400010	1 04			Unknown	NA	424	5	2103	A042400049	2 10		
	C90810M	Unknown	NA	914	1	905	A032700074	0.905			Unknown	NA	239	5	1182	A042400053	1 18		
	C90811M	Unknown	NA	413	1	389	A022100066	0 389			Unknown	NA	201	5	945	A042400054	0 945		
	C90812M	Unknown	NA	687	1	679	A022100067	0.679			Unknown	Low, Not Confirmed	129	40	5103	A081500065	5 10		
	C90815M	Unknown	NA	535	1	482	A022100068	0.482			Unknown	NA	409	5	1841	A042400055	1 84		
	C90823M	Unknown	NA	973	1	880	A032700075	0.880			Unknown	NA	886	5	4002	A042400101	4 00		
	C90829M	Unknown	NA	87 5	5	439	A022900083	0.439			Unknown	NA	234	5	1171	A042400056	1 17		
	C90830M	Unknown	NA	194	5	981	A022900086	0.981			Unknown	NA	286	5	1419	A042400057	1.42		
	C90832M	Unknown	2nd Analysis OK	74 5	20	1513	A081500079	1 51			Unknown	Low, Not Confirmed	453	20	9192	A081500079	9 19		
	C90835M	Unknown	2nd Analysis OK	160	5	800	A081500080	0.800			Unknown	NA	275	5	1371	A022900058	1.37		
	C90836M	Unknown	NA	454	1	561	A032700077	0 661		39.3	Unknown	NA	929	1	940	A032700077	0.940		106
	C90840M	Unknown	NA	997	1	1010	A032700078	1.01	0.744	0 294	Unknown	NA	542	5	2748	A030400059	2.75	1 86	2 00
Group 2 Low Dose 3.0 mg/kg	C91197F	Unknown	NA	704	1	672	A032700081	0 672			Unknown	NA	160	5	764	A030400039	0 764		
	C91201F	Unknown	NA	379	1	367	A032700082	0.367			Unknown	NA	474	1	459	A032700082	0.459		
	C91207F	Unknown	2nd Analysis OK	378	5	1470	A081500081	1.47			Unknown	NA	196	5	976	A030400044	0.976		
	C91211F	Unknown	NA	621	1	634	A032700083	0 634			Unknown	NA	174	5	889	A030400084	0 889		
	C91216F	Unknown	NA	132	5	671	A042400033	0.671			Unknown	NA	101	5	525	A030400045	0.525		
	C91220F	Unknown	NA	872	1	884	A032700085	0 884			Unknown	NA	217	5	1098	A030400046	1 10		
	C91221F	Unknown	NA	548	1	543	A032700084	0 543			Unknown	NA	117	5	580	A030400047	0 580		
	C91227F	Unknown	NA	390	1	386	A032700085	0.386			Unknown	NA	124	5	615	A030400050	0.615		
	C91232F	Unknown	NA	667	5	3350	A042400018	3 35			Unknown	NA	117	100	11723	A042400071	11 7		
	C91235F	Unknown	NA	1046	5	5197	A042400019	5.20			Unknown	NA	84 6	100	8407	A042400072	8 41		
	C91236F	Unknown	NA	628	5	3794	A042400040	3 79			Unknown	NA	68.4	100	6266	A042400073	6.27		
	C91242F	Unknown	NA	877	5	4353	A042400041	4.35		94 9	Unknown	Low, Not Confirmed	121	100	12061	A081500045	12.1		128
	C91243F	Unknown	NA	889	1	882	A032700088	0 882	1 82	1 72	Unknown	NA	203	5	1007	A030400057	1 01	3 49	4 47

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtPOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/Analyst 03 14/00, 03 16/00, 03 17/00, 04/27/00, 05 17/00, 05 18/00, 09 03/00, 03 26/01, 04 19/01 LAC/CSH
Date Verified/Analyst 03 09/01 hoj
Purity Entered/Verified 02/19/01 LAC

Corrected PFOS LOQ (0 0306 ug g) to include std correction factors
new LOQ is 0 0245 ug/g LAC 02/19/01

Study	104 Week Dietary Carcinogenicity Study with Narrow Range (98 1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix	Rat Liver
Method/Revision	ETS-8-6 0 & ETS-8-7 0
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version	MassLynx 3 3
Date of Extraction/Analyst	02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS
Date of Analysis/Analyst	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 03/27/00, 04/24/00, 04/25/00, 04/26/00, 08/15/00, 03/22/01 MMH/MEE/IAS/SAH
Date of Data Reduction/Analyst	02/28/00, 03/01/00, 03/03/00, 03/06/00, 04/17/00, 04/24/00, 04/25/00, 04/26/00, 04/27/00, 08/17/00, 03/23/01 IAS/MEE/MMH/ADV

Sample Data

WEEK 105 RAT LIVER lot Unknown lot NB 113047-80

Group Dose	Sample #	EtFOSE Purity Correction Factor	Surrogate Verified	EtFOSE Conc. ng/g	EtFOSE Dilution Factor	EtFOSE Calc. Conc. ng/g	Filename	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD	M556 Purity Correction Factor	Surrogate Verified	M556 Conc. ng/g	M556 Dilution Factor	M556 Calc. Conc. ng/g	Filename	Concentration of M556 ug/g or % Rec.	Mean M556 ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL021400-WBlk-1	Unknown	NA	0.00	1	0.00	A022100003	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A022100003	<LOQ (0.0308 ug/g)		
	RBL021400-WBlk-2	Unknown	NA	41.0	1	41.0	A032700003	<LOQ (0.0614 ug/g)	<LOQ	NA	Unknown	NA	0.00	1	0.00	D042400016	<LOQ (0.00615 ug/g)	<LOQ	NA
	RBL021600-WBlk-5	Unknown	NA	0.00	1	0.00	A030100003	<LOQ (0.123 ug/g)			Unknown	NA	0.00	1	0.00	A030100003	<LOQ (0.0123 ug/g)		
	RBL021600-WBlk-6	Unknown	NA	4.60	1	4.60	A030400003	<LOQ (0.0614 ug/g)	<LOQ	NA	Unknown	NA	0.00	1	0.00	A030400003	<LOQ (0.0615 ug/g)	<LOQ	NA
	RBL031501-WBlk-1	Unknown	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	AN	NA		
	RBL031501-WBlk-2	Unknown	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Matrix Blk	RBL021400-LBlk-1	Unknown	NA	0.00	1	0.00	A022100004	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A022100004	<LOQ (0.0308 ug/g)		
	RBL021400-LBlk-2	Unknown	NA	0.00	1	0.00	A032700004	<LOQ (0.0614 ug/g)	<LOQ	NA	Unknown	NA	0.00	1	0.00	D042400023	<LOQ (0.00615 ug/g)	<LOQ	NA
	RBL021600-LBlk-5	Unknown	NA	0.00	1	0.00	A030100004	<LOQ (0.123 ug/g)			Unknown	NA	0.00	1	0.00	A030100004	<LOQ (0.0123 ug/g)		
	RBL021600-LBlk-6	Unknown	NA	0.00	1	0.00	A030400004	<LOQ (0.0614 ug/g)	<LOQ	NA	Unknown	NA	0.00	1	0.00	A030400004	<LOQ (0.0615 ug/g)	<LOQ	NA
	RBL031501-LBlk-1	Unknown	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA	NA		
	RBL031501-LBlk-2	Unknown	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	RTL031501-LBlk-1	Unknown	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA	NA		
	RTL031501-LBlk-2	Unknown	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
QC - 250 ppb	C90716M-MS-1	NA	NA	375	1	368	A022100016	124%			NA	NA	282	1	277	A022100016	93%		
	C90716M-MS-2	NA	NA	295	1	289	A022100017	98%	111%	24%	NA	NA	301	1	295	A022100017	100%	97%	7%
	C90722M-MSD-1	NA	NA	268	1	166	A022100018	57%			NA	NA	256	1	248	A022100018	85%		
	C90722M-MSD-2	NA	NA	352	1	248	A022100019	85%	71%	40%	NA	NA	297	1	288	A022100019	98%	91%	15%
	C90712M-MS-5	NA	NA	269	1	267	A030400016	89%			NA	NA	317	1	315	A030400016	106%		
	C90712M-MSD-5	NA	NA	250	1	248	A030400017	83%	86%	7%	NA	NA	296	1	293	A030400017	99%	103%	7%
	C90726M-MS-6	NA	NA	218	1	107	A030400018	36%			NA	NA	273	1	272	A030400018	92%		
	C90726M-MSD-6	NA	NA	205	1	93.9	A030400019	31%	34%	13%	NA	NA	264	1	263	A030400019	89%	90%	4%
	C91157F-MS-5	NA	NA	322	1	244	A081500017	81%			NA	2nd Analysis OK	424	1	429	A081500017	141%		
	C91157F-MSD-5	NA	NA	159	1	78.9	A081500018	26%	53%	102%	NA	2nd Analysis OK	321	1	325	A081500018	107%	124%	27%
	RTL031501-Liver MS	NA	NA	NA	NA	NA	AN	NA			NA	NA	NA	NA	NA	NA	NA		
	RTL031501-Liver MSD	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 1 Control 0.0 mg/kg	C90712M-Blk-5	Unknown	NA	0.00	1	0.00	A022100045	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100045	<LOQ (0.0308 ug/g)		
	C90712M-Blk-6	Unknown	NA	0.00	1	0.00	A022100046	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100046	<LOQ (0.0308 ug/g)		
	C90716M-1	Unknown	NA	83.6	1	82.1	A022100022	0.0821			Unknown	NA	0.00	1	0.0000	A022100022	<LOQ (0.0308 ug/g)		
	C90716M-2	Unknown	NA	0.00	1	0.00	A022100023	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100023	<LOQ (0.0308 ug/g)		
	C90722M-1	Unknown	NA	96.6	1	93.6	A022100024	0.0936			Unknown	NA	0.00	1	0.0000	A022100024	<LOQ (0.0308 ug/g)		
	C90722M-2	Unknown	NA	128	1	124	A022100025	0.124			Unknown	NA	0.00	1	0.0000	A022100025	<LOQ (0.0308 ug/g)		
	C90724M	Unknown	NA	0.00	1	0.00	A022100026	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100026	<LOQ (0.0308 ug/g)		
	C90726M-Blk-5	Unknown	NA	110	1	110	A022100047	0.110			Unknown	NA	0.00	1	0.0000	A022100047	<LOQ (0.0308 ug/g)		
	C90726M-Blk-6	Unknown	NA	72.3	1	72.1	A022100050	0.0721			Unknown	NA	0.00	1	0.0000	A022100050	<LOQ (0.0308 ug/g)		
	C90728M	Unknown	NA	153	1	153	A022100029	0.153			Unknown	NA	0.00	1	0.0000	A022100029	<LOQ (0.0308 ug/g)		
	C90735M	Unknown	NA	58.2	1	54.7	A022100030	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100030	<LOQ (0.0308 ug/g)		
	C90742M	Unknown	NA	0.00	1	0.00	A022100031	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100031	<LOQ (0.0308 ug/g)		
	C90743M	Unknown	NA	0.00	1	0.00	A022100051	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100051	<LOQ (0.0308 ug/g)		
	C90747M	Unknown	NA	0.00	1	0.00	A022100032	<LOQ (0.0614 ug/g)			Unknown	NA	47.2	1	47.6	A022100032	0.0476		
	C90750M	Unknown	NA	123	1	122	A022100052	0.122			Unknown	NA	0.00	1	0.0000	A022100052	<LOQ (0.0308 ug/g)		
	C90758M	Unknown	NA	60.2	1	60.9	A022100053	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100053	<LOQ (0.0308 ug/g)		
	C90765M	Unknown	NA	0.00	1	0.00	A022100033	<LOQ (0.0614 ug/g)		36.1	Unknown	NA	0.00	1	0.0000	A022100033	<LOQ (0.0308 ug/g)		NA
	C90778M	Unknown	NA	22.9	1	23.2	D042800050	<LOQ (0.123 ug/g)	0.0796	0.0287	Unknown	NA	0.00	1	0.0000	D042500055	<LOQ (0.0123 ug/g)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	Unknown	NA	73.0	1	68.6	A022100036	0.0686			Unknown	NA	0.00	1	0.0000	A022100036	<LOQ (0.0308 ug/g)		
	C91131F	Unknown	NA	208	1	202	A022100037	0.202			Unknown	NA	0.00	1	0.0000	A022100037	<LOQ (0.0308 ug/g)		
	C91138F	Unknown	Confirmed High	29.6	1	29.1	D042800053	<LOQ (0.123 ug/g)			Unknown	Confirmed High	0.00	1	0.0000	D042800053	<LOQ (0.0615 ug/g)		
	C91144F	Unknown	NA	109	1	107	A022100054	0.107			Unknown	NA	0.00	1	0.0000	A022100054	<LOQ (0.0308 ug/g)		
	C91148F	Unknown	NA	117	1	117	A022100038	0.117			Unknown	NA	0.00	1	0.0000	A022100038	<LOQ (0.0308 ug/g)		
	C91157F-Blk-5	Unknown	NA	80.6	1	81.5	A022100057	0.0815			Unknown	NA	0.00	1	0.0000	A022100057	<LOQ (0.0308 ug/g)		
	C91157F-Blk-6	Unknown	NA	72.4	1	73.3	A022100058	0.0733			Unknown	NA	0.00	1	0.0000	A022100058	<LOQ (0.0308 ug/g)		
	C91159F	Unknown	NA	71.5	1	71.6	A022100039	0.0716			Unknown	NA	0.00	1	0.0000	A022100039	<LOQ (0.0308 ug/g)		
	C91166F	Unknown	NA	84.3	1	89.2	A022100059	0.0892			Unknown	NA	0.00	1	0.0000	A022100059	<LOQ (0.0308 ug/g)		
	C91172F	Unknown	NA	141	1	131	A022100040	0.131			Unknown	NA	0.00	1	0.0000	A022100040	<LOQ (0.0308 ug/g)		
	C91176F	Unknown	NA	0.00	1	0.00	A022100043	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.0000	A022100043	<LOQ (0.0308 ug/g)		
	C91183F	Unknown	NA	83.3	1	80.0	A022100044	0.0800			Unknown	NA	0.00	1	0.0000	A022100044	<LOQ (0.0308 ug/g)		
	C91188F	Unknown	NA	80.4	1	79.3	A022100060	0.0793		43.6	Unknown	NA	0.00	1	0.0000	A022100060	<LOQ (0.0308 ug/g)		NA
	C91190F	Unknown	NA	45.4	1	45.1	A022100061	0.0451	0.0906	0.0394	Unknown	NA	0.00	1	0.0000	A022100061	<LOQ (0.0308 ug/g)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90781M	Unknown	NA	0.00	1	0.00	A022100064	<LOQ (0.0614 ug/g)			Unknown	NA	121	1	112	A022100064	0.112		
	C90782M	Unknown	NA	86.6	1	88.3	A022100073	0.0883			Unknown	NA	285	1	291	A022100073	0.291		
	C90783M	Unknown	NA	57.1	1	57.7	A022100074	<LOQ (0.0614 ug/g)			Unknown	NA	597	1	604	A022100074	0.604		
	C90785M	Unknown	NA	0.00	1	0.00	A022100075	<LOQ (0.0614 ug/g)			Unknown	NA	65.3	1	64.4	A022100075	0.0644		
	C90787M	Unknown	NA	0.00	1	0.00	A022100078	<LOQ (0.0614 ug/g)			Unknown	NA	226	1	226	A022100078	0.226		
	C90788M	Unknown	NA	0.00	1	0.00	A032700089	<LOQ (0.0614 ug/g)			Unknown	NA	871	2	1771	A081500086	1.77		
	C90791M	Unknown	NA	0.00	1	0.00	A032700090	<LOQ (0.0614 ug/g)			Unknown	NA	535	1	540	A042400086	0.540		
	C90794M	Unknown	NA	60.3	1	60.1	A022100079	<LOQ (0.0614 ug/g)			Unknown	NA	402	1	400	A022100079	0.400		
	C90799M	Unknown	NA	41.1	1	39.4	A022100065	<LOQ (0.0614 ug/g)			Unknown	NA	435	1	417	A022100065	0.417		
	C90804M	Unknown	NA	0.00	1	0.00	A022100080	<LOQ (0.0614 ug/g)			Unknown	NA	195	1	197	A022100080	0.197		
	C90809M	Unknown	NA	0.00	1	0.00	A032700091	<LOQ (0.0614 ug/g)			Unknown	NA	222	5	1102	A042400049	1.102		
	C90810M	Unknown	NA	0.00	1	0.00	A022100081	<LOQ (0.0614 ug/g)			Unknown	NA	141	1	140	A022100081	0.140		
	C90811M	Unknown	NA	100	1	94.2	A022100066	0.0942			Unknown	NA	453	1	426	A022100066	0.426		
	C90812M	Unknown	NA	0.00	1	0.00	A022100067	<LOQ (0.0614 ug/g)			Unknown	NA	245	1	242	A022100067	0.242		
	C90815M	Unknown	NA	63.2	1	56.9	A022100068	0.0569			Unknown	NA	255	1	229	A022100068	0.229		
	C90823M	Unknown	NA	115	1	104	A022100071	0.104			Unknown	NA	558	1	504	A022100071	0.504		
	C90829M	Unknown	NA	52.7	1	52.8	A022100082	0.0528			Unknown	NA	495	1	497	A022100082	0.497		
	C90830M	Unknown	NA	0.00	1	0.00	A022100085	<LOQ (0.0614 ug/g)			Unknown	NA	580	1	575	A022100085	0.575		
	C90832M	Unknown	NA	50.4	1	51.1	A022100086	0.0511			Unknown	NA	659	1	668	A022100086	0.668		
	C90835M	Unknown	NA	0.00	1	0.00	A081500116	<LOQ (0.0614 ug/g)			Unknown	Confirmed High	185	1	185	A042400105	0.185		
	C90838M	Unknown	NA	0.00	1	0.00	A022100087	<LOQ (0.0614 ug/g)		20.1	Unknown	NA	196	1	198	A022100087	0.198		83.9
	C90840M	Unknown	NA	59.0	1	59.8	A022100072	0.0598	0.0649	0.0130	Unknown	NA	514	1	521	A022100072	0.521	0.450	0.378
Group 2 Low Dose 3.0 mg/kg	C91197F	Unknown	NA	0.00	1	0.00	A022100088	<LOQ (0.0614 ug/g)			Unknown	NA	480	1	457	A022100088	0.457		
	C91201F	Unknown	NA	0.00	1	0.00	A022100089	<LOQ (0.0614 ug/g)			Unknown	NA	458	1	444	A022100089	0.444		
	C91207F	Unknown	NA	99.0	1	98.5	A022100099	0.0985			Unknown	NA	798	1	795	A022100099	0.795		
	C91211F	Unknown	NA	99.1	1	101	A022100100	0.101			Unknown	NA	315	1	322	A022100100	0.322		
	C91216F	Unknown	NA	0.00	1	0.00	A022100092	<LOQ (0.0614 ug/g)			Unknown	NA	488	1	496	A022100092	0.496		
	C91220F	Unknown	NA	13.6	1	13.8	A032700095	<LOQ (0.0614 ug/g)			Unknown	NA	87.4	5	443	A030400046	0.443		
	C91221F	Unknown	NA	0.00	1	0.00	A022100093	<LOQ (0.0614 ug/g)			Unknown	NA	328	1	325	A022100093	0.325		
	C91227F	Unknown	NA	0.00	1	0.00	A022100094	<LOQ (0.0614 ug/g)			Unknown	NA	373	1	370	A022100094	0.370		
	C91232F	Unknown	NA	0.00	1	0.00	A022100101	<LOQ (0.0614 ug/g)			Unknown	NA	40.7	100	4092	A042400071	4.09		
	C91235F	Unknown	NA	47.2	1	46.9	A022100102	<LOQ (0.0614 ug/g)			Unknown	NA	60.8	100	6046	A042400072	6.05		
	C91236F	Unknown	NA	99.7	1	91.3	A022100095	0.0913			Unknown	NA	38.3	100	3512	A042400073	3.51		
	C91242F	Unknown	NA	121	1	120	A022100096	0.120		27.5	Unknown	Low, Not Confirmed	879	10	8724	A081500087	8.72		135
	C91243F	Unknown	NA	93.8	1	93.0	A022100103	0.0930	0.0766	0.0210	Unknown	NA	410	1	407	A022100103	0.407	2.03	2.74

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/Analyst	03/14/00, 03/16/00, 03/17/00, 04/27/00, 05/17/00, 05/18/00, 09/03/00, 03/26/01, 04/19/01 LAC/CSH
Date Verified/Analyst	03/09/01 hoj
Purity Entered/Verified	02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0.0245 ug/g LAC 02/19/01

Covance# 6329-212

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix	Rat Liver
Method/Revision	ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number	Amelia 062498, Davey 070799
Instrument Software/Version.	MassLynx 3.3
Date of Extraction/Analyst:	02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS
Date of Analysis/Analyst.	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 03/27/00, 04/24/00, 04/25/00, 04/26/00, 08/15/00, 03/22/01 MMH/MEE/IAS/SAH
Date of Data Reduction/Analyst:	02/28/00, 03/01/00, 03/03/00, 03/06/00, 04/17/00, 04/24/00, 04/25/00, 04/26/00, 04/27/00, 08/17/00, 03/23/01 IAS/MEE/MMH/ADV
Sample Data	

Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept.	See Attachments
	Box 00-012, 00-013

WEEK 105 RAT LIVER lot 529

Group Dose	Sample #	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Conc. ng/g	PFOSEA Dilution Factor	PFOSEA Calc. Conc. ng/g	Filename	Concentration of PFOSEA ug/g or % Rec.	Mean PFOSEA ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL021400-WBlk-1	Unknown	NA	0.00	1	0.00	A022100003	<LOQ (0.0307 ug/g)		
	RBL021400-WBlk-2	Unknown	NA	0.00	1	0.00	A032700003	<LOQ (0.0613 ug/g)	<LOQ	NA
	RBL021600-WBlk-5	Unknown	NA	0.00	1	0.00	A030100003	<LOQ (0.0307 ug/g)		
	RBL021600-WBlk-6	Unknown	NA	0.00	1	0.00	A030400003	<LOQ (0.0123 ug/g)	<LOQ	NA
	RBL031501-WBlk-1	NA	NA	NA	NA	NA	NA	NA		
	RBL031501-WBlk-2	NA	NA	NA	NA	NA	NA	NA	NA	NA
Matrix Blk	RBL021400-LBlk-1	Unknown	NA	0.00	1	0.00	A022100004	<LOQ (0.0307 ug/g)		
	RBL021400-LBlk-2	Unknown	NA	0.00	1	0.00	A032700004	<LOQ (0.0613 ug/g)	<LOQ	NA
	RBL021600-LBlk-5	Unknown	NA	0.00	1	0.00	A030100004	<LOQ (0.0307 ug/g)		
	RBL021600-LBlk-6	Unknown	NA	0.00	1	0.00	A030400004	<LOQ (0.0123 ug/g)	<LOQ	NA
	RBL031501-LBlk-1	NA	NA	NA	NA	NA	NA	NA		
	RBL031501-LBlk-2	NA	NA	NA	NA	NA	NA	NA	NA	NA
	RTL031501-LBlk-1	NA	NA	NA	NA	NA	NA	NA		
	RTL031501-LBlk-2	NA	NA	NA	NA	NA	NA	NA	NA	NA
QC - 250 ppb	C90716M-MS-1	NA	NA	353	1	347	A022100016	117%		
	C90716M-MS-2	NA	NA	324	1	318	A022100017	108%	113%	9%
	C90722M-MSD-1	NA	NA	310	1	300	A022100018	103%		
	C90722M-MSD-2	NA	NA	294	1	285	A022100019	98%	100%	5%
	C90712M-MS-5	NA	NA	274	1	272	A030400016	92%		
	C90712M-MSD-5	NA	NA	250	1	248	A030400017	84%	88%	9%
	C90726M-MS-6	NA	NA	260	1	259	A030400018	88%		
	C90726M-MSD-6	NA	NA	263	1	263	A030400019	89%	88%	1%
	C91157F-MS-5	NA	2nd Analysis OK	318	1	322	A081500017	106%		
	C91157F-MSD-5	NA	2nd Analysis OK	240	1	242	A081500018	80%	93%	28%
	RTL031501-Liver MS	NA	NA	NA	NA	NA	NA	NA		
	RTL031501-Liver MSD	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 1 Control 0.0 mg/kg	C90712M-Blk-5	Unknown	NA	0.00	1	0.00	A022100045	<LOQ (0.0307 ug/g)		
	C90712M-Blk-6	Unknown	NA	0.00	1	0.00	A022100046	<LOQ (0.0307 ug/g)		
	C90716M-1	Unknown	NA	0.00	1	0.00	A022100022	<LOQ (0.0307 ug/g)		
	C90716M-2	Unknown	NA	0.00	1	0.00	A022100023	<LOQ (0.0307 ug/g)		
	C90722M-1	Unknown	NA	0.00	1	0.00	A022100024	<LOQ (0.0307 ug/g)		
	C90722M-2	Unknown	NA	0.00	1	0.00	A022100025	<LOQ (0.0307 ug/g)		
	C90724M	Unknown	NA	0.00	1	0.00	A022100026	<LOQ (0.0307 ug/g)		
	C90726M-Blk-5	Unknown	NA	0.00	1	0.00	A022100047	<LOQ (0.0307 ug/g)		
	C90726M-Blk-6	Unknown	NA	0.00	1	0.00	A022100050	<LOQ (0.0307 ug/g)		
	C90728M	Unknown	NA	0.00	1	0.00	A022100029	<LOQ (0.0307 ug/g)		
	C90735M	Unknown	NA	0.00	1	0.00	A022100030	<LOQ (0.0307 ug/g)		
	C90742M	Unknown	NA	0.00	1	0.00	A022100031	<LOQ (0.0307 ug/g)		
	C90743M	Unknown	NA	0.00	1	0.00	A022100051	<LOQ (0.0307 ug/g)		
	C90747M	Unknown	NA	0.00	1	0.00	A022100032	<LOQ (0.0307 ug/g)		
	C90750M	Unknown	NA	0.00	1	0.00	A022100052	<LOQ (0.0307 ug/g)		
	C90758M	Unknown	NA	0.00	1	0.00	A022100053	<LOQ (0.0307 ug/g)		
	C90765M	Unknown	NA	0.00	1	0.00	A022100033	<LOQ (0.0307 ug/g)		NA
	C90778M	Unknown	NA	31.9	1	32.3	A042800050	<LOQ (0.0613 ug/g)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	Unknown	NA	0.00	1	0.00	A022100036	<LOQ (0.0307 ug/g)		
	C91131F	Unknown	NA	0.00	1	0.00	A022100037	<LOQ (0.0307 ug/g)		
	C91138F	Unknown	Confirmed High	0.00	1	0.00	A032700064	<LOQ (0.0613 ug/g)		
	C91144F	Unknown	NA	0.00	1	0.00	A022100054	<LOQ (0.0307 ug/g)		
	C91148F	Unknown	NA	0.00	1	0.00	A022100038	<LOQ (0.0307 ug/g)		
	C91157F-Blk-5	Unknown	NA	0.00	1	0.00	A022100057	<LOQ (0.0307 ug/g)		
	C91157F-Blk-6	Unknown	NA	0.00	1	0.00	A022100058	<LOQ (0.0307 ug/g)		
	C91159F	Unknown	NA	0.00	1	0.00	A022100039	<LOQ (0.0307 ug/g)		
	C91166F	Unknown	NA	0.00	1	0.00	A022100059	<LOQ (0.0307 ug/g)		
	C91172F	Unknown	NA	0.00	1	0.00	A022100040	<LOQ (0.0307 ug/g)		
	C91176F	Unknown	NA	0.00	1	0.00	A022100043	<LOQ (0.0307 ug/g)		
	C91183F	Unknown	NA	0.00	1	0.00	A022100044	<LOQ (0.0307 ug/g)		
	C91188F	Unknown	NA	0.00	1	0.00	A022100060	<LOQ (0.0307 ug/g)		NA
	C91190F	Unknown	NA	0.00	1	0.00	A022100061	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90781M	Unknown	NA	0.00	1	0.00	A022100064	<LOQ (0.0307 ug/g)		
	C90782M	Unknown	NA	0.00	1	0.00	A022100073	<LOQ (0.0307 ug/g)		
	C90783M	Unknown	NA	0.00	1	0.00	A022100074	<LOQ (0.0307 ug/g)		
	C90785M	Unknown	NA	0.00	1	0.00	A022100075	<LOQ (0.0307 ug/g)		
	C90787M	Unknown	NA	0.00	1	0.00	A022100078	<LOQ (0.0307 ug/g)		
	C90788M	Unknown	NA	0.00	1	0.00	A032700089	<LOQ (0.0613 ug/g)		
	C90791M	Unknown	NA	0.00	1	0.00	A032700090	<LOQ (0.0613 ug/g)		
	C90794M	Unknown	NA	0.00	1	0.00	A022100079	<LOQ (0.0307 ug/g)		
	C90799M	Unknown	NA	0.00	1	0.00	A022100065	<LOQ (0.0307 ug/g)		
	C90804M	Unknown	NA	0.00	1	0.00	A022100080	<LOQ (0.0307 ug/g)		
	C90809M	Unknown	NA	0.00	1	0.00	A032700091	<LOQ (0.0613 ug/g)		
	C90810M	Unknown	NA	0.00	1	0.00	A022100081	<LOQ (0.0307 ug/g)		
	C90811M	Unknown	NA	0.00	1	0.00	A022100066	<LOQ (0.0307 ug/g)		
	C90812M	Unknown	NA	0.00	1	0.00	A022100067	<LOQ (0.0307 ug/g)		
	C90815M	Unknown	NA	0.00	1	0.00	A022100068	<LOQ (0.0307 ug/g)		
	C90823M	Unknown	NA	0.00	1	0.00	A022100071	<LOQ (0.0307 ug/g)		
	C90829M	Unknown	NA	0.00	1	0.00	A022100082	<LOQ (0.0307 ug/g)		
	C90830M	Unknown	NA	0.00	1	0.00	A022100085	<LOQ (0.0307 ug/g)		
	C90832M	Unknown	NA	0.00	1	0.00	A022100086	<LOQ (0.0307 ug/g)		
	C90835M	Unknown	Confirmed High	293	1	292	A042400105	0.292		
	C90838M	Unknown	NA	0.00	1	0.00	A022100087	<LOQ (0.0307 ug/g)		NA
	C90840M	Unknown	NA	0.00	1	0.00	A022100072	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C91197F	Unknown	NA	0.00	1	0.00	A022100088	<LOQ (0.0307 ug/g)		
	C91201F	Unknown	NA	0.00	1	0.00	A022100089	<LOQ (0.0307 ug/g)		
	C91207F	Unknown	NA	0.00	1	0.00	A022100099	<LOQ (0.0307 ug/g)		
	C91211F	Unknown	NA	0.00	1	0.00	A022100100	<LOQ (0.0307 ug/g)		
	C91216F	Unknown	NA	0.00	1	0.00	A022100092	<LOQ (0.0307 ug/g)		
	C91220F	Unknown	NA	0.00	1	0.00	A032700095	<LOQ (0.0613 ug/g)		
	C91221F	Unknown	NA	0.00	1	0.00	A022100093	<LOQ (0.0307 ug/g)		
	C91227F	Unknown	NA	0.00	1	0.00	A022100094	<LOQ (0.0307 ug/g)		
	C91232F	Unknown	NA	0.00	1	0.00	A022100101	<LOQ (0.0307 ug/g)		
	C91235F	Unknown	NA	0.00	1	0.00	A022100102	<LOQ (0.0307 ug/g)		
	C91236F	Unknown	NA	0.00	1	0.00	A022100095	<LOQ (0.0307 ug/g)		
	C91242F	Unknown	NA	0.00	1	0.00	A022100096	<LOQ (0.0307 ug/g)		NA
	C91243F	Unknown	NA	0.00	1	0.00	A022100103	<LOQ (0.0307 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/Analyst. 03/14/00, 03/16/00, 03/17/00, 04/27/00, 05/17/00, 05/18/00, 09/03/00, 03/26/01, 04/19/01 LAC/CSH
Date Verified/Analyst. 03/09/01 hoj
Purity Entered/Verified. 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0.0245 ug/g LAC 02/19/01

ETS-8-7.0

AMD1# 092597.1
Covance# 6329-212

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98 1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number/Test Substance: T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: RTS-8-6.0 & RTS-8-7.0
Analytical Equipment System Number: Amelia 062498, Dewey 070799
Instrument Software/Version: MassLynx 3.1
Date of Extraction/Analyst: 02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS
Date of Analysis/Analyst: 02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 03/27/00, 04/24/00, 04/25/00, 04/26/00, 05/15/00, 03/22/01 MMH/MEE/IAS/SAH
Date of Data Reduction/Analyst: 02/28/00, 03/01/00, 03/03/00, 03/06/00, 04/17/00, 04/24/00, 04/25/00, 04/26/00, 04/27/00, 06/17/00, 03/23/01 IAS/MEE/MMH/ADV
Sample Data:

Filename	See Attachments	
R-Squared Value	See Attachments	
Slope	See Attachments	
Y-Intercept	See Attachments	

WEEK 105 RAT LIVER

Group Dose	Sample #	PFOS Calc. Conc. ug/g	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD	PFOSA Calc. Conc. ug/g	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD	PFOSAA Calc. Conc. ug/g	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD	EtFOSE Calc. Conc. ug/g	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL021400-WBk-1	0.00	<LOQ (0.0245 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0307 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	RBL021400-WBk-2	0.00	<LOQ (0.0245 ug/g)	<LOQ	NA	0.00	<LOQ (0.0123 ug/g)	<LOQ	NA	0.00	<LOQ (0.00615 ug/g)	<LOQ	NA	41.0	<LOQ (0.0614 ug/g)	<LOQ	NA
	RBL021600-WBk-3	0.00	<LOQ (0.0245 ug/g)			4.17	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.123 ug/g)		
	RBL021600-WBk-6	0.00	<LOQ (0.0245 ug/g)	<LOQ	NA	1.16	<LOQ (0.00614 ug/g)	<LOQ	NA	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA	4.60	<LOQ (0.0614 ug/g)	<LOQ	NA
	RBL031501-WBk-1	0.00	<LOQ (0.0613 ug/g)			NA	NA			NA	NA			NA	NA		
	RBL031501-WBk-2	0.00	<LOQ (0.0613 ug/g)	<LOQ	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Matrix Blk	RBL021400-LBk-1	0.00	<LOQ (0.0245 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0307 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	RBL021400-LBk-2	0.00	<LOQ (0.0245 ug/g)	<LOQ	NA	0.00	<LOQ (0.0123 ug/g)	<LOQ	NA	0.00	<LOQ (0.00615 ug/g)	<LOQ	NA	0.00	<LOQ (0.0614 ug/g)	<LOQ	NA
	RBL021600-LBk-5	0.00	<LOQ (0.0245 ug/g)			1.89	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.123 ug/g)		
	RBL021600-LBk-6	0.00	<LOQ (0.0245 ug/g)	<LOQ	NA	0.670	<LOQ (0.00614 ug/g)	<LOQ	NA	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA	0.00	<LOQ (0.0614 ug/g)	<LOQ	NA
	RBL031501-LBk-1	0.00	<LOQ (0.0613 ug/g)			NA	NA			NA	NA			NA	NA		
	RBL031501-LBk-2	0.00	<LOQ (0.0613 ug/g)	<LOQ	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
	RTL031501-LBk-1	12.11	<LOQ (0.0613 ug/g)			NA	NA			NA	NA			NA	NA		
	RTL031501-LBk-2	16.27	0.01627	<LOQ	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
QC - 250 ppb	C90716M-MS-1	242	89%			328	111%			230	84%			368	134%		
	C90716M-MS-2	327	111%	100%	22%	274	92%	102%	18%	356	120%	102%	35%	289	98%	111%	24%
	C90722M-MSD-1	206	71%			103	104%			264	90%			166	57%		
	C90722M-MSD-2	278	95%	83%	30%	313	107%	105%	3%	281	96%	93%	6%	248	85%	71%	40%
	C90721M-MS-5	287	96%			371	124%			275	92%			267	89%		
	C90721M-MSD-5	307	103%	100%	7%	346	116%	120%	7%	282	94%	92%	3%	248	83%	86%	7%
	C90726M-MS-6	180	60%			382	128%			275	92%			107	36%		
	C90726M-MSD-6	285	95%	78%	45%	257	86%	107%	19%	327	109%	100%	17%	94	31%	34%	13%
	C91157F-MS-5	441	146%			338	111%			447	147%			244	81%		
	C91157F-MSD-5	410	136%	141%	7%	353	117%	114%	5%	392	129%	138%	13%	79	26%	53%	102%
	RTL031501-Liver MS	360	114%			NA	NA			NA	NA			NA	NA		
	RTL031501-Liver MSD	301	96%	105%	18%	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 1 Control 0.0 mg/kg	C90721M-Blk-5	18.5	<LOQ (0.0245 ug/g)			0.456	<LOQ (0.0123 ug/g)			28.5	<LOQ (0.0615 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C90721M-Blk-6	23.9	<LOQ (0.0245 ug/g)			4.08	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0615 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C90716M-1	23.5	<LOQ (0.0245 ug/g)			2.41	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0307 ug/g)			82.1	0.0821		
	C90716M-2	39.8	0.0398			4.96	<LOQ (0.0123 ug/g)			31.2	0.0312			0.00	<LOQ (0.0614 ug/g)		
	C90722M-1	141	0.141			2.67	<LOQ (0.0123 ug/g)			51.4	0.0514			93.6	0.0936		
	C90722M-2	140	0.140			2.40	<LOQ (0.0123 ug/g)			55.0	0.0550			124	0.124		
	C90724M	290	0.290			0.600	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0307 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C90726M-Blk-5	55.3	0.0553			1.45	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0615 ug/g)			110	0.110		
	C90726M-Blk-6	57.6	0.0576			3.04	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0615 ug/g)			72.1	0.0721		
	C90728M	208	0.208			4.58	<LOQ (0.0123 ug/g)			0.0	<LOQ (0.0307 ug/g)			153	0.153		
	C90715M	130	0.130			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0307 ug/g)			54.7	<LOQ (0.0614 ug/g)		
	C90742M	315	0.315			1.46	<LOQ (0.0123 ug/g)			37.8	0.0378			0.00	<LOQ (0.0614 ug/g)		
	C90743M	22.0	<LOQ (0.0245 ug/g)			0.00	<LOQ (0.0123 ug/g)			41.25	<LOQ (0.0615 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C90747M	347	0.347			76.7	0.06			64.24	0.0642			0.00	<LOQ (0.0614 ug/g)		
	C90750M	417	0.417			1.69	<LOQ (0.0123 ug/g)			21.25	<LOQ (0.0615 ug/g)			122	0.122		
	C90755M	43.0	0.0430			3.18	<LOQ (0.0123 ug/g)			52.10	<LOQ (0.0615 ug/g)			60.9	<LOQ (0.0614 ug/g)		
	C90765M	19.8	<LOQ (0.0245 ug/g)		93.5	0.523	<LOQ (0.0123 ug/g)		NA	0.00	<LOQ (0.0307 ug/g)		37.8	0.00	<LOQ (0.0614 ug/g)		36.1
	C90778M	224	0.224	0.143	0.113	0.00	<LOQ (0.0123 ug/g)	<LOQ	NA	0.00	<LOQ (0.00615 ug/g)	0.0461	0.0174	23.2	<LOQ (0.123 ug/g)	0.0796	0.0287
Group 1 Control 0.0 mg/kg	C91127F	177	0.177			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0307 ug/g)			68.6	0.0686		
	C91131F	427	0.427			0.796	<LOQ (0.0123 ug/g)			0.0	<LOQ (0.0307 ug/g)			202	0.202		
	C91138F	55.1	0.055			8.97	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.00615 ug/g)			29.1	<LOQ (0.123 ug/g)		
	C91144F	215	0.215			8.88	<LOQ (0.0123 ug/g)			49.90	<LOQ (0.0615 ug/g)			107	0.107		
	C91148F	117	0.117			0.90	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0307 ug/g)			117	0.117		
	C91157F-Blk-5	181	0.181			1.81	<LOQ (0.0123 ug/g)			0.0	<LOQ (0.0615 ug/g)			81.5	0.0815		
	C91157F-Blk-6	181	0.181			4.06	<LOQ (0.0123 ug/g)			25.81	<LOQ (0.0615 ug/g)			73.3	0.0733		
	C91159F	317	0.317			4.19	<LOQ (0.0123 ug/g)			41.38	0.0414			71.6	0.0716		
	C91166F	54.3	0.0543			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0615 ug/g)			89.2	0.0892		
	C91172F	223	0.223			12.8	0.0128			0.00	<LOQ (0.0307 ug/g)			131	0.131		
	C91176F	5.66	<LOQ (0.0245 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0615 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91183F	205	0.205			1.94	<LOQ (0.0123 ug/g)			18.64	0.02			80.0	0.0800		
	C91188F	243	0.243		62.5	0.98	<LOQ (0.0123 ug/g)		NA	0.0000	<LOQ (0.0615 ug/g)		45.6	79.3	0.0793		43.6
	C91190F	65.0	0.0650	0.178	0.111	13.8	0.0138	<LOQ	NA	22.395	<LOQ (0.0615 ug/g)	0.0154	0.0	45.1	0.0451	0.0906	0.0394
Group 2 Low Dose 3.0 mg/kg	C90781M	2977	2.98			929	0.929			1335	1.335			0.00	<LOQ (0.0614 ug/g)		
	C90782M	1504	1.50			894	0.894			1796	1.80			88.3	0.0883		
	C90783M	7907	7.91			865	0.865			970	0.970			57.7	<LOQ (0.0614 ug/g)		
	C90785M	373	0.373			550	0.550			110	0.110			0.00	<LOQ (0.0614 ug/g)		
	C90787M	497	0.497			378	0.378			260	0.260			0.00	<LOQ (0.0614 ug/g)		
	C90788M	24.09	24.3			426	0.426			688	0.688			0.00	<LOQ (0.0614 ug/g)		
	C90791M	12.94	12.4			593	0.593			1060	1.06			0.00	<LOQ (0.0614 ug/g)		
	C90794M	8255	8.26			957	0.957			931	0.931			60.1	<LOQ (0.0614 ug/g)		
	C90799M	14019	14.0			721	0.721			1043	1.04			39.4	<LOQ (0.0614 ug/g)		
	C90804M	14183	14.2			250	0.250			478	0.478			0.00	<LOQ (0.0614 ug/g)		
	C90809M	5897	5.90			1045	1.04			2.08	2.10			0.00	<LOQ (0.0614 ug/g)		
	C90810M	1077	1.08			905	0.905			1182	1.18			0.00	<LOQ (0.0614 ug/g)		
	C90811M	1358	1.36			389	0.389			945	0.945			94.2	0.0942		
	C90812M	2987	2.99			679	0.679			5.08	5.10			0.00	<LOQ (0.0614 ug/g)		
	C90815M	5689	5.69			482	0.482			1841	1.84			56.9	0.0569		
	C90823M	17157	17.2			880	0.880			4002	4.00			104	0.104		
	C90829M	10279	10.3			439	0.439			1171	1.17			52.8	0.0528		
	C90830M	21330	21.3			981	0.981			1419	1.42			0.00	<LOQ (0.0614 ug/g)		
	C90832M	114	0.114			1513	1.513			9192	9.19			51.1	0.0511		
	C90835M	9405	9.41			800	0.800			1371	1.37			0.00	<LOQ (0.0614 ug/g)		
	C90838M	2721	2.72		91.9	661	0.661		39.5	940	0.940		108	0.00	<LOQ (0.0614 ug/g)		20.1
	C90840M	1990	1.99	7.56	7.10	1010	1.01	0.744	0.294	2746	2.75	1.86	2.00	59.8	0.0598	0.0649	0.0130
Group 2 Low Dose 3.0 mg/kg	C91197F	25746	25.7			672	0.672			784	0.784			0.00	<LOQ (0.0614 ug/g)		
	C91201F	26030	26.0			167	0.167			459	0.459			0.00	<LOQ (0.0614 ug/g)		
	C91207F	53763	53.8			1170	1.17			976	0.976			98.5	0.0985		
	C91211F	32550	32.6			634	0.634			889	0.889			101	0.101		
	C91216F	71151	71.2			671	0.671			525	0.525			0.00	<LOQ (0.0614 ug/g)		
	C91220F	20499	20.5			984	0.984			1098	1.10			13.60	<LOQ (0.0614 ug/g)		
	C91221F	38750	38.8			543	0.543			580	0.580			0.00	<LOQ (0.0614 ug/g)		
	C91227F	12540	12.5			386	0.386			615	0.615			0.00	<LOQ (0.0614 ug/g)		
	C91232F	32853	32.9			1353	1.35			11725	11.7			0.00	<LOQ (0.0614 ug/g)		
	C91235F	24425	24.4			5197	5.20			8407	8.41			46.9	<LOQ (0.0614 ug/g)		
	C91236F	20754	20.8			3794	3.79			6366	6.37			91.3	0.0913		
	C91242F	54632	54.6		56.9	4353	4.35		94.9	12061	12.1		128	120	0.120		27.5
	C91243F	88444	88.8	38.5	21.9	882	0.882	1.82	1.72	1007	1.01	3.49	4.47	93.0	0.0930	0.0766	0.0210

Group Dose	Sample #	M556 Calc. Conc. ug/g	Concentration of M556 ug/g or % Rec.	Mean M556 ug/g	RSD Std Dev MS/MSD RPD	PFOSEA Calc. Conc. ug/g	Concentration of PFOSEA ug/g or % Rec.	Mean PFOSEA ug/g	RSD Std Dev MS/MSD RPD
Method Blk	RBL021400-WBk-1	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	RBL021400-WBk-2	0.00	<LOQ (0.00615 ug/g)	<LOQ	NA	0.00	<LOQ (0.0613 ug/g)	<LOQ	NA
	RBL021600-WBk-3	0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	RBL021600-WBk-6	0.00	<LOQ (0.0615 ug/g)	<LOQ	NA	0.00	<LOQ (0.0123 ug/g)	<LOQ	NA
	RBL031501-WBk-1	NA	NA			NA	NA		
	RBL031501-WBk-2	NA	NA	NA	NA	NA	NA	NA	NA
Matrix Blk	RBL021400-LBk-1	0.00	<LOQ (0.0307 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	RBL021400-LBk-2	0.00	<LOQ (0.00615 ug/g)	<LOQ	NA	0.00	<LOQ (0.0613 ug/g)	<LOQ	NA
	RBL021600-LBk-5	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	RBL021600-LBk-6	0.00	<LOQ (0.0613 ug/g)	<LOQ	NA	0.00	<LOQ (0.0123 ug/g)	<LOQ	NA
	RBL031501-LBk-1	NA	NA			NA	NA		
	RBL031501-LBk-2	NA	NA	NA	NA	NA	NA	NA	NA
	RTL031501-LBk-1	NA	NA			NA	NA		
	RTL031501-LBk-2	NA	NA	NA	NA	NA	NA	NA	NA
QC - 250 ppb	C90716M-MS-1	277	93%			347	117%		
	C90716M-MS-2	295	100%	97%	7%	318	108%	113%	9%
	C90722M-MSD-1	244	85%			300	103%		
	C90722M-MSD-2	288	98%	91%	15%	285	98%	100%	5%
	C90721M-MS-5	315	106%			272	92%		
	C90721M-MSD-5	293	99%	103%	7%	248	84%	88%	9%
	C90726M-MS-6	272	92%			259	88%		
	C90726M-MSD-6	283	88%	90%	4%	263	89%	88%	1%
	C91157F-MS-5	429	141%			322	106%		
	C91157F-MSD-5	325	107%	124%	27%	242	80%	93%	28%
	RTL031501-Liver MS	NA	NA			NA	NA		
	RTL031501-Liver MSD	NA	NA	NA	NA	NA	NA	NA	NA
Group 1 Control 0.0 mg/kg	C90721M-Blk-5	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90721M-Blk-6	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90716M-1	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90716M-2	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90722M-1	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90722M-2	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90724M	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90726M-Blk-5	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90726M-Blk-6	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90728M	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90715M	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90742M	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90743M	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90747M	47.6	0.0476			0.00	<LOQ (0.0307 ug/g)		
	C90750M	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90755M	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C90765M	0.00	<LOQ (0.0308 ug/g)		NA	0.00	<LOQ (0.0307 ug/g)		NA
	C90778M	0.00	<LOQ (0.0308 ug/g)	<LOQ	NA	32.3	<LOQ (0.0613 ug/g)	<LOQ	NA
Group 1 Control 0.0 mg/kg	C91127F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91131F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91138F	0.00	<LOQ (0.0615 ug/g)			0.00	<LOQ (0.0613 ug/g)		
	C91144F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91148F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91157F-Blk-5	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91157F-Blk-6	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91159F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91166F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91172F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91176F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91183F	0.00	<LOQ (0.0308 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91188F	0.00	<LOQ (0.0308 ug/g)		NA	0.00	<LOQ (0.0307 ug/g)		NA
	C91190F	0.00	<LOQ (0.0308 ug/g)	<LOQ	NA	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C90781M	112	0.112			0.00	<LOQ (0.0307 ug/g)		
	C90782M	291	0.291			0.00	<LOQ (0.0307 ug/g)		
	C90783M	604	0.604			0.00	<LOQ (0.0307 ug/g)		
	C90785M	64.4	0.0644			0.00	<LOQ (0.0307 ug/g)		
	C90787M	226	0.226			0.00	<LOQ (0.0307 ug/g)		
	C90788M	1771	1.77			0.00	<LOQ (0.0613 ug/g)		
	C90791M	540	0.540			0.00	<LOQ (0.0613 ug/g)		
	C90794M	400	0.400			0.00	<LOQ (0.0307 ug/g)		
	C90799M	417	0.417			0.00	<LOQ (0.0307 ug/g)		
	C90804M	197	0.197			0.00	<LOQ (0.0307 ug/g)		
	C90809M	1102	1.10			0.00	<LOQ (0.0613 ug/g)		
	C90810M	140	0.140			0.00	<LOQ (0.0307 ug/g)		
	C90811M	426	0.426			0.00	<LOQ (0.0307 ug/g)		
	C90812M	242	0.242			0.00	<LOQ (0.0307 ug/g)		
	C90815M	229	0.229			0.00	<LOQ (0.0307 ug/g)		
	C90823M	504	0.504			0.00	<LOQ (0.0307 ug/g)		
	C90829M	497	0.497			0.00	<LOQ (0.0307 ug/g)		
	C90830M	575	0.575			0.00	<LOQ (0.0307 ug/g)		
	C90832M	668	0.668			0.00	<LOQ (0.0307 ug/g)		
	C90835M	185	0.185			292	0.292		
	C90838M	198	0.198		83.9	0.00	<LOQ (0.0307 ug/g)		
	C90840M	521	0.521	0.450	0.378	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 2 Low Dose 3.0 mg/kg	C91197F	457	0.457			0.00	<LOQ (0.0307 ug/g)		
	C91201F	444	0.444			0.00	<LOQ (0.0307 ug/g)		
	C91207F	795	0.795			0.00	<LOQ (0.0613 ug/g)		
	C91211F	322	0.322			0.00	<LOQ (0.0307 ug/g)		
	C91216F	496	0.496			0.00	<LOQ (0.0307 ug/g)		
	C91220F	443	0.443			0.00	<LOQ (0.0613 ug/g)		
	C91221F	325	0.325			0.00	<LOQ (0.0307 ug/g)		
	C91227F	370	0.370			0.00	<LOQ (0.0307 ug/g)		
	C91232F	4092	4.09			0.00	<LOQ (0.0307 ug/g)		
	C91235F	6046	6.05			0.00	<LOQ (0.0307 ug/g)		
	C91236F	3512	3.51			0.00	<LOQ (0.0307 ug/g)		
	C91242F	8724	8.72		115	0.00	<LOQ (0.0307 ug/g)		NA
	C91243F	407	0.407	2.03	2.74	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N(Et)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

* Sample was reextracted 03/15/01, analyzed 03/22/01, and the PFOS concentration reported from 03/15/01 extraction. LAC 03/26/01

Date Entered/Analyst 03/14/00, 03/16/00, 03/17/00, 04/27/00, 05/17/00, 05/18/00, 09/03/00, 03/26/01, 04/19/01 LAC/CSH
Date Verified/Analyst 03/09/01 hcg
Purity Entered/Verified 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0.0245 ug/g LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Liver
Method/Revision:	ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	MassLynx 3.3
Date of Extraction/Analyst:	02/14/00, 02/16/00 RWW/SAL
Date of Analysis/Analyst:	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 07/28/00, 08/15/00 MMH/MEE/IAS
Date of Data Reduction/Analyst:	02/28/00, 03/01/00, 03/03/00, 03/06/00, 07/31/00, 08/17/00 IAS/MMH

Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
	Box 00-012, 00-013

Sample Data

WEEK 105 RAT LIVER Lot 171

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor	Surrogate Verified	PFOS Conc. ng/g	PFOS Dilution Factor	PFOS Calc. Conc. ng/g	Filename	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD
Group 3	C90842M	0.9962	NA	0.9275	0.8640	NA	975	200	156858	A030300037	157		
Mid Dose	C90844M	1.0160	NA	0.9275	0.8640	NA	268	200	42289	A030300038	42.3		
30.0 mg/kg	C90850M	0.9930	NA	0.9275	0.8640	NA	287	200	46376	A030300039	46.4		
	C90851M	1.0565	NA	0.9275	0.8640	NA	796	200	120793	A030300040	121		
	C90854M	0.9872	NA	0.9275	0.8640	NA	1104	200	179256	A030300041	179		
	C90855M	0.9921	NA	0.9275	0.8640	NA	458	200	73931	A030300044	73.9		
	C90856M	1.0485	NA	0.9275	0.8640	NA	106	200	16276	A030300045	16.3		
	C90858M	1.0156	NA	0.9275	0.8640	NA	836	200	131992	A030300046	132		
	C90860M	1.0070	NA	0.9275	0.8640	Low, Not Confirmed	424	1000	337224	A081500088	337		
	C90864M	1.0137	NA	0.9275	0.8640	NA	774	200	122357	A03030048	122		
	C90865M	1.0105	NA	0.9275	0.8640	NA	469	200	74361	A03030051	74.4		
	C90867M	0.9923	NA	0.9275	0.8640	NA	635	200	102548	A03030052	103		
	C90869M	1.1355	NA	0.9275	0.8640	NA	298	200	42001	A03030053	42.0		
	C90871M	1.0511	NA	0.9275	0.8640	NA	595	200	90688	A03030054	90.7		
	C90881M	1.0129	NA	0.9275	0.8640	NA	267	200	42229	A03030055	42.2		
	C90882M	0.9909	NA	0.9275	0.8640	NA	492	200	79628	A03030058	79.6		
	C90883M	0.9926	NA	0.9275	0.8640	NA	239	200	38587	A03030059	38.6		
	C90884M	0.9874	NA	0.9275	0.8640	Low, Not Confirmed	376	200	61036	A081500091	61.0		
	C90885M	1.0068	NA	0.9275	0.8640	NA	642	200	102254	A03030060	102		
	C90889M	1.0299	NA	0.9275	0.8640	NA	232	200	36130	A03030061	36.1		
	C90893M	1.0178	NA	0.9275	0.8640	NA	371	200	58405	A03030062	58.4		78.1
	C90900M	1.0072	NA	0.9275	0.8640	NA	165	200	26321	A03030065	26.3	90.1	70.3
Group 3	C91252F	1.1046	NA	0.9275	0.8640	NA	437	500	158371	A030300017	158		
Mid Dose	C91253F	1.0730	NA	0.9275	0.8640	NA	270	500	100973	A030300018	101		
30.0 mg/kg	C91254F	0.9834	NA	0.9275	0.8640	NA	533	500	217110	A030300019	217		
	C91256F	0.9963	NA	0.9275	0.8640	NA	403	500	161881	A030300020	162		
	C91264F	1.0678	NA	0.9275	0.8640	NA	381	500	143127	A030300023	143		
	C91278F	1.1455	NA	0.9275	0.8640	NA	663	500	231922	A030300024	232		
	C91279F	1.0120	NA	0.9275	0.8640	NA	100	500	39747	A030300016	39.7		
	C91286F	1.0412	NA	0.9275	0.8640	NA	592	500	227867	A030300025	228		
	C91292F	1.0162	NA	0.9275	0.8640	NA	124	500	48865	A030300026	48.9		
	C91294F	1.0046	NA	0.9275	0.8640	NA	294	500	117280	A030300027	117		
	C91295F	1.0159	NA	0.9275	0.8640	NA	56.0	500	22079	A030300030	22.1		
	C91301F	1.1103	NA	0.9275	0.8640	NA	235	500	84658	A030300031	84.7		
	C91303F	0.9908	NA	0.9275	0.8640	NA	411	500	166184	A030300034	166		
	C91307F	1.0083	NA	0.9275	0.8640	NA	141	500	56102	A030300033	56.1		54.0
	C91310F	1.0132	NA	0.9275	0.8640	NA	319	500	126168	A030300032	126	127	68.3

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 08/15/00, 09/03/00 LAC
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Amelia 062498, Davey 070799
Instrument Software/Version: MassLynx 3.3
Date of Extraction/Analyst: 02/14/00, 02/16/00 RWW/SAL
Date of Analysis/Analyst: 02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 07/28/00, 08/15/00 MMH/MEE/IAS
Date of Data Reduction/Analyst: 02/28/00, 03/01/00, 03/03/00, 03/06/00, 07/31/00, 08/17/00 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Box 00-012, 00-013

Sample Data

WEEK 105 RAT LIVER

Group Dose	Sample #	PFOSA Purity Correction Factor (lot L-15709)	Surrogate Verified	PFOSA Conc. ng/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ng/g	Filename	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD	PFOSAA Purity Correction Factor (lot T-7121.1)	Surrogate Verified	PFOSAA Conc. ng/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ng/g	Filename	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD
Group 3	C90842M	Unknown	NA	366	20	7349	A030300087	7.35			Unknown	NA	291	20	5840	A030300087	5.84		
Mid Dose	C90844M	Unknown	NA	188	20	3696	A030300088	3.70			Unknown	NA	354	20	6967	A030300088	6.97		
30.0 mg/kg	C90850M	Unknown	NA	323	20	6514	A030300089	6.51			Unknown	NA	506	20	10194	A030300089	10.2		
	C90851M	Unknown	NA	361	20	6828	A030300090	6.83			Unknown	NA	362	20	6845	A030300090	6.85		
	C90854M	Unknown	NA	472	20	9569	A030300093	9.57			Unknown	NA	180	200	36513	A030300041	36.5		
	C90855M	Unknown	NA	518	20	10434	A030300094	10.4			Unknown	NA	977	20	19689	A030300094	19.7		
	C90856M	Unknown	NA	435	20	8293	A030300095	8.29			Unknown	NA	545	20	10390	A030300095	10.4		
	C90858M	Unknown	NA	314	20	6178	A030300096	6.18			Unknown	NA	471	20	9279	A030300096	9.28		
	C90860M	Unknown	NA	633	20	12569	A030300097	12.6			Unknown	NA	583	20	11573	A030300097	11.6		
	C90864M	Unknown	NA	478	20	9434	A030300100	9.43			Unknown	NA	550	20	10854	A030300100	10.9		
	C90865M	Unknown	NA	224	20	4441	A030300101	4.44			Unknown	NA	241	20	4761	A030300101	4.76		
	C90867M	Unknown	NA	271	20	5466	A030300102	5.47			Unknown	NA	253	20	5093	A030300102	5.09		
	C90869M	Unknown	NA	313	20	5521	A030300103	5.52			Unknown	NA	705	20	12412	A030300103	12.4		
	C90871M	Unknown	NA	454	20	8631	A030300104	8.63			Unknown	NA	620	20	11794	A030300104	11.8		
	C90881M	Unknown	NA	324	20	6403	A030300107	6.40			Unknown	NA	350	20	6916	A030300107	6.92		
	C90882M	Unknown	NA	371	20	7493	A030300108	7.49			Unknown	NA	420	20	8486	A030300108	8.49		
	C90883M	Unknown	NA	270	20	5439	A030300109	5.44			Unknown	NA	295	20	5935	A030300109	5.94		
	C90884M	Unknown	High, Not Confirmed	53.6	200	10849	A081500091	10.8			Unknown	NA	839	20	17002	A081500092	17.0		
	C90885M	Unknown	NA	424	20	8430	A030300110	8.43			Unknown	NA	866	20	17198	A030300110	17.2		
	C90889M	Unknown	NA	338	20	6567	A030300111	6.57			Unknown	NA	802	20	15569	A030300111	15.6		
	C90893M	Unknown	NA	354	20	6949	A030300114	6.95		29.1	Unknown	NA	430	20	8459	A030300114	8.46		60.8
	C90900M	Unknown	NA	334	20	6625	A030300115	6.62	7.44	2.17	Unknown	NA	488	20	9694	A030300115	9.69	11.4	6.95
Group 3	C91252F	Unknown	NA	386	20	6992	A030300067	6.99			Unknown	NA	282	20	5114	A030300067	5.11		
Mid Dose	C91253F	Unknown	NA	387	20	7206	A030300068	7.21			Unknown	NA	430	20	8009	A030300068	8.01		
30.0 mg/kg	C91254F	Unknown	NA	452	20	9190	A030300069	9.19			Unknown	NA	408	20	8292	A030300069	8.29		
	C91256F	Unknown	NA	382	20	7676	A030300072	7.68			Unknown	NA	533	20	10693	A030300072	10.7		
	C91264F	Unknown	NA	334	20	6251	A030300073	6.25			Unknown	NA	271	20	5070	A030300073	5.07		
	C91278F	Unknown	NA	513	20	8954	A030300074	8.95			Unknown	NA	699	20	12207	A030300074	12.2		
	C91279F	Unknown	NA	236	20	4670	A030300066	4.67			Unknown	NA	239	20	4729	A030300066	4.73		
	C91286F	Unknown	NA	497	20	9554	A030300075	9.55			Unknown	NA	911	20	17499	A030300075	17.5		
	C91292F	Unknown	NA	354	20	6960	A030300076	6.96			Unknown	NA	351	20	6906	A030300076	6.91		
	C91294F	Unknown	NA	377	20	7509	A030300079	7.51			Unknown	NA	471	20	9383	A030300079	9.38		
	C91295F	Unknown	NA	166	20	3266	A030300080	3.27			Unknown	NA	261	20	5143	A030300080	5.14		
	C91301F	Unknown	NA	309	20	5559	A030300081	5.56			Unknown	NA	248	20	4470	A030300081	4.47		
	C91303F	Unknown	NA	330	20	6664	A030300082	6.66			Unknown	NA	422	20	8525	A030300082	8.52		
	C91307F	Unknown	NA	297	20	5896	A030300083	5.90		25.4	Unknown	NA	356	20	7067	A030300083	7.07		43.6
	C91310F	Unknown	NA	266	20	5242	A030300086	5.24	6.77	1.72	Unknown	NA	337	20	6627	A030300086	6.66	7.98	3.48

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide
Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 08/15/00, 09/03/00 LAC
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Amelia 062498, Davey 070799
Instrument Software/Version: MassLynx 3.3
Date of Extraction/Analyst: 02/14/00, 02/16/00 RWW/SAL
Date of Analysis/Analyst: 02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 07/28/00, 08/15/00 MMH/MEE/IAS
Date of Data Reduction/Analyst: 02/28/00, 03/01/00, 03/03/00, 03/06/00, 07/31/00, 08/17/00 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 105 RAT LIVER lot Unknown lot NB113047-80

Group Dose	Sample #	EtFOSE Purity Correction Factor	Surrogate Verified	EtFOSE Conc. ng/g	EtFOSE Dilution Factor	EtFOSE Calc. Conc. ng/g	Filename	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD	M556 Purity Correction Factor	Surrogate Verified	M556 Conc. ng/g	M556 Dilution Factor	M556 Calc. Conc. ng/g	Filename	Concentration of M556 ug/g or % Rec.	Mean M556 ug/g	RSD Std Dev MS/MSD RPD
Group 3	C90842M	Unknown	NA	95.0	1	95.3	A072800017	0.0953			Unknown	NA	541	20	10857	A030300087	10.9		
Mid Dose	C90844M	Unknown	NA	16.8	1	16.6	A072800018	<LOQ (0.0614 ug/g)			Unknown	NA	345	20	6798	A030300088	6.80		
30.0 mg/kg	C90850M	Unknown	NA	39.1	1	39.3	A072800019	<LOQ (0.0614 ug/g)			Unknown	NA	421	20	8480	A030300089	8.48		
	C90851M	Unknown	NA	167	1	158	A072800020	0.158			Unknown	NA	614	20	11631	A030300090	11.6		
	C90854M	Unknown	NA	39.1	1	39.6	A072800021	<LOQ (0.0614 ug/g)			Unknown	NA	797	20	16151	A030300093	16.2		
	C90855M	Unknown	NA	96.3	1	97.0	A072800024	0.0970			Unknown	NA	427	20	8603	A030300094	8.60		
	C90856M	Unknown	NA	34.5	1	32.9	A072800025	<LOQ (0.0614 ug/g)			Unknown	NA	255	20	4869	A030300095	4.87		
	C90858M	Unknown	NA	66.2	1	65.2	A072800026	0.0652			Unknown	NA	590	20	11618	A030300096	11.6		
	C90860M	Unknown	NA	80.5	1	80.0	A072800027	0.0800			Unknown	NA	778	20	15449	A030300097	15.4		
	C90864M	Unknown	NA	76.3	1	75.2	A072800028	0.0752			Unknown	NA	597	20	11779	A030300100	11.8		
	C90865M	Unknown	NA	137	1	135	A072800031	0.135			Unknown	NA	399	20	7888	A030300101	7.89		
	C90867M	Unknown	NA	80.6	1	81.2	A072800032	0.0812			Unknown	NA	312	20	6286	A030300102	6.29		
	C90869M	Unknown	NA	86.0	1	75.7	A072800033	0.0757			Unknown	NA	453	20	7985	A030300103	7.99		
	C90871M	Unknown	NA	51.3	1	48.8	A072800034	<LOQ (0.0614 ug/g)			Unknown	NA	545	20	10364	A030300104	10.4		
	C90881M	Unknown	NA	58.8	1	58.0	A072800035	<LOQ (0.0614 ug/g)			Unknown	NA	326	20	6427	A030300107	6.43		
	C90882M	Unknown	NA	153	1	155	A072800038	0.155			Unknown	NA	520	20	10500	A030300108	10.5		
	C90883M	Unknown	NA	48.4	1	48.8	A072800039	<LOQ (0.0614 ug/g)			Unknown	NA	264	20	5327	A030300109	5.33		
	C90884M	Unknown	NA	72.4	1	73.3	A072800040	0.0733			Unknown	NA	729	20	14760	A081500092	14.8		
	C90885M	Unknown	NA	219	1	218	A072800041	0.218			Unknown	NA	618	20	12272	A030300110	12.3		
	C90889M	Unknown	NA	49.2	1	47.7	A072800042	<LOQ (0.0614 ug/g)			Unknown	NA	413	20	8025	A030300111	8.03		
	C90893M	Unknown	NA	81.3	1	79.9	A072800045	0.0799		47.1	Unknown	NA	393	20	7724	A030300114	7.72		34.6
	C90900M	Unknown	NA	0.00	1	0.00	A072800046	<LOQ (0.0614 ug/g)	0.0883	0.0415	Unknown	NA	272	20	5398	A030300115	5.40	9.51	3.29
Group 3	C91252F	Unknown	NA	84.1	1	76.2	A072800047	0.0762			Unknown	NA	537	20	9729	A030300067	9.73		
Mid Dose	C91253F	Unknown	NA	112	1	105	A072800048	0.105			Unknown	NA	455	20	8473	A030300068	8.47		
30.0 mg/kg	C91254F	Unknown	NA	96.9	1	98.5	A072800049	0.0985			Unknown	NA	770	20	15661	A030300069	15.7		
	C91256F	Unknown	NA	97.8	1	98.1	A072800052	0.0981			Unknown	NA	512	20	10275	A030300072	10.3		
	C91264F	Unknown	NA	111	1	104	A072800053	0.104			Unknown	NA	357	20	6689	A030300073	6.69		
	C91278F	Unknown	NA	147	1	129	A072800054	0.129			Unknown	NA	584	20	10199	A030300074	10.2		
	C91279F	Unknown	NA	24.1	1	23.8	A072800055	<LOQ (0.0614 ug/g)			Unknown	NA	226	20	4468	A030300066	4.47		
	C91286F	Unknown	NA	111	1	107	A072800056	0.107			Unknown	NA	523	20	10051	A030300075	10.1		
	C91292F	Unknown	NA	154	1	151	A072800059	0.151			Unknown	NA	479	20	9421	A030300076	9.42		
	C91294F	Unknown	NA	138	1	137	A072800060	0.137			Unknown	NA	517	20	10286	A030300079	10.3		
	C91295F	Unknown	NA	9.69	1	9.54	A072800061	<LOQ (0.0614 ug/g)			Unknown	NA	150	20	2961	A030300080	2.96		
	C91301F	Unknown	NA	66.0	1	59.4	A072800062	0.0594			Unknown	NA	415	20	7474	A030300081	7.47		
	C91303F	Unknown	NA	120	1	122	A072800063	0.122			Unknown	NA	620	20	12522	A030300082	12.5		
	C91307F	Unknown	NA	89.1	1	88.4	A072800066	0.0884		26.9	Unknown	NA	268	20	5325	A030300083	5.32		38.8
	C91310F	Unknown	NA	33.7	1	33.3	A072800067	<LOQ (0.0614 ug/g)	0.103	0.0276	Unknown	NA	254	20	5006	A030300086	5.01	8.57	5.33

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide
Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 08/15/00, 09/03/00 LAC
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Amelia 062498, Davey 070799
Instrument Software/Version: MassLynx 3.3
Date of Extraction/Analyst: 02/14/00, 02/16/00 RWW/SAL
Date of Analysis/Analyst: 02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 07/28/00, 08/15/00 MMH/MEE/IAS
Date of Data Reduction/Analyst: 02/28/00, 03/01/00, 03/03/00, 03/06/00, 07/31/00, 08/17/00 IAS/MMH

Filename: See Below
R-Squared Valu See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Box 00-012, 00-013

Sample Data

WEEK 105 RAT LIVER lot 529

Group Dose	Sample #	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Conc. ng/g	PFOSEA Dilution Factor	PFOSEA Calc. Conc. ug/g	Filename	Concentration of PFOSEA ug/g or % Rec.	Mean PFOSEA ug/g	RSD Std Dev MS/MSD RPD
Group 3	C90842M	Unknown	NA	0.00	1	0.00	A072800017	<LOQ (0.0307 ug/g)		
Mid Dose	C90844M	Unknown	NA	0.00	1	0.00	A072800018	<LOQ (0.0307 ug/g)		
30.0 mg/kg	C90850M	Unknown	NA	0.00	1	0.00	A072800019	<LOQ (0.0307 ug/g)		
	C90851M	Unknown	NA	0.00	1	0.00	A072800020	<LOQ (0.0307 ug/g)		
	C90854M	Unknown	NA	2.94	1	2.98	A072800021	<LOQ (0.0307 ug/g)		
	C90855M	Unknown	NA	2.53	1	2.55	A072800024	<LOQ (0.0307 ug/g)		
	C90856M	Unknown	NA	3.93	1	3.75	A072800025	<LOQ (0.0307 ug/g)		
	C90858M	Unknown	NA	16.0	1	15.7	A072800026	<LOQ (0.0307 ug/g)		
	C90860M	Unknown	NA	0.00	1	0.00	A072800027	<LOQ (0.0307 ug/g)		
	C90864M	Unknown	NA	0.00	1	0.00	A072800028	<LOQ (0.0307 ug/g)		
	C90865M	Unknown	NA	0.00	1	0.00	A072800031	<LOQ (0.0307 ug/g)		
	C90867M	Unknown	NA	0.00	1	0.00	A072800032	<LOQ (0.0307 ug/g)		
	C90869M	Unknown	NA	0.780	1	0.687	A072800033	<LOQ (0.0307 ug/g)		
	C90871M	Unknown	NA	3.64	1	3.46	A072800034	<LOQ (0.0307 ug/g)		
	C90881M	Unknown	NA	6.14	1	6.06	A072800035	<LOQ (0.0307 ug/g)		
	C90882M	Unknown	NA	0.00	1	0.00	A072800038	<LOQ (0.0307 ug/g)		
	C90883M	Unknown	NA	0.00	1	0.00	A072800039	<LOQ (0.0307 ug/g)		
	C90884M	Unknown	NA	0.180	1	0.182	A072800040	<LOQ (0.0307 ug/g)		
	C90885M	Unknown	NA	3.29	1	3.27	A072800041	<LOQ (0.0307 ug/g)		
	C90889M	Unknown	NA	0.00	1	0.00	A072800042	<LOQ (0.0307 ug/g)		
	C90893M	Unknown	NA	3.43	1	3.37	A072800045	<LOQ (0.0307 ug/g)		NA
	C90900M	Unknown	NA	0.00	1	0.00	A072800046	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 3	C91252F	Unknown	NA	1.03	1	0.932	A072800047	<LOQ (0.0307 ug/g)		
Mid Dose	C91253F	Unknown	NA	0.230	1	0.214	A072800048	<LOQ (0.0307 ug/g)		
30.0 mg/kg	C91254F	Unknown	NA	0.00	1	0.00	A072800049	<LOQ (0.0307 ug/g)		
	C91256F	Unknown	NA	0.00	1	0.00	A072800052	<LOQ (0.0307 ug/g)		
	C91264F	Unknown	NA	4.13	1	3.87	A072800053	<LOQ (0.0307 ug/g)		
	C91278F	Unknown	NA	2.91	1	2.54	A072800054	<LOQ (0.0307 ug/g)		
	C91279F	Unknown	NA	0.00	1	0.00	A072800055	<LOQ (0.0307 ug/g)		
	C91286F	Unknown	NA	3.35	1	3.22	A072800056	<LOQ (0.0307 ug/g)		
	C91292F	Unknown	NA	0.00	1	0.00	A072800059	<LOQ (0.0307 ug/g)		
	C91294F	Unknown	NA	2.55	1	2.54	A072800060	<LOQ (0.0307 ug/g)		
	C91295F	Unknown	NA	0.00	1	0.00	A072800061	<LOQ (0.0307 ug/g)		
	C91301F	Unknown	NA	3.18	1	2.86	A072800062	<LOQ (0.0307 ug/g)		
	C91303F	Unknown	NA	0.00	1	0.00	A072800063	<LOQ (0.0307 ug/g)		
	C91307F	Unknown	NA	16.8	1	16.6	A072800066	<LOQ (0.0307 ug/g)		NA
	C91310F	Unknown	NA	0.00	1	0.00	A072800067	<LOQ (0.0307 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuorooctane sulfonyl ethylamide
Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 08/15/00, 09/03/00 LAC
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver	Filename:	See Attachments
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Amelia 062498, Davey 070799	Slope:	See Attachments
Instrument Software/Version:	MassLynx 3.3	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	02/14/00, 02/16/00 RWW/SAL		
Date of Analysis/Analyst:	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 07/28/00, 08/15/00 MMH/MEE/IAS		
Date of Data Reduction/Analyst:	02/28/00, 03/01/00, 03/03/00, 03/06/00, 07/31/00, 08/17/00 IAS/MMH		

Sample Data

WEEK 105 RAT LIVER

Group Dose	Sample #	PFOS Calc. Conc. ng/g	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD	PFOSA Calc. Conc. ng/g	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD	PFOSAA Calc. Conc. ng/g	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD	EtFOSE Calc. Conc. ng/g	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD
Group 3	C90842M	156858	157			7349	7.35			5840	5.84			95.3	0.0953		
Mid Dose	C90844M	42289	42.3			3696	3.70			6967	6.97			16.6	<LOQ (0.0614 ug/g)		
30.0 mg/kg	C90850M	46376	46.4			6514	6.51			10194	10.2			39.3	<LOQ (0.0614 ug/g)		
	C90851M	120793	121			6828	6.83			6845	6.85			158	0.158		
	C90854M	179256	179			9569	9.57			36513	36.5			39.6	<LOQ (0.0614 ug/g)		
	C90855M	73931	73.9			10434	10.4			19689	19.7			97.0	0.0970		
	C90856M	16276	16.3			8293	8.29			10390	10.4			32.9	<LOQ (0.0614 ug/g)		
	C90858M	131992	132			6178	6.18			9279	9.28			65.2	0.0652		
	C90860M	337224	337			12569	12.6			11573	11.6			80.0	0.0800		
	C90864M	122357	122			9434	9.43			10854	10.9			75.2	0.0752		
	C90865M	74361	74.4			4441	4.44			4761	4.76			135	0.135		
	C90867M	102548	103			5466	5.47			5093	5.09			81.2	0.0812		
	C90869M	42001	42.0			5521	5.52			12412	12.4			75.7	0.0757		
	C90871M	90688	91			8631	8.63			11794	11.8			48.8	<LOQ (0.0614 ug/g)		
	C90881M	42229	42.2			6403	6.40			6916	6.92			58.0	<LOQ (0.0614 ug/g)		
	C90882M	79628	79.6			7493	7.49			8486	8.49			155	0.155		
	C90883M	38587	38.6			5439	5.44			5935	5.94			48.8	<LOQ (0.0614 ug/g)		
	C90884M	61036	61.0			10849	10.8			17002	17.0			73.3	0.0733		
	C90885M	102254	102			8430	8.43			17198	17.2			218	0.218		
	C90889M	36130	36.1			6567	6.57			15569	15.6			47.7	<LOQ (0.0614 ug/g)		
	C90893M	58405	58.4		78.1	6949	6.95		29.1	8459	8.46		60.8	79.9	0.0799		47.1
	C90900M	26321	26.3	90.1	70.3	6625	6.62	7.44	2.17	9694	9.69	11.4	6.95	0.00	<LOQ (0.0614 ug/g)	0.0883	0.0415
Group 3	C91252F	158371	158			6992	6.99			5114	5.11			76.2	0.0762		
Mid Dose	C91253F	100973	101			7206	7.21			8009	8.01			105	0.105		
30.0 mg/kg	C91254F	217110	217			9190	9.19			8292	8.29			98.5	0.0985		
	C91256F	161881	162			7676	7.68			10693	10.7			98.1	0.0981		
	C91264F	143127	143			6251	6.25			5070	5.07			104	0.104		
	C91278F	231922	232			8954	8.95			12207	12.2			129	0.129		
	C91279F	39747	39.7			4670	4.67			4729	4.73			23.8	<LOQ (0.0614 ug/g)		
	C91286F	227867	228			9554	9.55			17499	17.5			107	0.107		
	C91292F	48865	48.9			6960	6.96			6906	6.91			151	0.151		
	C91294F	117280	117			7509	7.51			9383	9.38			137	0.137		
	C91295F	22079	22.1			3266	3.27			5143	5.14			9.54	<LOQ (0.0614 ug/g)		
	C91301F	84658	84.7			5559	5.56			4470	4.47			59.4	0.0594		
	C91303F	166184	166			6664	6.66			8525	8.52			122	0.122		
	C91307F	56102	56.1		54.0	5896	5.90		25.4	7067	7.07		43.6	88.4	0.0884		26.9
	C91310F	126168	126	127	68.5	5242	5.24	6.77	1.72	6657	6.66	7.98	3.48	33.3	<LOQ (0.0614 ug/g)	0.1028	0.0276

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/Analyst:	03/14/00, 03/16/00, 03/17/00, 08/15/00, 09/03/00 LAC
Date Verified/Analyst:	03/09/01 hoj
Purity Entered/Verified:	02/19/01 LAC

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver	Filename:	See Below
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Amelia 062498, Davey 070799	Slope:	See Attachments
Instrument Software/Version:	MassLynx 3.3	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	02/14/00, 02/16/00 RWW/SAL		Box 00-012, 00-013
Date of Analysis/Analyst:	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 07/28/00, 08/15/00 MMH/MEE/IAS		
Date of Data Reduction/Analyst:	02/28/00, 03/01/00, 03/03/00, 03/06/00, 07/31/00, 08/17/00 IAS/MMH		

Sample Data

WEEK 105 RAT LIVER

Group Dose	Sample #	M556 Calc. Conc. ng/g	Concentration of M556 ug/g or % Rec.	Mean M556 ug/g	RSD Std Dev MS/MSD RPD	PFOSEA Calc. Conc. ug/g	Concentration of PFOSEA ug/g or % Rec.	Mean PFOSEA ug/g	RSD Std Dev MS/MSD RPD
Group 3	C90842M	10857	10.9			0.00	<LOQ (0.0307 ug/g)		
Mid Dose	C90844M	6798	6.80			0.00	<LOQ (0.0307 ug/g)		
30.0 mg/kg	C90850M	8480	8.48			0.00	<LOQ (0.0307 ug/g)		
	C90851M	11631	11.6			0.00	<LOQ (0.0307 ug/g)		
	C90854M	16151	16.2			2.98	<LOQ (0.0307 ug/g)		
	C90855M	8603	8.60			2.55	<LOQ (0.0307 ug/g)		
	C90856M	4869	4.87			3.75	<LOQ (0.0307 ug/g)		
	C90858M	11618	11.6			15.7	<LOQ (0.0307 ug/g)		
	C90860M	15449	15.4			0.00	<LOQ (0.0307 ug/g)		
	C90864M	11779	11.8			0.00	<LOQ (0.0307 ug/g)		
	C90865M	7888	7.89			0.00	<LOQ (0.0307 ug/g)		
	C90867M	6286	6.29			0.00	<LOQ (0.0307 ug/g)		
	C90869M	7985	7.99			0.687	<LOQ (0.0307 ug/g)		
	C90871M	10364	10.4			3.46	<LOQ (0.0307 ug/g)		
	C90881M	6427	6.43			6.06	<LOQ (0.0307 ug/g)		
	C90882M	10500	10.5			0.00	<LOQ (0.0307 ug/g)		
	C90883M	5327	5.33			0.00	<LOQ (0.0307 ug/g)		
	C90884M	14760	14.76			0.182	<LOQ (0.0307 ug/g)		
	C90885M	12272	12.3			3.27	<LOQ (0.0307 ug/g)		
	C90889M	8025	8.03			0.00	<LOQ (0.0307 ug/g)		
	C90893M	7724	7.72		34.6	3.37	<LOQ (0.0307 ug/g)		NA
	C90900M	5398	5.40	9.51	3.29	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 3	C91252F	9729	9.73			0.932	<LOQ (0.0307 ug/g)		
Mid Dose	C91253F	8473	8.47			0.214	<LOQ (0.0307 ug/g)		
30.0 mg/kg	C91254F	15661	15.7			0.00	<LOQ (0.0307 ug/g)		
	C91256F	10275	10.3			0.00	<LOQ (0.0307 ug/g)		
	C91264F	6689	6.69			3.87	<LOQ (0.0307 ug/g)		
	C91278F	10199	10.2			2.54	<LOQ (0.0307 ug/g)		
	C91279F	4468	4.47			0.00	<LOQ (0.0307 ug/g)		
	C91286F	10051	10.1			3.22	<LOQ (0.0307 ug/g)		
	C91292F	9421	9.42			0.00	<LOQ (0.0307 ug/g)		
	C91294F	10286	10.3			2.54	<LOQ (0.0307 ug/g)		
	C91295F	2961	2.96			0.00	<LOQ (0.0307 ug/g)		
	C91301F	7474	7.47			2.86	<LOQ (0.0307 ug/g)		
	C91303F	12522	12.5			0.00	<LOQ (0.0307 ug/g)		
	C91307F	5325	5.32		38.8	16.6	<LOQ (0.0307 ug/g)		NA
	C91310F	5006	5.01	8.57	3.33	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
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Date Entered/Analyst:	03/14/00, 03/16/00, 03/17/00, 08/15/00, 09/03/00 LAC
Date Verified/Analyst:	03/09/01 hoj
Purity Entered/Verified:	02/19/01 LAC

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Soup 020199, Amelia 062498
Instrument Software/Version: MassLynx 3.3
Date of Extraction/Analyst: 02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS
Date of Analysis/Analyst: 02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 4/28/00, 08/01/00, 08/14/00, 08/15/00, 03/22/01 MMH MEE/IAS
Date of Data Reduction/Analyst: 02/28/00, 03/01/00, 03/03/00, 03/06/00, 5/3/00, 5/4/00, 08/02/00, 08/15/00, 08/17/00, 03/23/01 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments
Box 00-012, 00-013

Sample Data

WEEK 105 RAT LIVER Lot 171

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor	Surrogate Verified	PFOS Conc. ng/g	PFOS Dilution Factor	PFOS Calc. Conc. ng/g	Filename	Concentration of PFOS ug/g or % Rec.		Mean PFOS ug/g	RSD Std Dev MS/MSD RPD
Group 4	C90904M	1.0750	NA	0.9275	0.8640	NA	867	500	323257	A030100041	323			
Mid-High Dose	C90908M	1.0601	NA	0.9275	0.8640	NA	829	500	313374	A030100044	313			
100 mg/kg	C90910M	1.0130	NA	0.9275	0.8640	NA	250	500	98979	A030100045	99.0			
	C90911M	0.9883	NA	0.9275	0.8640	NA	886	1000	718021	A041200037	718			
	C90916M	0.9913	NA	0.9275	0.8640	NA	584	500	236132	A030100047	236			
	C90926M	1.0448	NA	0.9275	0.8640	NA	313	500	120112	A030100048	120			
	C90927M	1.0776	NA	0.9275	0.8640	NA	70.6	500	26240	A030100051	26.2			
	C90928M	1.0152	NA	0.9275	0.8640	NA	796	1000	628332	A041200038	628			
	C90933M	1.0884	NA	0.9275	0.8640	NA	416	500	153299	A030100053	153			
	C90944M	0.9935	NA	1.0000	1.0000	High, Not Confirmed	561	1000	565043	A010322030	565	A		
	C90947M	1.0069	NA	0.9275	0.8640	NA	742	500	295355	A030100055	295			
	C90948M	0.9872	NA	0.9275	0.8640	NA	460	500	186556	A030100058	187			
	C90953M	1.0290	NA	0.9275	0.8640	NA	155	500	60324	A030100059	60.3			
	C90955M	1.0011	NA	0.9275	0.8640	NA	663	500	265167	A030100060	265			
	C90956M	1.0111	NA	1.0000	1.0000	High, Not Confirmed	407	500	201182	A010322035	201	A		
	C90965M	0.9962	NA	0.9275	0.8640	NA	857	1000	689313	A041200040	689			70.4
	C90968M	1.1563	NA	0.9275	0.8640	NA	642	1000	445034	A041200041	445		313	220
Group 4	C91315F	0.9933	NA	0.9275	0.8640	NA	570	500	230009	A030100016	230			
Mid-High Dose	C91324F	0.9989	NA	0.9275	0.8640	NA	635	1000	509159	A041200029	509			
100 mg/kg	C91327F	0.9827	NA	0.9275	0.8640	NA	778	50	31737	A030100093	31.7			
	C91332F	1.1502	NA	0.9275	0.8640	NA	542	1000	377842	A041200030	378			
	C91335F	1.1177	NA	0.9275	0.8640	NA	614	500	220157	A030100020	220			
	C91336F	1.0214	NA	0.9275	0.8640	NA	841	500	329837	A030100023	330			
	C91338F	0.9986	NA	0.9275	0.8640	NA	851	500	341389	A030100024	341			
	C91340F	1.0204	NA	0.9275	0.8640	NA	738	1000	579769	A041200031	580			
	C91341F	1.0345	NA	0.9275	0.8640	NA	626	500	242581	A030100026	243			
	C91342F	1.0056	NA	0.9275	0.8640	NA	891	500	355109	A030100027	355			
	C91356F	1.0115	NA	0.9275	0.8640	NA	214	250	42389	A041200045	42.4			
	C91357F	0.9988	NA	0.9275	0.8640	NA	481	500	192963	A030100031	193			
	C91360F	1.0132	NA	0.9275	0.8640	NA	522	500	206612	A030100032	207			
	C91361F	0.9998	NA	0.9275	0.8640	NA	889	1000	712319	A041200032	712			
	C91362F	1.0130	NA	0.9275	0.8640	NA	220	500	86896	A030100034	86.9			
	C91372F	0.9926	NA	0.9275	0.8640	NA	639	500	257883	A030100037	258			
	C91376F	1.0544	NA	0.9275	0.8640	NA	668	500	253932	A030100038	254			
	C91378F	1.0032	NA	0.9275	0.8640	NA	853	500	340658	A030100039	341			57.7
	C91380F	0.9840	NA	0.9275	0.8640	NA	797	500	324616	A030100040	325		297	171
Group 6	C91047M	0.9884	NA	0.9275	0.8640	NA	251	10	2034	A081500102	2.03			
Mid-High Dose	C91054M	1.0156	NA	0.9275	0.8640	NA	251	1	198	A042800044	0.198			
Recovery	C91056M	1.0019	NA	0.9275	0.8640	Low, not Confirmed	131	100	10494	A081500105	10.5			
100 mg/kg	C91060M	1.0062	NA	0.9275	0.8640	NA	252	10	2006	A081500106	2.01			
	C91065M	0.9918	NA	0.9275	0.8640	NA	45.2	1	36.5	A042800017	0.0365			
	C91070M	0.9943	NA	0.9275	0.8640	NA	254	1	205	A042800018	0.205			
	C91071M	0.9973	NA	0.9275	0.8640	NA	51.2	1	41.1	A042800019	0.0411			
	C91072M	0.9986	NA	0.9275	0.8640	NA	479	1	384	A042800022	0.384			
	C91073M	1.0016	NA	0.9275	0.8640	NA	104	1	83.1	A042800023	0.0831			164
	C91076M	1.0102	NA	0.9275	0.8640	High, Not Confirmed	633	10	5025	A010322065	5.02		2.05	3.36
Group 6	C91451F	1.0161	NA	0.9275	0.8640	Low, not Confirmed	195	100	15396	A030400022	15.4			
Mid-High Dose	C91453F	1.0133	NA	0.9275	0.8640	NA	572	10	4525	A081500107	4.53			
Recovery	C91455F	1.0117	NA	0.9275	0.8640	Low, not Confirmed	237	100	18803	A030400024	18.8			
100 mg/kg	C91459F	1.0140	NA	0.9275	0.8640	NA	62.6	1	49.4	A042800030	0.0494			
	C91462F	1.0082	NA	0.9275	0.8640	Low, not Confirmed	221	100	17533	A030400026	17.5			
	C91467F	1.0130	NA	0.9275	0.8640	NA	174	1	137	A042800032	0.137			
	C91473F	0.9879	NA	0.9275	0.8640	Low, not Confirmed	171	100	13901	A030400030	13.9			
	C91479F	1.0018	NA	0.9275	0.8640	NA	25.8	1	20.7	A042800036	<LOQ (0.0245 ug/g)			
	C91480F	0.9882	NA	0.9275	0.8640	NA	183	1	149	A042800037	0.149			
	C91485F	0.9812	NA	0.9275	0.8640	Low, not Confirmed	253	50	10329	A081500108	10.3			
	C91487F	1.0175	NA	0.9275	0.8640	NA	79.7	1	62.8	A042800039	0.0628			99.0
	C91488F	1.0101	NA	0.9275	0.8640	Low, not Confirmed	143	100	11369	A030400037	11.4		7.69	7.62

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M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Value estimated, above the range of the calibration curve. LAC 03/14/01
** Data may be biased high as CCVs weren't within criteria. LAC 03/14/01
A = Sample was reextracted 03/15/01, analyzed 03/22/01, and the PFOS concentration reported from 03/15/01 extraction. LAC 03/26/01

Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 05/05/00, 05/18/00, 08/15/00, 08/21/00, 09/03/00, 03/26/01 LAC/CSH/MMH
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0.0245 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver	Filename:	See Below
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Soup 020199, Amelia 062498	Slope:	See Attachments
Instrument Software/Version:	MassLynx 3.3	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS	Box 00-012, 00-013	
Date of Analysis/Analyst:	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 4/28/00, 08/01/00, 08/14/00, 08/15/00, 03/22/01 MMH/MEE/IAS		
Date of Data Reduction/Analyst:	02/28/00, 03/01/00, 03/03/00, 03/06/00, 5/3/00, 5/4/00, 08/02/00, 08/15/00, 08/17/00, 03/23/01 IAS/MMH		

Sample Data

WEEK 105 RAT LIVER

Group Dose	Sample #	PFOSA Purity Correction Factor (lot L-15709)	Surrogate Verified	PFOSA Conc. ng/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ng/g	Filename	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD	PFOSAA Purity Correction Factor (lot T-7121.1)	Surrogate Verified	PFOSAA Conc. ng/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ng/g	Filename	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD
Group 4	C90904M	Unknown	NA	316	50	14679	A030100066	14.7			Unknown	NA	459	50	21358	A030100066	21.4		
Mid-High Dose	C90908M	Unknown	NA	316	50	14920	A030100067	14.9			Unknown	NA	392	50	18476	A030100067	18.5		
100 mg/kg	C90910M	Unknown	NA	251	50	12376	A030100068	12.4			Unknown	NA	909	50	44847	A030100068	44.8		
	C90911M	Unknown	NA	367	50	18544	A030100069	18.5			Unknown	NA	696	50	35222	A030100069	35.2		
	C90916M	Unknown	NA	353	50	17812	A030100072	17.8			Unknown	NA	475	50	23948	A030100072	23.9		
	C90926M	Unknown	NA	84.9	50	4061	A030100073	4.06			Unknown	NA	37.3	50	1784	A030100073	1.78		
	C90927M	Unknown	NA	249	50	11539	A030100074	11.5			Unknown	NA	333	50	15462	A030100074	15.5		
	C90928M	Unknown	NA	358	50	17644	A030100075	17.6			Unknown	NA	819	50	40328	A030100075	40.3		
	C90933M	Unknown	NA	288	50	13218	A030100076	13.2			Unknown	NA	403	50	18511	A030100076	18.5		
	C90944M	Unknown	NA	299	50	15070	A030100079	15.1			Unknown	NA	457	50	23008	A030100079	23.0		
	C90947M	Unknown	NA	198	50	9823	A030100080	9.82			Unknown	NA	358	50	17773	A030100080	17.8		
	C90948M	Unknown	NA	186	50	9411	A030100081	9.41			Unknown	NA	226	50	11445	A030100081	11.4		
	C90953M	Unknown	NA	206	50	10031	A030100082	10.0			Unknown	NA	377	50	18340	A030100082	18.3		
	C90955M	Unknown	NA	291	50	14511	A030100083	14.5			Unknown	NA	548	50	27358	A030100083	27.4		
	C90956M	Unknown	NA	176	50	8680	A030100086	8.68			Unknown	NA	95.5	50	4725	A030100086	4.72		
	C90965M	Unknown	NA	407	50	20448	A030100087	20.4		31.3	Unknown	NA	1031	50	51758	A030100087	51.8		56.5
	C90968M	Unknown	NA	384	50	16616	A030100088	16.6	13.5	4.23	Unknown	NA	622	50	26905	A030100088	26.9	23.6	13.3
Group 4	C91315F	Unknown	NA	254	50	12800	A030100089	12.8			Unknown	NA	314	50	15794	A030100089	15.8		
Mid-High Dose	C91324F	Unknown	NA	324	50	16226	A030100090	16.2			Unknown	NA	564	50	28252	A030100090	28.3		
100 mg/kg	C91327F	Unknown	NA	209	50	10627	A030100093	10.6			Unknown	NA	758	50	38590	A030100093	38.6		
	C91332F	Unknown	NA	392	50	17062	A030100094	17.1			Unknown	NA	585	50	25423	A030100094	25.4		
	C91335F	Unknown	NA	325	50	14551	A030100095	14.6			Unknown	NA	408	50	18251	A030100095	18.3		
	C91336F	Unknown	NA	305	50	14949	A030100096	14.9			Unknown	NA	402	50	19676	A030100096	19.7		
	C91338F	Unknown	NA	361	50	18058	A030100097	18.1			Unknown	NA	418	50	20954	A030100097	21.0		
	C91340F	Unknown	NA	329	50	16126	A030100100	16.1			Unknown	NA	537	50	26299	A030100100	26.3		
	C91341F	Unknown	NA	316	50	15274	A030100101	15.3			Unknown	NA	524	50	25334	A030100101	25.3		
	C91342F	Unknown	NA	338	50	16810	A030100102	16.8			Unknown	NA	673	50	33469	A030100102	33.5		
	C91356F	Unknown	NA	125	50	6157	A030100103	6.16			Unknown	NA	349	50	17230	A030100103	17.2		
	C91357F	Unknown	NA	336	50	16831	A030100104	16.8			Unknown	NA	413	50	20686	A030100104	20.7		
	C91360F	Unknown	NA	364	50	17955	A030100107	18.0			Unknown	NA	577	50	28490	A030100107	28.5		
	C91361F	Unknown	NA	446	50	22322	A030100108	22.3			Unknown	NA	631	50	31554	A030100108	31.6		
	C91362F	Unknown	NA	220	50	10883	A030100109	10.9			Unknown	NA	608	50	30022	A030100109	30.0		
	C91372F	Unknown	NA	386	50	19454	A030100110	19.5			Unknown	NA	840	50	42332	A030100110	42.3		
	C91376F	Unknown	NA	344	50	16296	A030100111	16.3			Unknown	NA	578	50	27417	A030100111	27.4		
	C91378F	Unknown	NA	380	50	18952	A030100114	19.0		23.1	Unknown	NA	604	50	30114	A030100114	30.1		26.3
	C91380F	Unknown	NA	303	50	15385	A030100115	15.4	15.6	3.61	Unknown	NA	604	50	30712	A030100115	30.7	26.9	7.08
Group 6	C91047M	Unknown	NA	2.14	1	2.17	A042800043	<LOQ (0.00614 ug/g)			Unknown	NA	217	1	220	A042800043	0.220		
Mid-High Dose	C91054M	Unknown	NA	2.50	1	2.46	A042800044	<LOQ (0.00614 ug/g)			Unknown	NA	19.8	1	19.5	A042800044	0.0195		
Recovery	C91056M	Unknown	NA	43.0	1	42.9	A042800015	0.0429			Unknown	NA	74.7	1	74.5	A042800015	0.0745		
100 mg/kg	C91060M	Unknown	NA	1.84	1	1.83	A042800016	<LOQ (0.00614 ug/g)			Unknown	NA	44.8	1	44.5	A042800016	0.0445		
	C91065M	Unknown	NA	2.72	1	2.74	A042800017	<LOQ (0.00614 ug/g)			Unknown	NA	28.6	1	28.9	A042800017	0.0289		
	C91070M	Unknown	NA	1.78	1	1.79	A042800018	<LOQ (0.00614 ug/g)			Unknown	NA	19.0	1	19.1	A042800018	0.0191		
	C91071M	Unknown	NA	2.52	1	2.53	A042800019	<LOQ (0.00614 ug/g)			Unknown	NA	43.7	1	43.8	A042800019	0.0438		
	C91072M	Unknown	NA	2.67	1	2.67	A042800022	<LOQ (0.00614 ug/g)			Unknown	NA	30.0	1	30.1	A042800022	0.0301		
	C91073M	Unknown	NA	0.85	1	0.849	A042800023	<LOQ (0.00614 ug/g)		NA	Unknown	NA	21.8	1	21.8	A042800023	0.0218		118
	C91076M	Unknown	NA	1.76	1	1.74	A042800024	<LOQ (0.00614 ug/g)	<LOQ	NA	Unknown	NA	20.3	1	20.1	A042800024	0.0201	0.0522	0.0614
Group 6	C91451F	Unknown	NA	1.66	1	1.63	A042800025	<LOQ (0.00614 ug/g)			Unknown	NA	0.00	1	0.00	A042800025	<LOQ (0.0123 ug/g)		
Mid-High Dose	C91453F	Unknown	NA	1.31	1	1.29	A042800026	<LOQ (0.00614 ug/g)			Unknown	NA	0.00	1	0.00	A042800026	<LOQ (0.0123 ug/g)		
Recovery	C91455F	Unknown	NA	2.46	1	2.43	A042800029	<LOQ (0.00614 ug/g)			Unknown	NA	0.00	1	0.00	A042800029	<LOQ (0.0123 ug/g)		
100 mg/kg	C91459F	Unknown	NA	0.380	1	0.375	A042800030	<LOQ (0.00614 ug/g)			Unknown	NA	0.00	1	0.00	A042800030	<LOQ (0.0123 ug/g)		
	C91462F	Unknown	NA	2.51	1	2.49	A042800031	<LOQ (0.00614 ug/g)			Unknown	NA	0.00	1	0.00	A042800031	<LOQ (0.0123 ug/g)		
	C91467F	Unknown	NA	2.71	1	2.68	A042800032	<LOQ (0.00614 ug/g)			Unknown	NA	0.00	1	0.00	A042800032	<LOQ (0.0123 ug/g)		
	C91473F	Unknown	NA	2.30	1	2.33	A042800033	<LOQ (0.00614 ug/g)			Unknown	NA	0.00	1	0.00	A042800033	<LOQ (0.0123 ug/g)		
	C91479F	Unknown	NA	1.62	1	1.62	A042800036	<LOQ (0.00614 ug/g)			Unknown	NA	0.00	1	0.00	A042800036	<LOQ (0.0123 ug/g)		
	C91480F	Unknown	NA	0.870	1	0.880	A042800037	<LOQ (0.00614 ug/g)			Unknown	NA	11.1	1	11.2	A042800037	<LOQ (0.0123 ug/g)		
	C91485F	Unknown	NA	2.21	1	2.25	A042800038	<LOQ (0.00614 ug/g)			Unknown	NA	7.07	1	7.21	A042800038	<LOQ (0.0123 ug/g)		
	C91487F	Unknown	NA	0.910	1	0.894	A042800039	<LOQ (0.00614 ug/g)		NA	Unknown	NA	0.00	1	0.00	A042800039	<LOQ (0.0123 ug/g)		NA
	C91488F	Unknown	NA	2.97	1	2.94	A042800040	<LOQ (0.00614 ug/g)	<LOQ	NA	Unknown	NA	13.1	1	12.9	A042800040	<LOQ (0.0123 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Value estimated, above the range of the calibration curve. LAC 03/14/01
** Data may be biased high as CCVs weren't within criteria. LAC 03/14/01
A = Sample was reextracted 03/15/01, analyzed 03/22/01, and the PFOS concentration reported from 03/15/01 extraction. LAC 03/26/01

Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 05/05/00, 05/18/00, 08/15/00, 08/21/00, 09/03/00, 03/26/01 LAC/CSH/MMH
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug g) to include std correction factors
new LOQ is 0.0245 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver	Filename:	See Below
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Soup 020199, Amelia 062498	Slope:	See Attachments
Instrument Software/Version:	MassLynx 3.3	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS	Box 00-012, 00-013	
Date of Analysis/Analyst:	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 4/28/00, 08/01/00, 08/14/00, 08/15/00, 03/22/01 MMH/MEE/IAS		
Date of Data Reduction/Analyst:	02/28/00, 03/01/00, 03/03/00, 03/06/00, 5/3/00, 5/4/00, 08/02/00, 08/15/00, 08/17/00, 03/23/01 IAS/MMH		

Sample Data

WEEK 105 RAT LIVER lot Unknown lot NB113047-80

Group Dose	Sample #	EtFOSE Purity Correction Factor	Surrogate Verified	EtFOSE Conc. ng/g	EtFOSE Dilution Factor	EtFOSE Calc. Conc. ng/g	Filename	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD	M556 Purity Correction Factor	Surrogate Verified	M556 Conc. ng/g	M556 Dilution Factor	M556 Calc. Conc. ng/g	Filename	Concentration of M556 ug/g or % Rec.	Mean M556 ug/g	RSD Std Dev MS/MSD RPD
Group 4	C90904M	Unknown	NA	469	1	437	A080100017	0.437			Unknown	NA	623	50	28960	A030100066	29.0		
Mid-High Dose	C90908M	Unknown	NA	293	1	276	A080100018	0.276			Unknown	NA	633	50	29855	A030100067	29.9		
100 mg/kg	C90910M	Unknown	NA	833	1	823	A080100019	0.823			Unknown	NA	619	50	30567	A030100068	30.6		
	C90911M	Unknown	NA	464	1	470	A080100020	0.470			Unknown	NA	648	50	32781	A030100069	32.8		
	C90916M	Unknown	NA	331	1	334	A080100021	0.334			Unknown	NA	642	50	32395	A030100072	32.4		
	C90926M	Unknown	NA	0.00	1	0.00	A080100024	<LOQ (0.0614 ug/g)			Unknown	NA	177	50	8469	A030100073	8.47		
	C90927M	Unknown	NA	313	1	291	A080100025	0.291			Unknown	NA	500	50	23216	A030100074	23.2		
	C90928M	Unknown	NA	314	1	309	A080100026	0.309			Unknown	NA	626	50	30818	A030100075	30.8		
	C90933M	Unknown	NA	363	1	334	A080100027	0.334			Unknown	NA	553	50	25401	A030100076	25.4		
	C90944M	Unknown	NA	321	1	323	A080100028	0.323			Unknown	NA	547	50	27552	A030100079	27.6		
	C90947M	Unknown	NA	310	1	308	A080100031	0.308			Unknown	NA	351	50	17406	A030100080	17.4		
	C90948M	Unknown	NA	232	1	235	A080100032	0.235			Unknown	NA	510	50	25822	A030100081	25.8		
	C90953M	Unknown	NA	213	1	207	A080100033	0.207			Unknown	NA	420	50	20389	A030100082	20.4		
	C90955M	Unknown	NA	466	1	466	A080100034	0.466			Unknown	NA	727	50	36299	A030100083	36.3		
	C90956M	Unknown	NA	314	1	310	A080100035	0.310			Unknown	NA	188	50	9313	A030100086	9.31		
	C90965M	Unknown	NA	1020	1	1024	A080100038	1.02		57.8	Unknown	NA	887.7	50	44554	A030100087	44.6		34.7
	C90968M	Unknown	NA	594	1	514	A080100039	0.514	0.395	0.229	Unknown	NA	866.6	50	37472	A030100088	37.5	27.1	9.41
Group 4	C91315F	Unknown	NA	439	1	442	A080100040	0.442			Unknown	NA	457	50	22980	A030100089	23.0		
Mid-High Dose	C91324F	Unknown	NA	377	2	755	A081500098	0.755			Unknown	NA	655	50	32766	A030100090	32.8		
100 mg/kg	C91327F	Unknown	NA	274	1	279	A080100042	0.279			Unknown	NA	193	50	9807	A030100093	9.81		
	C91332F	Unknown	NA	496	1	432	A080100045	0.432			Unknown	NA	740	50	32164	A030100094	32.2		
	C91335F	Unknown	NA	776	2	1389	A081500099	1.39			Unknown	NA	552	50	24677	A030100095	24.7		
	C91336F	Unknown	NA	479	1	469	A080100047	0.469			Unknown	NA	553	50	27071	A030100096	27.1		
	C91338F	Unknown	NA	916	1	917	A080100048	0.917			Unknown	NA	614	50	30744	A030100097	30.7		
	C91340F	Unknown	NA	416	1	408	A080100049	0.408			Unknown	NA	636	50	31154	A030100100	31.2		
	C91341F	Unknown	NA	746	1	721	A080100052	0.721			Unknown	NA	542	50	26187	A030100101	26.2		
	C91342F	Unknown	NA	782	1	777	A080100053	0.777			Unknown	NA	839	50	41708	A030100102	41.7		
	C91356F	Unknown	NA	184	1	182	A080100054	0.182			Unknown	NA	228	50	11248	A030100103	11.2		
	C91357F	Unknown	NA	842	1	843	A080100055	0.843			Unknown	NA	536	50	26842	A030100104	26.8		
	C91360F	Unknown	NA	611	10	6034	A081500100	6.034			Unknown	NA	507	50	25014	A030100107	25.0		
	C91361F	Unknown	NA	1106	1	1106	A080100059	1.11 #			Unknown	NA	889	50	44467	A030100108	44.5		
	C91362F	Unknown	NA	213	1	211	A080100060	0.211			Unknown	NA	480	50	23712	A030100109	23.7		
	C91372F	Unknown	NA	518	1	522	A080100061	0.522			Unknown	NA	789	50	39723	A030100110	39.7		
	C91376F	Unknown	NA	774	1	734	A080100062	0.734			Unknown	NA	717	50	33977	A030100111	34.0		
	C91378F	Unknown	NA	1062	2	2117	A030400085	2.12 **		131	Unknown	NA	475	50	23656	A030100114	23.7		31.1
	C91380F	Unknown	NA	549	1	557	A080100066	0.557	0.994	1.30	Unknown	NA	656	50	33352	A030100115	33.4	28.5	8.86
Group 6	C91047M	Unknown	NA	0.00	1	0.00	A081400028	<LOQ (0.0614 ug/g)			Unknown	NA	2.70	1	2.73	A081400028	<LOQ (0.00615 ug/g)		
Mid-High Dose	C91054M	Unknown	NA	0.00	1	0.00	A081400029	<LOQ (0.0614 ug/g)			Unknown	NA	3.18	1	3.13	A081400029	<LOQ (0.00615 ug/g)		
Recovery	C91056M	Unknown	NA	0.00	1	0.00	A081400030	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800015	<LOQ (0.123 ug/g)		
100 mg/kg	C91060M	Unknown	NA	0.00	1	0.00	A081400031	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800016	<LOQ (0.123 ug/g)		
	C91065M	Unknown	NA	0.00	1	0.00	A081400034	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800017	<LOQ (0.123 ug/g)		
	C91070M	Unknown	NA	0.00	1	0.00	A081400035	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800018	<LOQ (0.123 ug/g)		
	C91071M	Unknown	NA	0.00	1	0.00	A081400036	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800019	<LOQ (0.123 ug/g)		
	C91072M	Unknown	NA	0.00	1	0.00	A081400037	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800022	<LOQ (0.123 ug/g)		
	C91073M	Unknown	NA	0.00	1	0.00	A081400038	<LOQ (0.0614 ug/g)		NA	Unknown	NA	0.00	1	0.00	A042800023	<LOQ (0.123 ug/g)		NA
	C91076M	Unknown	NA	0.00	1	0.00	A081400041	<LOQ (0.0614 ug/g)	<LOQ	NA	Unknown	NA	0.00	1	0.00	A042800024	<LOQ (0.123 ug/g)	<LOQ	NA
Group 6	C91451F	Unknown	NA	0.00	1	0.00	A081400042	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800025	<LOQ (0.123 ug/g)		
Mid-High Dose	C91453F	Unknown	NA	0.00	1	0.00	A081400043	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800026	<LOQ (0.123 ug/g)		
Recovery	C91455F	Unknown	NA	0.00	1	0.00	A081400044	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800029	<LOQ (0.123 ug/g)		
100 mg/kg	C91459F	Unknown	NA	0.00	1	0.00	A081400045	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800030	<LOQ (0.123 ug/g)		
	C91462F	Unknown	NA	0.00	1	0.00	A081400048	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800031	<LOQ (0.123 ug/g)		
	C91467F	Unknown	NA	0.00	1	0.00	A081400049	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800032	<LOQ (0.123 ug/g)		
	C91473F	Unknown	NA	0.00	1	0.00	A081400050	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A042800033	<LOQ (0.123 ug/g)		
	C91479F	Unknown	NA	0.00	1	0.00	A081400027	<LOQ (0.0614 ug/g)			Unknown	NA	1.36	1	1.36	A081400027	<LOQ (0.00615 ug/g)		
	C91480F	Unknown	NA	0.00	1	0.00	A081400051	<LOQ (0.0614 ug/g)			Unknown	NA	0.00	1	0.00	A081400051	<LOQ (0.00615 ug/g)		
	C91485F	Unknown	NA	0.00	1	0.00	A081400052	<LOQ (0.0614 ug/g)			Unknown	NA	4.62	1	4.71	A081400052	<LOQ (0.00615 ug/g)		
	C91487F	Unknown	NA	0.00	1	0.00	A081400055	<LOQ (0.0614 ug/g)		NA	Unknown	NA	1.21	1	1.19	A081400055	<LOQ (0.00615 ug/g)		NA
	C91488F	Unknown	NA	0.00	1	0.00	A081400056	<LOQ (0.0614 ug/g)	<LOQ	NA	Unknown	NA	2.96	1	2.93	A081400056	<LOQ (0.00615 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Value estimated, above the range of the calibration curve. LAC 03/14/01
** Data may be biased high as CCVs weren't within criteria. LAC 03/14/01
A = Sample was reextracted 03/15/01, analyzed 03/22/01, and the PFOS concentration reported from 03/15/01 extraction. LAC 03/26/01

Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 05/05/00, 05/18/00, 08/15/00, 08/21/00, 09/03/00, 03/26/01 LAC/CSH/MMH
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0.0245 ug/g. LAC 02/19/01

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver — Filename: See Below
Method/Revision: ETS-8-6.0 & ETS-8-7.0 — R-Squared Value: See Attachments
Analytical Equipment System Number: Soup 020199, Amelia 062498 — Slope: See Attachments
Instrument Software/Version: MassLynx 3.3 — Y-Intercept: See Attachments
Date of Extraction/Analyst: 02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS — Box 00-012, 00-013
Date of Analysis/Analyst: 02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 4/28/00, 08/01/00, 08/14/00, 08/15/00, 03/22/01 MMH/MEE/IAS
Date of Data Reduction/Analyst: 02/28/00, 03/01/00, 03/03/00, 03/06/00, 5/3/00, 5/4/00, 08/02/00, 08/15/00, 08/17/00, 03/23/01 IAS/MMH
Sample Data

WEEK 105 RAT LIVER — lot 529

Group Dose	Sample #	PFOSEA Purity Correction Factor	Surrogate Verified	PFOSEA Conc. ng/g	PFOSEA Dilution Factor	PFOSEA Calc. Conc. ng/g	Filename	Concentration of PFOSEA ug/g or % Rec.	Mean PFOSEA ug/g	RSD Std Dev MS/MSD RPD
Group 4	C90904M	Unknown	NA	0.00	1	0.00	A080100017	<LOQ (0.0123 ug/g)		
Mid-High Dose	C90908M	Unknown	NA	21.7	1	20.5	A080100018	0.0205		
100 mg/kg	C90910M	Unknown	NA	34.1	1	33.7	A080100019	0.0337		
	C90911M	Unknown	NA	0.00	1	0.00	A080100020	<LOQ (0.0123 ug/g)		
	C90916M	Unknown	NA	2.39	1	2.41	A080100021	<LOQ (0.0123 ug/g)		
	C90926M	Unknown	NA	50.4	1	48.3	A080100024	0.0483		
	C90927M	Unknown	NA	3.75	1	3.48	A080100025	<LOQ (0.0123 ug/g)		
	C90928M	Unknown	NA	31.1	1	30.7	A080100026	0.0307		
	C90933M	Unknown	NA	31.7	1	29.1	A080100027	0.0291		
	C90944M	Unknown	NA	54.4	1	54.8	A080100028	0.0548		
	C90947M	Unknown	NA	0.00	1	0.00	A080100031	<LOQ (0.0123 ug/g)		
	C90948M	Unknown	NA	26.0	1	26.3	A080100032	0.0263		
	C90953M	Unknown	NA	0.00	1	0.00	A080100033	<LOQ (0.0123 ug/g)		
	C90955M	Unknown	NA	0.00	1	0.00	A080100034	<LOQ (0.0123 ug/g)		
	C90956M	Unknown	NA	117	1	115	A080100035	0.115		
	C90965M	Unknown	NA	235	1	236	A080100038	0.236		139
	C90968M	Unknown	NA	3.03	1	2.62	A080100039	<LOQ (0.0123 ug/g)	0.0408	0.0566
Group 4	C91315F	Unknown	NA	57.8	1	58.1	A080100040	0.0581		
Mid-High Dose	C91324F	Unknown	NA	32.7	1	32.7	A080100041	0.0327		
100 mg/kg	C91327F	Unknown	NA	32.7	1	33.3	A080100042	0.0333		
	C91332F	Unknown	NA	2.34	1	2.03	A080100045	<LOQ (0.0123 ug/g)		
	C91335F	Unknown	NA	4.61	1	4.12	A080100046	<LOQ (0.0123 ug/g)		
	C91336F	Unknown	NA	10.7	1	10.5	A080100047	<LOQ (0.0123 ug/g)		
	C91338F	Unknown	NA	13.0	1	13.0	A080100048	0.0130		
	C91340F	Unknown	NA	24.9	1	24.4	A080100049	0.0244		
	C91341F	Unknown	NA	10.7	1	10.3	A080100052	<LOQ (0.0123 ug/g)		
	C91342F	Unknown	NA	10.9	1	10.8	A080100053	<LOQ (0.0123 ug/g)		
	C91356F	Unknown	NA	56.7	1	56.0	A080100054	0.0560		
	C91357F	Unknown	NA	11.5	1	11.5	A080100055	<LOQ (0.0123 ug/g)		
	C91360F	Unknown	NA	25.6	1	25.2	A080100056	0.0252		
	C91361F	Unknown	NA	23.2	1	23.2	A080100059	0.0232		
	C91362F	Unknown	NA	2.86	1	2.82	A080100060	<LOQ (0.0123 ug/g)		
	C91372F	Unknown	NA	26.6	1	26.8	A080100061	0.0268		
	C91376F	Unknown	NA	25.6	1	24.3	A080100062	0.0243		
	C91378F	Unknown	NA	28.7	1	28.6	A080100063	0.0286		57.6
	C91380F	Unknown	NA	25.6	1	26.0	A080100066	0.0260	0.0241	0.0139
Group 6	C91047M	Unknown	NA	0.00	1	0.00	A042800043	<LOQ (0.0307 ug/g)		
Mid-High Dose	C91054M	Unknown	NA	0.00	1	0.00	A042800044	<LOQ (0.0307 ug/g)		
Recovery	C91056M	Unknown	NA	0.00	1	0.00	A042800015	<LOQ (0.0307 ug/g)		
100 mg/kg	C91060M	Unknown	NA	0.00	1	0.00	A042800016	<LOQ (0.0307 ug/g)		
	C91065M	Unknown	NA	0.00	1	0.00	A042800017	<LOQ (0.0307 ug/g)		
	C91070M	Unknown	NA	0.00	1	0.00	A042800018	<LOQ (0.0307 ug/g)		
	C91071M	Unknown	NA	0.00	1	0.00	A042800019	<LOQ (0.0307 ug/g)		
	C91072M	Unknown	NA	0.00	1	0.00	A042800022	<LOQ (0.0307 ug/g)		
	C91073M	Unknown	NA	0.00	1	0.00	A042800023	<LOQ (0.0307 ug/g)		NA
	C91076M	Unknown	NA	0.00	1	0.00	A042800024	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 6	C91451F	Unknown	NA	0.00	1	0.00	A042800025	<LOQ (0.0307 ug/g)		
Mid-High Dose	C91453F	Unknown	NA	0.00	1	0.00	A042800026	<LOQ (0.0307 ug/g)		
Recovery	C91455F	Unknown	NA	0.00	1	0.00	A042800029	<LOQ (0.0307 ug/g)		
100 mg/kg	C91459F	Unknown	NA	0.00	1	0.00	A042800030	<LOQ (0.0307 ug/g)		
	C91462F	Unknown	NA	0.00	1	0.00	A042800031	<LOQ (0.0307 ug/g)		
	C91467F	Unknown	NA	0.00	1	0.00	A042800032	<LOQ (0.0307 ug/g)		
	C91473F	Unknown	NA	0.00	1	0.00	A042800033	<LOQ (0.0307 ug/g)		
	C91479F	Unknown	NA	0.00	1	0.00	A042800036	<LOQ (0.0307 ug/g)		
	C91480F	Unknown	NA	0.00	1	0.00	A042800037	<LOQ (0.0307 ug/g)		
	C91485F	Unknown	NA	0.00	1	0.00	A042800038	<LOQ (0.0307 ug/g)		
	C91487F	Unknown	NA	0.00	1	0.00	A042800039	<LOQ (0.0307 ug/g)		NA
	C91488F	Unknown	NA	0.00	1	0.00	A042800040	<LOQ (0.0307 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO).
PFOSEA = Perfluorooctane sulfonyl ethylamide

Value estimated, above the range of the calibration curve. LAC 03/14/01
** Data may be biased high as CCVs weren't within criteria. LAC 03/14/01
A = Sample was reextracted 03/15/01, analyzed 03/22/01, and the PFOS concentration reported from 03/15/01 extraction. LAC 03/26/01

Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 05/05/00, 05/18/00, 08/15/00, 08/21/00, 09/03/00, 03/26/01 LAC/CSH/MMH
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0.0245 ug/g. LAC 02/19/01

Study: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Soup 020199, Amelia 062498
Instrument Software/Version: MassLynx 3.3
Date of Extraction/Analyst: 02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS
Date of Analysis/Analyst: 02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 4/28/00, 08/01/00, 08/14/00, 08/15/00, 03/22/01 MMH/MEE/IAS
Date of Data Reduction/Analyst: 02/28/00, 03/01/00, 03/03/00, 03/06/00, 5/3/00, 5/4/00, 08/02/00, 08/15/00, 08/17/00, 03/23/01 IAS/MMH

Filename: See Attachments
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 105 RAT LIVER

Group Dose	Sample #	PFOS Calc. Conc. ng/g	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std Dev MS/MSD RPD	PFOSA Calc. Conc. ng/g	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std Dev MS/MSD RPD	PFOSAA Calc. Conc. ng/g	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std Dev MS/MSD RPD	EtFOSE Calc. Conc. ng/g	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std Dev MS/MSD RPD
Group 4	C90904M	323257	323			14679	14.7			21358	21.4			437	0.437		
Mid-High Dose	C90908M	313374	313			14920	14.9			18476	18.5			276	0.276		
100 mg/kg	C90910M	98979	99.0			12376	12.4			44847	44.8			823	0.823		
	C90911M	718021	718			18544	18.5			35222	35.2			470	0.470		
	C90916M	236132	236			17812	17.8			23948	23.9			334	0.334		
	C90926M	120112	120			4061	4.06			1784	1.78			0.00	<LOQ (0.0614 ug/g)		
	C90927M	26240	26.2			11539	11.5			15462	15.5			291	0.291		
	C90928M	628332	628			17644	17.6			40328	40.3			309	0.309		
	C90933M	153299	153			13218	13.2			18511	18.5			334	0.334		
	C90944M	565043	565 A			15070	15.1			23008	23.0			323	0.323		
	C90947M	295355	295			9823	9.82			17773	17.8			308	0.308		
	C90948M	186556	187			9411	9.41			11445	11.4			235	0.235		
	C90953M	60324	60.3			10031	10.0			18340	18.3			207	0.207		
	C90955M	265167	265			14511	14.5			27358	27.4			466	0.466		
	C90956M	201182	201 A			8680	8.68			4725	4.72			310	0.310		
	C90965M	689313	689		70.4	20448	20.4		31.3	51758	51.8		56.5	1024	1.02		57.8
	C90968M	445034	445	313	220	16616	16.6	13.5	4.23	26905	26.9	23.6	13.3	514	0.514	0.395	0.229
	C91315F	230009	230			12800	12.8			15794	15.8			442	0.442		
	C91324F	509159	509			16226	16.2			28252	28.3			755	0.755		
	C91327F	31737	31.7			10627	10.6			38590	38.6			279	0.279		
	C91332F	377842	378			17062	17.1			25423	25.4			432	0.432		
	C91335F	220157	220			14551	14.6			18251	18.3			1389	1.389		
	C91336F	329837	330			14949	14.9			19676	19.7			469	0.469		
	C91338F	341389	341			18058	18.1			20954	21.0			917	0.917		
	C91340F	579769	580			16126	16.1			26299	26.3			408	0.408		
	C91341F	242581	243			15274	15.3			25334	25.3			721	0.721		
	C91342F	355109	355			16810	16.8			33469	33.5			777	0.777		
	C91356F	42389	42.4			6157	6.2			17230	17.2			182	0.182		
	C91357F	192963	193			16831	16.8			20686	20.7			843	0.843		
	C91360F	206612	207			17955	18.0			28490	28.5			6034	6.03		
	C91361F	712319	712			22322	22.3			31554	31.6			1106	1.11 #		
	C91362F	86896	86.9			10883	10.9			30022	30.0			211	0.211		
	C91372F	257883	258			19454	19.5			42332	42.3			522	0.522		
	C91376F	253932	254			16296	16.3			27417	27.4			734	0.734		
	C91378F	340658	341		57.7	18952	19.0		23.1	30114	30.1		26.3	2117	2.12 **		131.0
	C91380F	324616	325	297	171	15385	15.4	15.6	3.61	30712	30.7	26.9	7.08	557	0.557	0.994	1.303
Group 6	C91047M	2034	2.03			2.17	<LOQ (0.00614 ug/g)			220	0.220			0.00	<LOQ (0.0614 ug/g)		
Mid-High Dose	C91054M	198	0.198			2.46	<LOQ (0.00614 ug/g)			19.5	0.0195			0.00	<LOQ (0.0614 ug/g)		
Recovery	C91056M	10494	10.5			42.9	0.0429			74.5	0.0745			0.00	<LOQ (0.0614 ug/g)		
100 mg/kg	C91060M	2006	2.01			1.83	<LOQ (0.00614 ug/g)			44.5	0.0445			0.00	<LOQ (0.0614 ug/g)		
	C91065M	36.5	0.0365			2.74	<LOQ (0.00614 ug/g)			28.9	0.0289			0.00	<LOQ (0.0614 ug/g)		
	C91070M	205	0.205			1.79	<LOQ (0.00614 ug/g)			19.1	0.0191			0.00	<LOQ (0.0614 ug/g)		
	C91071M	41.1	0.0411			2.53	<LOQ (0.00614 ug/g)			43.8	0.0438			0.00	<LOQ (0.0614 ug/g)		
	C91072M	584	0.584			2.67	<LOQ (0.00614 ug/g)			30.1	0.0301			0.00	<LOQ (0.0614 ug/g)		
	C91073M	83.1	0.0831		164	0.849	<LOQ (0.00614 ug/g)		NA	21.8	0.0218		118	0.00	<LOQ (0.0614 ug/g)		NA
	C91076M	5024.960	5.024960 ##	2.05	3.36	1.74	<LOQ (0.00614 ug/g)	<LOQ	NA	20.1	0.0201	0.0522	0.0614	0.00	<LOQ (0.0614 ug/g)	<LOQ	NA
	C91451F	15396	15.4			1.63	<LOQ (0.00614 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91453F	4525	4.53			1.29	<LOQ (0.00614 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91455F	18803	18.8			2.43	<LOQ (0.00614 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91459F	49.4	0.0494			0.37	<LOQ (0.00614 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91462F	17533	17.5			2.49	<LOQ (0.00614 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91467F	137	0.137			2.68	<LOQ (0.00614 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91473F	13901	13.9			2.33	<LOQ (0.00614 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91479F	20.7	<LOQ (0.0245 ug/g)			1.62	<LOQ (0.00614 ug/g)			0.00	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91480F	149	0.149			0.880	<LOQ (0.00614 ug/g)			11.2	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91485F	10328.58	10.33			2.25	<LOQ (0.00614 ug/g)			7.21	<LOQ (0.0123 ug/g)			0.00	<LOQ (0.0614 ug/g)		
	C91487F	62.8	0.0628		99.0	0.894	<LOQ (0.00614 ug/g)		NA	0.00	<LOQ (0.0123 ug/g)		NA	0.00	<LOQ (0.0614 ug/g)		NA
	C91488F	11369	11.4	7.69	7.62	2.94	<LOQ (0.00614 ug/g)	<LOQ	NA	12.9	<LOQ (0.0123 ug/g)	<LOQ	NA	0.00	<LOQ (0.0614 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Value estimated, above the range of the calibration curve. LAC 03/14/01
** Data may be biased high as CCVs weren't within criteria. LAC 03/14/01
A = Sample was reextracted 03/15/01, analyzed 03/22/01, and the PFOS concentration reported from 03/15/01 extraction. LAC 03/26/01

Date Entered/Anal 03/14/00, 03/16/00, 03/17/00, 05/05/00, 05/18/00, 08/15/00, 08/21/00, 09/03/00, 03/26/01 LAC/CSH/MMH
Date Verified/Ana 03/09/01 hoj
Purity Entered/Ver 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0.0245 ug/g. LAC 02/19/01

Study:	104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver	Filename:	See Attachments
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Soup 020199, Amelia 062498	Slope:	See Attachments
Instrument Software/Version:	MassLynx 3.3	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	02/14/00, 02/16/00, 03/15/01 RWW/SAL/KJS	Box 00-012, 00-013	
Date of Analysis/Analyst:	02/21/00, 02/29/00, 03/01/00, 03/03/00, 03/04/00, 4/28/00, 08/01/00, 08/14/00, 08/15/00, 03/22/01 MMH/MEE/IAS		
Date of Data Reduction/Analyst:	02/28/00, 03/01/00, 03/03/00, 03/06/00, 5/3/00, 5/4/00, 08/02/00, 08/15/00, 08/17/00, 03/23/01 IAS/MMH		

Sample Data

WEEK 105 RAT LIVER

Group Dose	Sample #	M556 Calc. Conc. ng/g	Concentration of M556 ug/g or % Rec.	Mean M556 ug/g	RSD Std Dev MS/MSD RPD	PFOSEA Calc. Conc. ng/g	Concentration of PFOSEA ug/g or % Rec.	Mean PFOSEA ug/g	RSD Std Dev MS/MSD RPD
Group 4	C90904M	28960	29.0			0.00	<LOQ (0.0123 ug/g)		
Mid-High Dose	C90908M	29855	29.9			20.5	0.0205		
100 mg/kg	C90910M	30567	30.6			33.7	0.0337		
	C90911M	32781	32.8			0.00	<LOQ (0.0123 ug/g)		
	C90916M	32395	32.4			2.41	<LOQ (0.0123 ug/g)		
	C90926M	8469	8.47			48.3	0.0483		
	C90927M	23216	23.2			3.48	<LOQ (0.0123 ug/g)		
	C90928M	30818	30.8			30.7	0.0307		
	C90933M	25401	25.4			29.1	0.0291		
	C90944M	27552	27.6			54.8	0.0548		
	C90947M	17406	17.4			0.00	<LOQ (0.0123 ug/g)		
	C90948M	25822	25.8			26.3	0.0263		
	C90953M	20389	20.4			0.00	<LOQ (0.0123 ug/g)		
	C90955M	36299	36.3			0.00	<LOQ (0.0123 ug/g)		
	C90956M	9313	9.31			115	0.115		
	C90965M	44554	44.6		34.7	236	0.236		139
	C90968M	37472	37.5	27.1	9.4	2.62	<LOQ (0.0123 ug/g)	0.0408	0.0566
Group 4	C91315F	22980	23.0			58.1	0.0581		
Mid-High Dose	C91324F	32766	32.8			32.7	0.0327		
100 mg/kg	C91327F	9807	9.81			33.3	0.0333		
	C91332F	32164	32.2			2.03	<LOQ (0.0123 ug/g)		
	C91335F	24677	24.7			4.12	<LOQ (0.0123 ug/g)		
	C91336F	27071	27.1			10.5	<LOQ (0.0123 ug/g)		
	C91338F	30744	30.7			13.0	0.0130		
	C91340F	31154	31.2			24.4	0.0244		
	C91341F	26187	26.2			10.3	<LOQ (0.0123 ug/g)		
	C91342F	41708	41.7			10.8	<LOQ (0.0123 ug/g)		
	C91356F	11248	11.2			56.0	0.0560		
	C91357F	26842	26.8			11.5	<LOQ (0.0123 ug/g)		
	C91360F	25014	25.0			25.2	0.0252		
	C91361F	44467	44.5			23.2	0.0232		
	C91362F	23712	23.7			2.82	<LOQ (0.0123 ug/g)		
	C91372F	39723	39.7			26.8	0.0268		
	C91376F	33977	34.0			24.3	0.0243		
	C91378F	23656	23.7		31.1	28.6	0.0286		57.6
	C91380F	33352	33.4	28.5	8.86	26.0	0.0260	0.0241	0.0139
Group 6	C91047M	2.73	<LOQ (0.00615 ug/g)			0.00	<LOQ (0.0307 ug/g)		
Mid-High Dose	C91054M	3.13	<LOQ (0.00615 ug/g)			0.00	<LOQ (0.0307 ug/g)		
Recovery	C91056M	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
100 mg/kg	C91060M	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91065M	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91070M	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91071M	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91072M	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91073M	0.00	<LOQ (0.123 ug/g)		NA	0.00	<LOQ (0.0307 ug/g)		NA
	C91076M	0.00	<LOQ (0.123 ug/g)	<LOQ	NA	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA
Group 6	C91451F	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
Mid-High Dose	C91453F	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
Recovery	C91455F	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
100 mg/kg	C91459F	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91462F	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91467F	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91473F	0.00	<LOQ (0.123 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91479F	1.36	<LOQ (0.00615 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91480F	0.00	<LOQ (0.00615 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91485F	4.71	<LOQ (0.00615 ug/g)			0.00	<LOQ (0.0307 ug/g)		
	C91487F	1.19	<LOQ (0.00615 ug/g)		NA	0.00	<LOQ (0.0307 ug/g)		NA
	C91488F	2.93	<LOQ (0.00615 ug/g)	<LOQ	NA	0.00	<LOQ (0.0307 ug/g)	<LOQ	NA

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfuoroocotane sulfonyl ethylamide

Value estimated, above the range of the calibration curve. LAC 03/14/01
** Data may be biased high as CCVs weren't within criteria. LAC 03/14/01
A = Sample was reextracted 03/15/01, analyzed 03/22/01, and the PFOS concentration reported from 03/15/01 extraction. LAC 03/26/01

Date Entered/Analyst: 03/14/00, 03/16/00, 03/17/00, 05/05/00, 05/18/00, 08/15/00, 08/21/00, 09/03/00, 03/26/01 LAC/CSH/MMH
Date Verified/Analyst: 03/09/01 hoj
Purity Entered/Verified: 02/19/01 LAC

Corrected PFOS LOQ (0.0306 ug/g) to include std correction factors
new LOQ is 0 0245 ug/g. LAC 02/19/01

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
LRN-U2103

Appendix F: Example Calculations

Formula Used for Sera Analyses in Study FACT TOX-001

$$\text{AR (ng/mL)} \times \text{DF} \times \text{SC} \times \text{PF} \times \frac{\text{FV (mL)}}{\text{EV (mL)}} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \textbf{Reported Concentration (µg/mL)}$$

Calculation Used for Group 2, Week 105, Animal ID C90783M, determination of PFOS

$$189.2\ \text{ng/mL} \times 10 \times 0.9275 \times 0.8640 \times \frac{1\ \text{mL}}{1\ \text{mL}} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \mathbf{1.52\ \mu g/mL}$$

AR—Analytical result from MassLynx summary
DF—Dilution factor
SC—PFOS salt correction constant (0.9275)
PF—Purity correction factor
FV—Final extract volume (1.0 mL unless otherwise noted)
EV—Volume of sera extracted

Formula Used for Liver Analyses in Study FACT TOX-001

$$\text{AR (ng/g)} \times \frac{\partial\ \text{curve}^{(1)}}{\partial\ \text{sample}} \times \text{SC} \times \text{PF} \times \text{DF} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \textbf{Reported Concentration (µg/g)}$$

(1) ∂ curve is assumed to be: 1 g liver / 5 mL H_2O

Calculation Used for Group 2, Week 105, Animal ID C90783M, determination of PFOS

$$488.16\ \text{ng/g} \times \frac{1\ \text{g/ 5 mL}}{0.9895\ \text{g/ 5 mL}} \times 0.9275 \times 0.8640 \times 20 \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \mathbf{7.91\ \mu g/g}$$

AR—Analytical result from MassLynx summary
∂ curve—Density of the liver standard curve, assumed to be 1g liver/ 5 ml water
∂ sample—Density of the liver sample (g sample/ 5 mL H_2O)
SC—PFOS salt correction constant (0.9275)
PF—Purity correction factor
DF—Dilution factor

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
LRN-U2103

Appendix G: Interim Certificates of Analysis

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

INTERIM CERTIFICATE OF ANALYSIS

Revision 1(9/7/00)

Centre Analytical Laboratories COA Reference #: 023-018B
3M Product: PFOS, Lot 171
Reference #: SD-009
Purity: 86.4%

Test Name	Specifications	Result
Purity[1]		**86.4%**
Appearance	White Crystalline Powder	Conforms
Identification NMR		Positive
Metals (ICP/MS) 1. Calcium 2. Magnesium 3. Sodium 4. Potassium[2] 5. Nickel 6. Iron 7. Manganese		1. 0.017 wt./wt.% 2. 0.007 wt./wt.% 3. 1.355 wt./wt.% 4. 6.552 wt./wt.% 5. 0.003 wt./wt.% 6. 0.004 wt./wt.% 7. <0.001 wt./wt.%
Total % Impurity (NMR)		1.00 wt./wt.%
Total % Impurity (LC/MS)		10.60 wt./wt.%
Total % Impurity (GC/MS)		None Detected
Related Compounds – POAA		0.30 wt./wt.%
Residual Solvents (TGA)		None Detected
Purity by DSC		Not Applicable[3]
Inorganic Anions (IC) 1. Chloride 2. Fluoride 3. Bromide 4. Nitrate 5. Nitrite 6. Phosphate 7. Sulfate[4]		1. <0.015 wt./wt.% 2. 0.27 wt./wt.% 3. <0.040 wt./wt.% 4. <0.009 wt./wt.% 5. <0.006 wt./wt.% 6. <0.007 wt./wt.% 7. 8.82 wt./wt.%
Organic Acids [5] (IC) 1. TFA 2. PFPA 3. HFBA 4. NFPA		1. <0.1 wt./wt.% 2. <0.1 wt./wt.% 3. <0.1 wt./wt.% 4. <0.25 wt./wt.%
Elemental Analysis[6]: 1. Carbon 2. Hydrogen 3. Nitrogen 4. Sulfur 5. Fluorine	1. Theoretical Value = 17.8% 2. Theoretical Value = 0% 3. Theoretical Value = 0% 4. Theoretical Value = 5.95% 5. Theoretical Value = 60%	1. 12.08 wt./wt.% 2. 0.794 wt./wt.% 3. 1.61 wt./wt.% 4. 10.1 wt./wt.% 5. 50.4 wt./wt.%

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3048 Research Drive State College, PA 16801
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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-018B

Date of Last Analysis: 08/31/00

Expiration Date: 08/31/01

Storage Conditions: Frozen ≤-10°C

Re-assessment Date: 08/31/01

[1]Purity = 100% - (sum of metal impurities, 1.39% +LC/MS impurities, 10.60%+Inorganic Fluoride, 0.27%+NMR impurities, 1.00%+ POAA, 0.30%)

Total impurity from all tests = 13.56%

Purity = 100% - 13.56% = 86.4%

[2]Potassium is expected in this salt form and is therefore not considered an impurity.

[3]Purity by DSC is generally not applicable to materials of low purity. No endotherm was observed for this sample.

[4]Sulfur in the sample appears to be converted to SO_4 and hence detected using the inorganic anion method conditions. The anion result agrees well with the sulfur determination in the elemental analysis, lending confidence to this interpretation. Based on the results, the SO_4 is not considered an impurity.

[5]TFA	Trifluoroacetic acid
HFBA	Heptafluorobutyric acid
NFPA	Nonofluoropentanoic acid
PFPA	Pentafluoropropanoic acid

[6]Theoretical value calculations based on the empirical formula, $C_8F_{17}SO_3^-K^+$ (MW=538)

This work was conducted under EPA Good Laboratory Practice Standards (40 CFR 160).

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-018B

LC/MS Purity Profile:

Impurity	wt./wt. %
C4	1.03
C5	1.56
C6	6.38
C7	1.63
Total	**10.60**

Note: The C4 and C6 values were calculated using the C4 and C6 standard calibration curves, respectively. The C5 value was calculated using the average response factors from the C4 and C6 standard curves. Likewise, the C7 value was calculated using the average response factors from the C6 and C8 standard curves.

Prepared By: David S. Bell 9/10/00
Scientist, Centre Analytical Laboratories Date

Reviewed By: John M Flaherty 9/11/00
John Flaherty Date
Laboratory Manager, Centre Analytical Laboratories

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-2
3M Product: EtFOSE-OH
Test Control Reference #: TCR-00017-52
Purity: 97.4%

Test Name	Specifications	Result
Purity[1]		**97.4%**
Appearance	Yellow-white, waxy solid	Conforms
Identification NMR		Positive
Metals (ICP/MS) 1. Calcium 2. Magnesium 3. Sodium 4. Potassium 5. Nickel 6. Iron 7. Manganese		 1. <0.001 wt./wt.% 2. <0.001 wt./wt.% 3. <0.001 wt./wt.% 4. <0.001 wt./wt.% 5. <0.001 wt./wt.% 6. <0.001 wt./wt.% 7. <0.001 wt./wt.%
Total % Impurity (NMR)		1.26 wt./wt.%
Total % Impurity (LC/MS)		None Quantified
Total % Impurity (GC/MS)		1.29 wt./wt.%
Related Compounds – POAA		0.10 wt./wt.%
Residual Solvents (TGA)		None Detected
Purity by DSC		90.3 wt./wt.%.
Inorganic Anions (IC) 1. Chloride 2. Fluoride 3. Bromide 4. Nitrate 5. Nitrite 6. Phosphate 7. Sulfate		 1. <0.015 wt./wt.% 2. <0.005 wt./wt.% 3. <0.040 wt./wt.% 4. <0.009 wt./wt.% 5. <0.006 wt./wt.% 6. <0.007 wt./wt.% 7. <0.154 wt./wt.%
Organic Acids[2] (IC) 1. TFA 2. PFPA 3. HFBA 4. NFPA		 1. <0.1 wt./wt.% 2. <0.1 wt./wt.% 3. <0.1 wt./wt.% 4. <0.25 wt./wt.%
Elemental Analysis[3]: 1. Carbon 2. Hydrogen 3. Nitrogen 4. Sulfur 5. Fluorine	 1. Theoretical Value = 25.2% 2. Theoretical Value = 1.75% 3. Theoretical Value = 2.45% 4. Theoretical Value = 5.60% 5. Theoretical Value = 56.6%	 1. 25.04 wt./wt.% 2. 1.69 wt./wt.% 3. 2.61 wt./wt.% 4. 8.88 wt./wt.% 5. 56.8 wt./wt.%

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-2
3M Product: EtFOSE-OH
Test Control Reference #: TCR-00017-52

Date of Last Analysis: 11/26/00

Expiration Date: 11/26/01

Storage Conditions: <-10 °C

Re-assessment Date: 11/26/01

[1]Purity = 100% - (total NMR impurities, 1.26% + GC/MS impurities, 1.29 + POAA, 0.10%)

Total impurity from all tests = 2.65%
Purity = 100% - 2.65% = 97.4%

[2]TFA	Trifluoroacetic acid
HFBA	Heptafluorobutyric acid
NFPA	Nonafluoropentanoic acid
PFPA	Pentafluoropropanoic acid

[3]Theoretical value calculations based on the empirical formula, $C_{12}H_{10}F_{17}NO_3S$ (MW=571)

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-2
3M Product: EtFOSE-OH
Test Control Reference #: TCR-00017-52

GC/MS Purity Profile

Peak #	Retention Time (min)	Identity	% Impurity
1	13.934	PFOSDEA	0.36
2	17.307	C7	0.93
Total	-	-	**1.29**

This work was conducted under EPA Good Laboratory Practice Standards (40 CFR 160).

Prepared By: _____ 11/27/00
David S. Bell
Scientist
Centre Analytical Laboratories
Date

Reviewed By: _____ 11/27/00
John Flaherty
Laboratory Manager
Centre Analytical Laboratories
Date

Centre Analytical Laboratories, Inc.
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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-1
3M Product: EtFOSE-OH
Test Control Reference #: SD-013
Purity: 88.9%

Test Name	Specifications	Result
Purity[1]		**88.9%**
Appearance	Yellow-white, waxy solid	Conforms
Identification NMR		Positive
Metals (ICP/MS) 1. Calcium 2. Magnesium 3. Sodium 4. Potassium 5. Nickel 6. Iron 7. Manganese		1. <0.001 wt./wt.% 2. <0.001 wt./wt.% 3. <0.001 wt./wt.% 4. 0.002 wt./wt.% 5. <0.001 wt./wt.% 6. <0.001 wt./wt.% 7. <0.001 wt./wt.%
Total % Impurity (NMR)		0.90 wt./wt.%
Total % Impurity (LC/MS)		None Quantified
Total % Impurity (GC/MS)		10.21 wt./wt.%
Related Compounds – POAA		0.03 wt./wt.%
Residual Solvents (TGA)		None Detected
Purity by DSC		87.6 wt./wt.%.
Inorganic Anions (IC) 1. Chloride 2. Fluoride 3. Bromide 4. Nitrate 5. Nitrite 6. Phosphate 7. Sulfate		1. <0.015 wt./wt.% 2. <0.005 wt./wt.% 3. <0.040 wt./wt.% 4. <0.009 wt./wt.% 5. <0.006 wt./wt.% 6. <0.007 wt./wt.% 7. <0.040 wt./wt.%
Organic Acids[2] (IC) 1. TFA 2. PFPA 3. HFBA 4. NFPA		1. <0.1 wt./wt.% 2. <0.1 wt./wt.% 3. <0.1 wt./wt.% 4. <0.25 wt./wt.%
Elemental Analysis[3]: 1. Carbon 2. Hydrogen 3. Nitrogen 4. Sulfur 5. Fluorine	1. Theoretical Value = 25.2% 2. Theoretical Value = 1.75% 3. Theoretical Value = 2.45% 4. Theoretical Value = 5.60% 5. Theoretical Value = 56.6%	1. 24.42 wt./wt.% 2. 1.78 wt./wt.% 3. 2.72 wt./wt.% 4. 9.34 wt./wt.% 5. 58.4 wt./wt.%

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-1
3M Product: EtFOSE-OH
Test Control Reference #: SD-013

Date of Last Analysis: 11/26/00

Expiration Date: 11/26/01

Storage Conditions: <-10 °C

Re-assessment Date: 11/26/01

[1]Purity = 100% - (total metal impurities, 0.002% + total NMR impurities, 0.90% + GC/MS impurities, 10.21 + POAA, 0.03%)

Total impurity from all tests = 11.14%
Purity = 100% - 11.14% = 88.9%

[2]TFA	Trifluoroacetic acid
HFBA	Heptafluorobutyric acid
NFPA	Nonafluoropentanoic acid
PFPA	Pentafluoropropanoic acid

[3]Theoretical value calculations based on the empirical formula, $C_{12}H_{10}F_{17}NO_3S$ (MW=571)

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-1
3M Product: EtFOSE-OH
Test Control Reference #: SD-013

GC/MS Purity Profile

Peak #	Retention Time (min)	Identity	% Impurity
1	6.163	Unknown	0.12
2	8.011	Unknown	0.23
3	8.206	Unknown	0.51
4	9.065	Unknown	0.21
5	9.844	Unknown	0.34
6	13.93	Unknown	0.62
7	14.238	Unknown	0.11
8	15.130	C2	0.11
9	15.52	C3	1.11
10	15.941	C4	1.55
11	16.379	C5	1.07
12	16.801	C6	3.30
13	17.222	C7	0.93
Total	-	-	**10.21**

This work was conducted under EPA Good Laboratory Practice Standards (40 CFR 160).

Prepared By: _____ 11/27/00
David S. Bell Date
Scientist
Centre Analytical Laboratories

Reviewed By: _____ 11/27/00
John Flaherty Date
Laboratory Manager
Centre Analytical Laboratories

COA023-022-1

Page 3 of 3

ANALYTICAL REPORT

STUDY TITLE

PURITY DETERMINATION OF SAMPLE LOTS OF PFOS
Sample # TCR-00065-022

DATA REQUIREMENTS

Test Article Characterization

STUDY DIRECTOR

Kevin Lloyd

"THIS IS AN EXACT COPY OF THE ORIGINAL DOCUMENT"
BY STE DATE 10/25/00

ANALYTICAL REPORT COMPLETION DATE

October 25, 2000

PERFORMING LABORATORIE / TESTING FACILITIES

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

STUDY SPONSOR

3M Environmental Technology and Safety Services
Building 2-3E-09
PO Box 33331
St. Paul, MN 55133-3331

PROJECT IDENTIFICATION

Centre Study Number: 023-045

Total Pages: 15

Centre Study No.: 023-045

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

Centre Study Number 023-045, entitled "Purity Determination of Sample Lots of PFOS, Sample # TCR-00065-022" conducted for 3M Environmental Laboratory, was performed in compliance with US EPA Good Laboratory Practice Standards (40 CFR Part 160) by Centre Analytical Laboratories, Inc. with the following exceptions:

> The automated data collection systems used in this study were not fully compliant with 21 CFR 58.130 (e).

Kevin Lloyd
Study Director
Centre Analytical Laboratories, Inc.

10/25/00
Date

James K. Lundberg, Ph.D.
Sponsor Representative
3M Environmental Technology and Safety Services

10/27/00
Date

STE 10/25/00

Centre Study No.: 023-045

QUALITY ASSURANCE STATEMENT

Centre Study Number 023-045, entitled "Purity Determination of Sample Lots of PFOS, Sample # TCR-00065-022" was reviewed by Centre Analytical Laboratories' Quality Assurance Unit. All reviewed phases were reviewed for conduct according to Centre Analytical Laboratories' Standard Operating Procedures, the Study Protocol, and all applicable Good Laboratory Practice Standards. All findings were reported to the Study Director and to management.

Phase	Date Inspected	Date Reported to Study Director and Centre Management	Date Reported to Sponsor Management
1. Protocol Review	10/19/00	10/25/00	10/25/00
2. Raw Data Review	10/19/00	10/25/00	10/25/00
3. Report Review	10/24/00	10/25/00	10/25/00

William Spare
Quality Assurance Officer

10/25/00
Date

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BY STE DATE 10/25/00

Centre Study No.: 023-045

CERTIFICATION OF AUTHENTICITY

This report, for Centre Study Number 023-045, is a true and complete representation of the raw data for the study.

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

Study Director, Centre:

10/25/00

Kevin Lloyd
Study Director
Centre Analytical Laboratories, Inc.

Date

Centre Analytical Laboratories, Inc. Facility Management:

25-OCT-00

Richard A. Grazzini, Ph.D.
President
Centre Analytical Laboratories, Inc.

Date

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BY STF DATE 10/25/00

Centre Study No.: 023-045

STUDY IDENTIFICATION

PURITY DETERMINATION OF SAMPLE LOTS OF PFOS
Sample # TCR-00065-022

TYPE OF STUDY:	Characterization
TEST SYSTEM:	Not Applicable
TEST ARTICLE:	PFOS, Test Control Reference # TCR-00065-022
SPONSOR:	3M Environmental Technology and Safety Services Building 2-3E-09 PO Box 33331 St. Paul, MN 55133-3331
STUDY DIRECTOR:	Kevin Lloyd Center Analytical Laboratories, Inc. Phone: (814) 231-8032
TESTING FACILITIES:	Centre Analytical Laboratories, Inc. (Centre) 3048 Research Drive State College, PA 16801 Phone: 814-231-8032
ANALYTICAL PHASE TIMETABLE:	Study Initiation Date: 09/28/00 Analytical Start Date: 10/05/00 Analytical Termination Date: 10/24/00

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Centre Study No.: 023-045

PROJECT PERSONNEL

The Study Director for this project was Kevin Lloyd at Centre Analytical Laboratories, Inc. The following personnel from Centre Analytical Laboratories, Inc., were associated with various phases of the study:

Name	Title
Gerry Shero	Scientist
David S. Bell	Scientist
Emily R. Stauffer	Scientist
Mark Ammerman	Sample Custodian

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Appendix A: Study Protocol 00P-023-045: PURITY DETERMINATION OF SAMPLE LOTS OF PFOS, Sample # TCR-00065-022, Including Amendment 1 15

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1.0 SUMMARY

The purity of PFOS from sample # TCR-00065-022, was determined from the LC/MS test data to be 88.0% pure as compared with the PFOS control (TCR-00017-046, 97.9% purity).

2.0 INTRODUCTION

This report details the results of the analysis of PFOS, Test Control Reference# TCR-00065-022. The test was analysis by liquid chromatography – mass spectrometry (LC/MS). The LC/MS was conducted at Centre.

The study was initiated on September 28, 2000 when the Study Director signed protocol number 00P-023-045. The analytical start date was October 5, 2000, and the experimental termination date was October 9, 2000.

3.0 TEST SYSTEM

There is no test system associated with this characterization study, therefore the GLP requirement for test system description, justification, and identification do not apply. The route of administration, levels and frequency of administration also do not apply to characterization studies.

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4.0 TEST ARTICLE

The test article was PFOS, Test Control Reference# TCR-00065-022. 3M Environmental Laboratory supplied the test article and it was logged at Centre Analytical Laboratories, Inc. as follows:

Compound	Lot or NB Number	Centre Control No.	Purity	Date Received
PFOS TCR-00065-022	1195	00-023-068	TBD	10/05/00

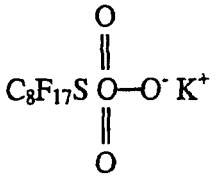
The sample received was a white, crystalline solid. It was received under frozen conditions and was stored in a temperature-monitored freezer kept at <-10°C.

The analytical/control standard PFOS was:

Control Article	Lot Number	Centre Control No.	Purity	Expiration Date
PFOS	TCR-00017-046	00-023-042	97.9%	8/31/2001

The chemical and physical data for PFOS is as follows.

Common Name: PFOS
Molecular weight: 499 ($C_8F_{17}SO_3^-$)
CAS Number: 2795-39-3
Structure:



Note: the neutral molecule and standard form that the PFOS (anion) is derived from is potassium perfluorooctane sulfonate ($C_8F_{17}SO_3K$), molecular weight 538.

5.0 EXPERIMENTAL PROCEDURES

LC/MS Spectral Analysis

PFOS sample TCR-00065-022 was analyzed for purity using liquid chromatography/mass spectrometry (LC/MS). The sample was analyzed according to the following procedure.

The sample was prepared at about 250 µg/mL in methanol and analyzed by LC/MS using a Hewlett-Packard 1100 HPLC system interfaced with a Hewlett-Packard 1100 mass selective detector. The mass spectrometer was run in negative ionization mode using electrospray ionization (ESI) using Selective Ionization Monitoring for ion m/z 499. The liquid chromatography system was operated in reversed-phase mode using a C18 silica-based column.

Each resulting chromatogram was calculated against the analytical control article (TCR-00017-046) calibration curve.

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6.0 RESULTS AND DISCUSSION

LC/MS Spectral Analysis

Responses were observed for the PFOS sample in negative ion mode using an ESI interface. Quantitation was performed using SIM mode, m/z 499, and PFOS was detected at 7.8 minutes. TH-PFOS was used as the internal standard, m/z 427, and was detected at 7.5 minutes. The total percent purity, calculated against the analytical control article (TCR-00017-046) calibration curve was determined to be 88.0% from the average of three replicate analyses.

8.0 CIRCUMSTANCES THAT MAY HAVE AFFECTED THE DATA

Electronic records are not fully compliant with 21 CFR 11, "Electronic records; Electronic Signature." However, approved SOPs were in place and all instrumentation used in this study was fully calibrated and operational. All original raw data were printed as hard copies and fully audited by quality assurance. Verified exact copies and the electronic data will be stored in the archives at Centre Analytical Laboratories. Original raw data will be returned to the Sponsor.

9.0 RETENTION OF DATA AND SAMPLES

When the final report is final, all original paper data generated by Centre Analytical Laboratories, Inc. will be shipped to the sponsor. This does not include facility-specific raw data such as instrument logs, however exact copies of temperature logs will be submitted. Exact copies of all raw data, as well as a signed copy of the final analytical report and all original facility-specific raw data, will be retained in the Centre Analytical Laboratories, Inc. archives for the period of time specified in 40 CFR Part 160. Retained samples of reference substances are archived by the sponsor.

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Centre Study No.: 023-045

Table I. Results From LC/MS Of TCR-00065-022, ESI Negative mode

3M ID	Retention Time (min)	Mass	Value µg/L	Determined µg/L	% Recovery
TCR-00065-022	7.79	499	250	219	88.0
TCR-00065-022	7.80	499	250	220	88.0
TCR-00065-022	7.80	499	250	220	88.0
				Average =	**88.0**
Standard	7.69	499	11	100*	*
Standard	7.73	499	11	17*	*
Standard	7.77	499	54	60	110
Standard	7.79	499	54	50	93
Standard	7.79	499	108	101	94
Standard	7.77	499	269	284	105
Standard	7.78	499	539	562	104
Standard	7.78	499	808	831	103
Standard	7.78	499	1078	1205	112
CCV	7.81	499	269	278	103
				Standards Average =	**103**
				Std Dev =	7

* First two injections during instrument warm up, not reported.
CCV = standard in run used to check calibration (C100500-4 at 269µg/L)

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Centre Study No.: 023-045

Figure 1. PFOS Calibration Standard (C100500-4) at 269µg/L

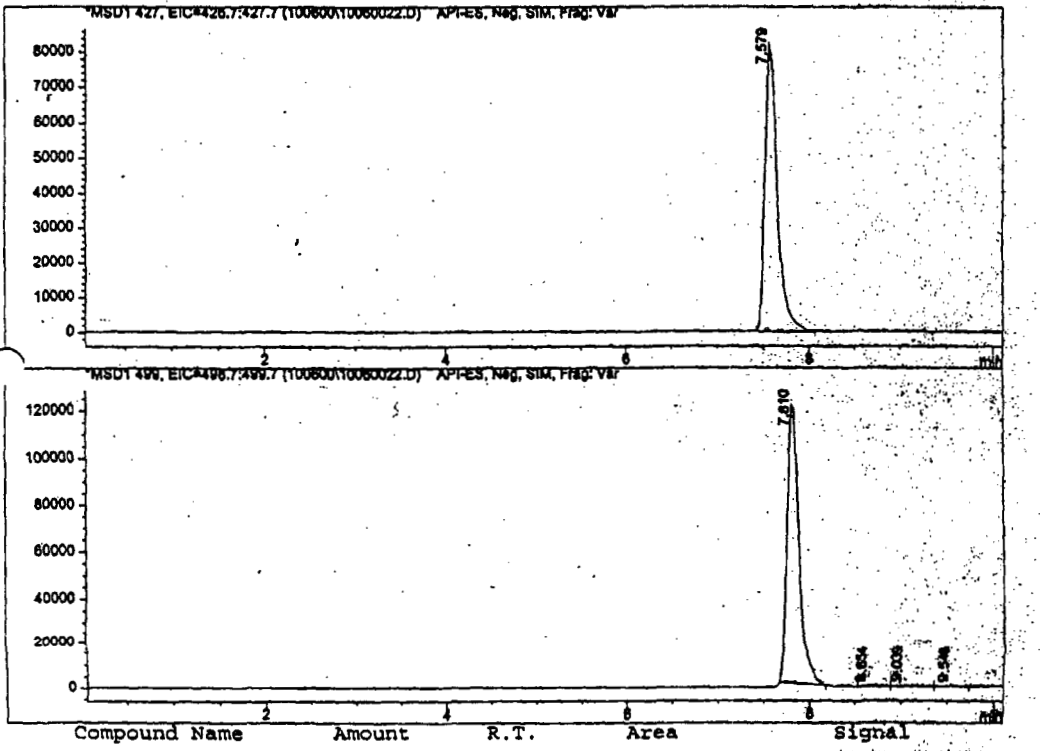
Sample Name:C100500-4
Sample Info: 1

Data file :C:\HPCHEM\1\DATA\100600\10060022.D

Injection Date	: 10/6/2000 3:11:22 PM	Seq Line :	20
Acq Operator	: GS GS 10-23-00	Vial No. :	14
Instrument	: Instrument 1	Inj. No. :	1
Dilution	: 1	Inj. Vol. :	5 µl

Acq. Method : PFOS.M
Analysis Method : C:\HPCHEM\1\METHODS\100600.M
Last Changed : 10/23/2000 11:44:26 am

Analysis for PFOS



Compound Name	Amount	R.T.	Area	Signal
TH-PFOS	500.0	7.58	785730	MSD1 427, EIC=426.7:42
PFOS	261.5	7.81	1078963	MSD1 499, EIC=498.7:49

*** End of Report ***

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Centre Study No.: 023-045

Figure 2. PFOS Calibration Standard (C100500-3) at 539µg/L

```
Sample Name:C100500-3
Sample Info:                                                          1

Data file  :C:\HPCHEM\1\DATA\100600\10060015.D
================================================================================
Injection Date  : 10/6/2000      1:13:21 PM      Seq Line    :          13
Acq Operator    : GS  GS 10.9.00                 Vial No.    :          15
Instrument      : Instrument 1                   Inj. No.    :           1
Dilution        : 1                              Inj. Vol.   :       5 µl

Acq. Method     : PFOS.M
Analysis Method : C:\HPCHEM\1\METHODS\100600.M
Last Changed    : 10/9/2000            01:45:43 pm
                  (modified after loading)
Analysis for PFOS
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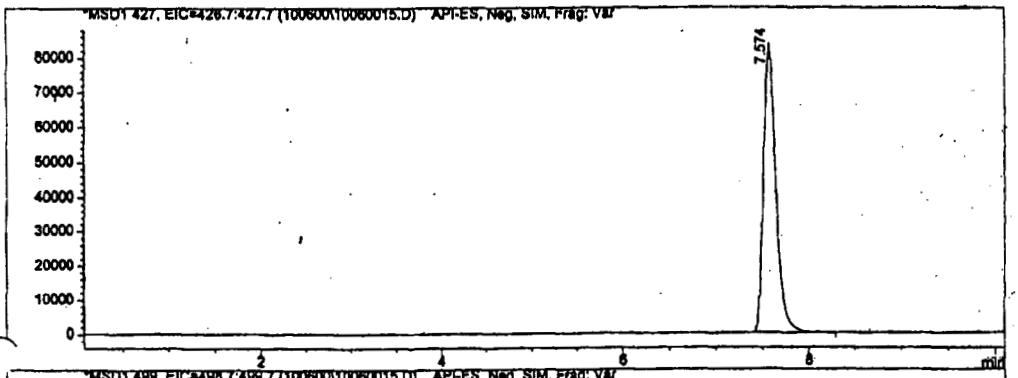

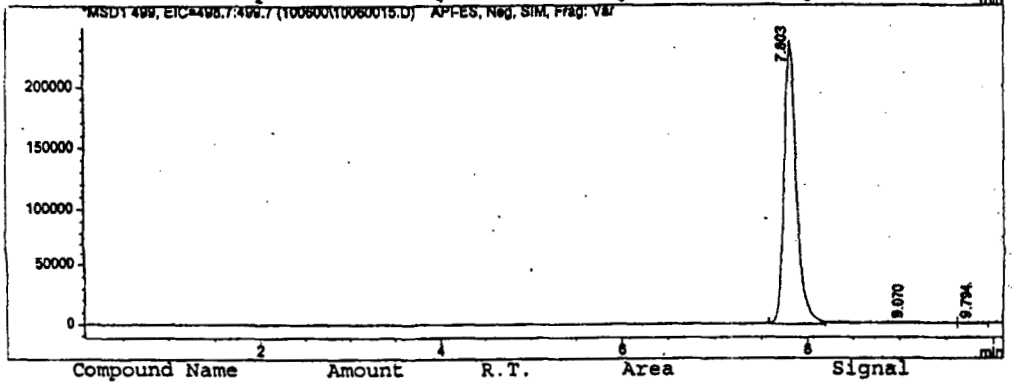

Compound Name	Amount	R.T.	Area	Signal
TH-PFOS	500.0	7.57	770327	MSD1 427, EIC=426.7:42
PFOS	499.8	7.80	2238071	MSD1 499, EIC=498.7:49

*** End of Report ***

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Centre Study No.: 023-045

Figure 3. PFOS (TCR-00065-022) at 250µg/L, ESI Negative Ion Mode

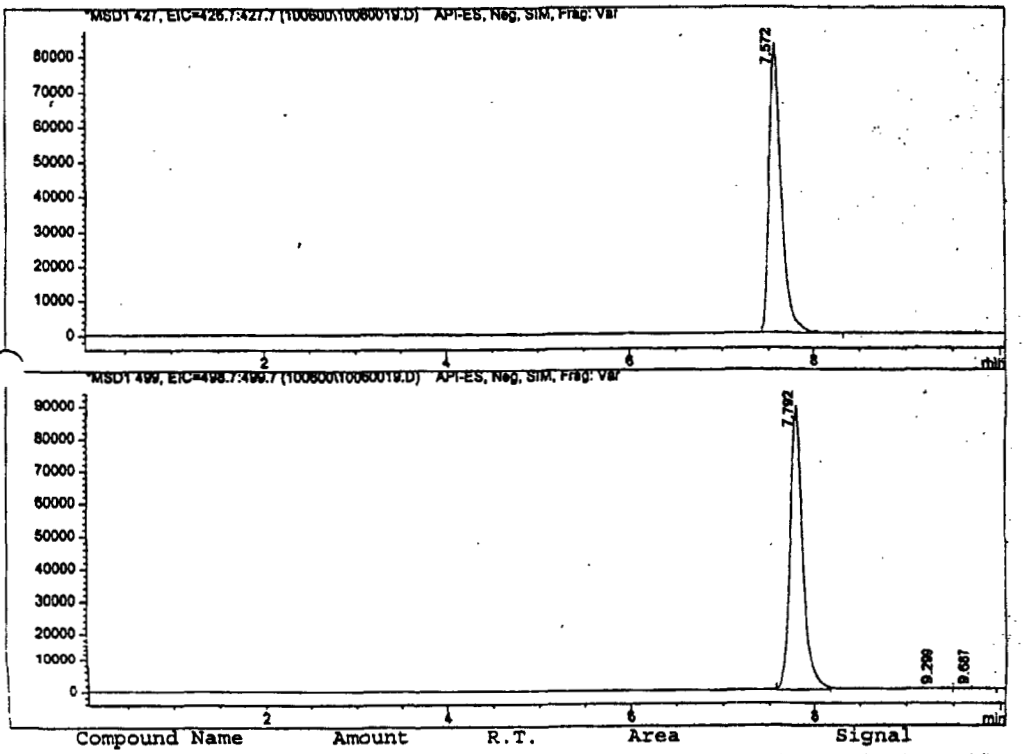
Sample Name:L29092-1
Sample Info:C100500-8 -> 1

Data file :C:\HPCHEM\1\DATA\100600\10060019.D

Injection Date	: 10/6/2000 2:20:49 PM	Seq Line	: 17
Acq Operator	: GS GS 10-23-00	Vial No.	: 21
Instrument	: Instrument 1	Inj. No.	: 1
Dilution	: 1	Inj. Vol.	: 5 µl

Acq. Method : PFOS.M
Analysis Method : C:\HPCHEM\1\METHODS\100600.M
Last Changed : 10/23/2000 11:44:26 am

Analysis for PFOS



Compound Name	Amount	R.T.	Area	Signal
TH-PFOS	500.0	7.57	776592	MSD1 427, EIC=426.7:42
PFOS	218.7	7.79	894026	MSD1 499, EIC=498.7:49

*** End of Report ***

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APPENDIX A

Study Protocol

Purity Determination of Sample Lots of PFOS

Including Amendment 1

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-001
LRN-U2103

Appendix H: Report Signature Page

John L. Butenhoff June 6, 2001

John L. Butenhoff, Ph.D., *Study Director* Date

Marvin T. Case 6 June 2001

Marvin T. Case, D.V.M., Ph.D., *Sponsor Representative* Date

05/31/01

Kristen J. Hansen, Ph.D., *Principal Analytical Investigator* Date

05/31/01

William K. Reagen, Ph.D., *Laboratory Manager* Date

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Study Title

104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Analytical Laboratory Report Title

Determination of the Presence and Concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in Serum and Liver Samples of Crl:CD®(SD) IGS BR Rats Exposed to N-Ethyl Perfluorooctanesulfonamido Ethanol

Data Requirement

Not Applicable

Author

3M Environmental Laboratory

Study Completion Date

June 06, 2001

Performing Laboratories

Liver and Serum Analyses

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

Project Identification

3M Medical Department Study: T-6316.1
Covance In-Life Study: 6329-228
Analytical Report: FACT TOX-003
3M Laboratory Request No. U2104

Total Number of Pages

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

This page has been reserved for specific country requirements.

3M Environmental Laboratory Page 2

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

GLP Compliance Statement

Analytical Laboratory Report Title: Determination of the Presence and Concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in Serum and Liver Samples of Crl:CD®(SD) IGS BR Rats Exposed to N-Ethyl Perfluorooctanesulfonamido Ethanol

Study Identification Numbers: T-6316.1, FACT TOX-003, LRN-U2104

This study was conducted in compliance with United States Food and Drug Administration (FDA) Good Laboratory Practice (GLP) Regulations 21 CFR Part 58, with the exceptions in the bulleted list below. The analytical phase completed at the 3M Environmental Laboratory was performed in accordance with 3M Environmental Laboratory Standard Operating Procedures.

Exceptions to GLP compliance:

- There were two study directors in this study. This study was designed as two separate studies. The in-life phase study was considered to end at the generation and shipment of specimens. The analytical study was considered to start at the receipt of these specimens for analysis. This resulted in having two separate study directors, one for each phase of the same study. However, since the technical performance of each phase was entirely separate, no effect is expected from this exception.
- Sample storage stability will not be determined.
- Characterization of the analytical standards is underway, but has not yet been completed (21 CFR 58.105 (a)). Lot No. 59905 of the surrogate, THPFOS, will not be characterized for purity since quantities of this standard are exhausted. Lot 936 of EtFOSE-OH, and Lot L2353 and TN-A-1886 of PFOSA will not be characterized since quantities of these standards are exhausted.
- The electronic data systems in use have not been validated and there is not an electronic audit trail of corrections currently available (21 CFR 58.130 (e)). Authenticated hard copies of chromatograms and associated documents will be considered as the original raw data.
- There were instances in the raw data where corrections were not made according to 21CFR58.130(e) GLP requirements. Data was crossed out with no reason given, or original entries were obscured or contained "write-overs" with no explanation, initial or date provided.
- Some reagents (00-022-2, 0.5M TBA solution and 00-003-28, 0.5M TBA solution) were not labeled with storage requirements.
- Persons responsible for equipment were not identified (021188-55 IEC Cetra GP*R and N-Evap.)

(See next page for signatures)

3M Environmental Laboratory Page 3

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

GLP Compliance Statement continued

John L. Butenhoff June 6, 2001
John L. Butenhoff, Ph. D., *Study Director* Date

Marvin T Case 6 June 2001
Marvin T. Case, D.V.M., Ph.D., *Sponsor Representative* Date

05/31/01
Kristen J. Hansen, Ph.D., *Principal Analytical Investigator* Date

05/31/01
William K. Reagen, Ph.D., Laboratory Manager Date

3M Environmental Laboratory Page 4

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

GLP Study—Quality Assurance Statement

Analytical Laboratory Report Title: Determination of the Presence and Concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in Serum and Liver Samples of Crl:CD®(SD) IGS BR Rats Exposed to N-Ethyl Perfluorooctanesulfonamido Ethanol

Study Identification Numbers: T-6316.1, FACT TOX-003, LRN-U2104

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

Inspection Dates	Phase	Date Reported to	
		Management	Study Director
4/20/00	Sample prep	8/8/00	8/8/00
6/30/00, 7/5/00	Sample prep	7/21/00	7/21/00
4/23/01—4/30/01 4/30/01—5/1/01	Data	5/2/01	5/2/01
5/22/01	Report	5/23/01	5/23/01

Casey Howell
QAU Representative

Jun 11, 2001
Date

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

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LRN-U2104

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Table 10. FACT TOX-003 Data Summary for PFOS, PFOSA, PFOSAA, EtFOSE, M556 and PFOSEA—Rat Liver (µg/g) **159**

Table 10. (Continued) FACT TOX-003 Data Summary for PFOS, PFOSA, PFOSAA, EtFOSE, M556 and PFOSEA—Rat Liver (µg/g) **160**

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Study Personnel and Contributors

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3M Environmental Laboratory
Kristen J. Hansen, Ph.D. *Principal Analytical Investigator*

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Megan C. Holloway*
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Sally A. Linda*
Ian A. Smith*
Kathleen M. Stock*
Anh-Dao Vo
Bob W. Wynne*
Richard Youngblom*

*Contract lab professional service employees

Location of Archives

All original raw data, protocol, and analytical report have been archived at the 3M Environmental Laboratory. The test substance and analytical reference standard reserve samples, as well as the specimens pertaining to the analytical phase of this study are archived at the 3M Environmental Laboratory.

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Introduction and Purpose

The purpose of the study is to determine the presence and concentration of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA in serum and liver specimens collected from Covance Study No.: 6329-228 titled: 104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats. The Covance in-life study was initiated on April 17, 1998. The analytical phase of the study was initiated on May 22, 1998.

Test System

A total of 140 males and 140 female rats were used as the test system. Table 1 outlines the rat population demographics and dosage levels for study 6329-228.

The test system species and strain selected was the Crl:CD®(SD) IGS BR rat received from Charles River Laboratories, Inc., identified using an implanted microchip device. At the initiation of treatment the rats were approximately 6–8 weeks old and weighed between approximately 100–300 g.

Table 1. Test System Population Demographics for Study 6329-228

Study Group	Number of Animals		Approximate Dietary Levels of EtFOSE-OH (ppm)
	Male	Female	
Group 8 Control	70	70	0
Group 9 Treated	70	70	1

Specimen Collection and Analysis

Specimens were collected by Covance (study 6329-228) and sent to the 3M Environmental Laboratory for analysis. On week 4, 20 serum specimens and 20 liver specimens were collected from Groups 8 and 9 males and females. On week 14, 20 serum specimens and 20 liver specimens were collected from Groups 8 and 9 males and females. On week 53, 20 serum specimens and 20 liver specimens were collected from Groups 8 and 9 males and females. On week 105, 40 liver specimens were collected from Groups 8 and 9 males and females. On week 105, 40 serum specimens were collected from Groups 8 and 9 males and females. The number and type of specimens collected for analyses in the analytical phase of this study are presented.

Specimens Collected from Study Groups 8 and 9
Serum Specimens—100 specimens
Liver Specimens—100 specimens

Liver and sera specimens were shipped to the 3M Environmental Laboratory frozen and on dry ice.

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Sera and liver samples were extracted beginning on May 27, 1998 using an ion pairing reagent and either ethyl acetate or methyl-*tert*-butyl ether (MtBE). Liver samples were homogenized prior to the extraction procedure. Sample extracts were analyzed using high-pressure liquid chromatography-electrospray/tandem mass spectrometry (HPLC-ESMSMS) in the multiple reaction monitoring mode. PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA levels were evaluated by external calibration using extracted curves. Analytical details are included in this report.

Specimen Receipt and Maintenance

The 3M Environmental Laboratory received liver and serum specimens collected at predetermined time points during and at the end of the *in-life* phase of Covance Study 6329-228 from Covance May 1998 through June 2000. All specimens were received frozen on dry ice and were immediately transferred to storage at -20°C ±10°C.

Control matrices used in liver and sera analyses performed during TOX-003 were obtained from commercial sources and are presented in Appendix A. Samples analyzed at the 3M Environmental Laboratory will be maintained for a period of 10 years and will be stored at the laboratory at -20°C ±10°C.

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Chemical Characterization of the Reference Standards

Chemical characterization information on the analytical reference materials used in this study is presented in tabular form below.

Table 2. Characterization of the Analytical Reference Standards in Study FACT TOX-003

Reference Standard / Formula	Acronym	Source	Expiration Date	Storage Conditions	Chemical Lot Number	Physical Description	Purity
Potassium Perfluorooctanesulfonate $C_8F_{17}SO_3$-K+	KPFOS[a]	3M	08/31/01	Ambient temperature	171	Light colored powder	86.4%
		3M	2010	Ambient temperature	193	White crystals	88.0%
		3M	2010	Ambient temperature	215	White powder	TBD[c]
N-Ethyl Perfluorooctanesulfonamido ethyl alcohol $C_8F_{17}SO_2N(C_2H_5)CH_2CH_2OH$	EtFOSE-OH	3M	11/26/01	Ambient temperature	936	Amber waxy solid	NA*
		3M	2010	Ambient temperature	Unknown (SD013)	Amber waxy solid	88.9%
Sodium Perfluorooctanesulfonylamido(ethyl) acetate $C_8F_{17}SO_2N(CH_2CH_3)CH_2COO$-Na	PFOSAA[b]	3M	01/01/2010	Ambient temperature	617	Yellow to amber liquid	TBD[d]
Perfluorooctanesulfonylamido(ethyl)acid $C_8F_{17}SO_2N(CH_2CH_3)CH_2COO$- H		3M	01/01/2010	Ambient temperature	Unknown (98-0207-0303-3)	Light yellow powder	TBD[c]
Perfluorooctanesulfonylamide $C_8F_{17}SO_2NH_2$	PFOSA	3M	01/01/2010	Ambient temperature	L2353	Amber to brown waxy solid	NA*
		3M	01/01/2010	Ambient temperature	L15709	Light yellow waxy solid	TBD[c]
Perfluorooctanesulfonylethylamide $C_8F_{17}SO_2NHCH_2CH_3$	PFOSEA	3M	01/01/2010	Ambient temperature	529	Amber waxy solid	TBD[c]
		3M	01/01/2010	Ambient temperature	Unknown (TN-A-1885)	Amber waxy solid	NA*
M556 $C_8F_{17}SO_2N(H)CH_2COOH$	M556	3M	01/01/2010	Ambient temperature	NB113047-80	White powder	TBD[c]
1H, 1H, 2H, 2H-Tetrahydroperfluorooctanesulfonic acid $C_8H_4F_{13}SO_3H$	THPFOS	ICN	01/01/2010	Ambient temperature	59909	Brown powder	NA*

*This lot is exhausted and cannot be characterized.
NR—Not recorded
NA—Not applicable
TBD—To be determined

[a]Target analyte is PFOS, $C_8F_{17}SO_3^-$
[b]Target analyte is $C_8F_{17}SO_2\{(CH_2CH_3)(CH_2COO^-)\}$
[c]Unless otherwise indicated, at the time of quantitation, the purity for all analytes was assumed to be 100%.
[d]For reference lot 617, a purity of 53.82% was used for PFOSAA result quantitation.

Dose Confirmation Analyses

Dose preparation methods and analysis were performed by Covance, using a validated analytical method provided by Covance (MP-M312-MA), and are reported separately (Reference Covance 6329-228).

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Method Summaries

Following is a brief description of the methods used during this analytical study by the 3M Environmental Laboratory. Detailed descriptions of the methods used in this study are located in Appendix C.

Data collected prior to November 1999 was reworked in 2000 to accommodate improvements in data reduction methods. Both the original and "reworked" data are archived; reworked data is presented in the final results. The improved methods are documented in the form of method modifications.

As the present study progressed, more advanced methods evolved and earlier methods were used with deviations until amendments to the protocol were written. Protocol and method deviations are located in Appendix B of this report.

3M Environmental Laboratory

PREPARATORY METHODS

- **FACT-M-1.0**. "Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry". This method was used for week 4 samples extracted on 6/9/98 and week 14 samples extracted on 7/17/98.
- **FACT-M-3.0**, "Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry". This method was used for week 4 samples extracted on 5/27/98 and week 14 samples extracted on 7/15/98.
- **ETS-8-4.1**, "Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry". This method was used for week 53 samples extracted on 4/20/00, and week 105 samples extracted on 6/29/00.
- **ETS-8-6.0**, "Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry". This method was used for week 53 and week 105 samples extracted on 4/14/00.

  An ion-pairing reagent was added to the sample and the analyte ion pair was partitioned into ethyl acetate (FACT-M-1.0 and FACT-M-3.0) or MtBE (ETS-8-4.1 and ETS-8-6.0). The extract was transferred to a centrifuge tube and put onto a nitrogen evaporator until dry. Each extract was reconstituted in 1.0 mL of methanol, and then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

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ANALYTICAL METHODS

- **FACT-M-2.0**, "Analysis of Liver Extracts for Fluorochemicals Using HPLC-Electrospray/Mass Spectrometry".
- **FACT-M-4.0**, "Analysis of Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry".
- **ETS-8-5.1**, "Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry".
- **ETS-8-7.0**, "Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry".

The analyses were performed by monitoring one or more product ions selected from a single primary ion characteristic of a particular fluorochemical using HPLC-ES/MS/MS. For example, molecular ion 499, selected as the primary ion for PFOS ($C_8F_{17}SO_3^-$) analysis, was fragmented further to produce ion 99 (FSO_3^-). The characteristic product ion 99 was monitored for quantitative analysis.

ANALYTICAL EQUIPMENT

The actual analytical equipment settings used in the present analytical phase of this study varied slightly during actual data collection. The following is representative of the settings used during the analytical phase of this study.

Liquid Chromatograph: Hewlett-Packard® Series 1100 Liquid Chromatograph system
Analytical column: Keystone® Betasil™ C_{18} 2x50 mm (5 µm)
Column temperature: Ambient
Mobile phase components:
Component A: 2mM ammonium acetate
Component B: methanol
Flow rate: 300 µL/min
Injection volume: 10 µL
Solvent Gradient: 9.0 minutes

Time (minutes)	*%B*
0.0	40%
1.0	40%
4.5	95%
6.5	95%
7.0	40%
9.0	40%

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Mass Spectrometer: Micromass® API/Mass Spectrometer Quattro II™ Triple Quadrupole system
Software: Mass Lynx™ 3.1, 3.2, 3.3, 3.4
Cone Voltage: 30–60 V
Collision Gas Energy: 25–45 eV
Mode: Electrospray Negative
Source Block Temperature: 150°C ±10°C
Electrode: Z-spray
Analysis Type: Multiple Reaction Monitoring (MRM)

Table 3. Target Ions Monitored in 3M Laboratory Analyses

Target Analyte	Primary Ion (AMU)	Product Ion (AMU)
PFOS	499	80, 99, 130
PFOSA	498	78
PFOSAA	584	83, 169
EtFOSE-OH	630	59
PFOSEA	526	65
M556	556	65, 78, 83, 169
THPFOS	427	80

Data Quality Objectives and Data Integrity

The following data quality objectives were indicated in the method performance section of ETS-8-5.1, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry and ETS-8-7.0, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry:

- **Linearity:** The coefficient of determination (r^2) equal to or greater than 0.980.
- **Limits of Quantitation (LOQ):** The LOQ is equal to the lowest acceptable standard in the calibration curve.
- **Acceptable Precision:** Precision is better than 30% for the method.
- **Acceptable Spike Recoveries:** 70–130%.

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Data Summary, Analyses, and Results

Data quality objectives for the analytical phase of this study outlined in the 3M Environmental Laboratory protocol for FACT TOX-003 (see Appendix B) were met with the exceptions noted in this report.

Summary of Quality Control Analyses Results

- **Linearity:** The coefficient of determination (r^2) of the standard curve was ≥0.980.
- **Calibration Standards:** Quantitation of the target analytes was based on linear regression analysis weighted 1/x of a single, opening or two bracketing extracted matrix curves for each group of samples. Rat and rabbit sera were used for matrix curves in sera analyses, while rabbit liver was used for matrix curves in liver analyses. High or low points on the curve may have been deactivated to provide a better linear fit over the curve range most appropriate to the data. Low curve points with peak areas less than two times that of the extraction blanks were deactivated to disqualify a data range that may have been significantly affected by background levels of the analyte. Occasionally, a single mid-range curve point that was an obvious outlier may have been deactivated. Quantitation of each analyte was based on the response of one or more specific product ion(s) using the multiple reaction monitoring mode of the instrument (see Appendix C, Analytical Methods). Calibration standards were prepared to run, undiluted, approximately within the linear range of the instrument (approximately 5–1000 ng/g).
- **Limits of Quantitation (LOQ):** The LOQ is equal to the lowest acceptable standard in the calibration curve (defined as a standard within ±30% of the theoretical value), and is at least two times the analyte peak area detected in the surrogate matrix blanks. (See Appendix D).
- **Blanks:** All blanks were below the lower limit of quantitation for the compounds of interest. To simplify analyses that were complicated by endogenous levels of fluorochemicals in unexposed rat sera and liver, rabbit sera and liver were selected as suitable surrogate matrices for method blanks.
- **Precision:** Instrumental precision was determined by replicate injections of a single serum extract from a related study (TOX-001). Instrumental precision was determined for PFOS, PFOSAA, and M556; variation was less than 8.5% for all targeted analytes.
- **Matrix Spikes:** Sera—Matrix spike samples were prepared from control rat sera along with each batch of sera samples. Samples were spiked between 75–250 ng/mL, levels that approximate the levels detected in Groups 8 and 9 samples, depending upon the analyte. All spikes were prepared to run, undiluted, within a ten-fold limit of the linear range of the calibration curve. For some analytes, samples for some high dose animals may be much higher than the range of these prepared spikes. Sera matrix spike recoveries are presented in tabular form below.

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Table 4. Matrix Spikes—Sera

	# of Spikes Prepared	Average Spike Recovery	Range	Standard Deviation
PFOS	8	111%	73–144%	27%
PFOSA	8	107%	79–126%	16%
PFOSAA	8	106%	74–140%	26%
M556*	4	139%	108–174%	35%
EtFOSE-OH	4	39%	16–61%	25%
PFOSEA	4	66%	36–90%	28%

*M556 was identified as a metabolite of EtFOSE-OH after initiation of the analytical work. Standard material was unavailable and had to be synthesized. No M556 data is provided for the Week 4 through Week 14.

Liver—Matrix spike samples were prepared from a control group rat liver specimen along with each batch of liver samples. Samples were spiked between 100–500 ng/g, levels that approximate the levels detected in Groups 8 and 9 samples, depending on the analyte. All spikes were prepared to run, undiluted, within a ten-fold limit of the linear range of the calibration curve. For some analytes, samples for some high dose animals may be much higher than the range of these prepared spikes. Liver matrix spike recoveries are presented in tabular form below.

Table 5. Matrix Spikes—Liver

	# of Spikes Prepared	Average Spike Recovery	Range	Standard Deviation
PFOS	12	94%	65–137%	23%
PFOSA	12	87%	58–132%	23%
PFOSAA	12	96%	53–200%	39%
M556*	6	103%	80–122%	18%
EtFOSE-OH	4	118%	77–154%	39%
PFOSEA	6	108%	69–144%	27%

*M556 was identified as a metabolite of EtFOSE-OH after initiation of the analytical work. Standard material was unavailable and had to be synthesized. No M556 data or QC is provided for the Week 4 through Week 14 samples.

- **Surrogates:** The surrogate (THPFOS) was added to all samples and standards, with the exception of Week 4 liver and sera samples. THPFOS was not used for quantitation, but was used to monitor for gross (±50%) instrument failure.

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Statement of Data Quality

It is not possible to verify true recovery of endogenous analyte from tissues without radio-labeled reference material. The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered to be accurate to within one standard deviation of the average fortified samples recovery. Refer to Tables 4 and 5 for matrix spikes from each analyte and matrix.

The results of quality control analyses (curve fit, CCVs, and MS/MSDs) for EtFOSE-OH and PFOSEA were inconsistent and indicate that data presented for this analyte should be considered to be qualitative only. Values for these analytes are presented in data tables in the spirit of full disclosure, but should not be used in any quantitative assessment of the data.

Summary of Sample Results

PFOS results (those obtained using lot # 171 and #193) and EtFOSE-OH results (those obtained with lot # SD013) have been corrected for purity of the analytical reference material. Uncorrected results are noted in the data tables.

- **Samples from Control Animals:** The target analytes were often detected in the sera and liver samples from the control animals. These levels were usually lower than those found in the low dose test animals.
- **Samples from Dosed Animals:** In general, target analytes were present in the sera and liver samples in the dose group. Detailed sample data tables are presented in Appendices D and E.

Statistical Methods and Calculations

Statistical methods were limited to the calculation of means and standard deviations. See Appendix F for example calculations used to generate the liver and serum sample data in FACT TOX-003.

Statement of Conclusion

Under the conditions of the present studies, the concentrations of PFOS, PFOSA, PFOSAA, EtFOSE-OH, M556, and PFOSEA were determined in the sera and liver of rats dosed with N-Ethyl Perfluoroctanesulfonamido Ethanol during the in-life phase of the study.

References

Covance Study No.: 6329-228 titled: 104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

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Appendix A: Chemical Characterization and Control Matrices

Table 6. Characterization of the Control Matrices Used for Sera Analyses in Study FACT TOX-003

Control Matrix	Rat Serum	Rat Serum	Rabbit Serum	Rabbit Serum
Source	Sigma	Sigma	Sigma	Sigma
Expiration Date	2010	01/01/2010	01/01/2010	2010
Storage Conditions	-20°C ±10°C	-20°C ±10°C	-20°C ±10°C	-20°C ±10°C
Chemical Lot #	17H9306	19H89291	47H4641	118H8418
Physical Description	Rat Serum	Rat Serum	Rabbit Serum	Rabbit Serum

Table 7. Characterization of the Control Matrices Used for Liver Analyses in Study FACT TOX-003

Control Matrix	Rabbit Liver	Rabbit Liver	Rabbit Liver
Source	Covance Laboratories, Inc.	Covance Laboratories, Inc.	Covance Laboratories, Inc.
Expiration Date	2010	2010	2010
Storage Conditions	-20°C ±10°C	-20°C ±10°C	-20°C ±10°C
Chemical Lot #	F00014	F00007	F00016
Physical Description	Rabbit Liver	Rabbit Liver	Rabbit Liver

Table 8. Characterization of Test Article in Study FACT TOX-003

	Test Article
Chemical Name	**EtFOSE-OH** N-Ethyl Perfluorooctanesulfonamido ethanol T-6316
Source	3M
Expiration Date	11/26/01
Storage Conditions	Ambient temperature
Chemical Lot #	(30035, 30037, 30039)
Physical Description	Light yellow, waxy solid/flakes
Purity	97.4%

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Appendix B: Protocol, Amendments and Deviations

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COVANCE
THE DEVELOPMENT SERVICES COMPANY

Sponsor:

3M
St. Paul, Minnesota

PROTOCOL

Study Title:

T-6316.1

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Date:

April 1, 1998

Performing Laboratory:

Covance Laboratories Inc.
3301 Kinsman Boulevard
Madison, Wisconsin 53704-2595

Laboratory Study Identification:

Proposal No. 23124A

Covance 6329-228
3M T-6316.1

Study
104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Purpose
To assess the carcinogenicity of the test material when administered in the diet to rats for at least 104 weeks

Sponsor
3M
Toxicology Services
Building 220-2E-02, 3M Center
St. Paul, Minnesota 55144-1000

Study Monitor
Andrew M. Seacat, PhD
3M
Telephone No.: 612.575.3161
Facsimile No.: 612.733.1773

Study Location
Covance Laboratories Inc.
3301 Kinsman Boulevard
Madison, Wisconsin 53704-2595

Mailing Address: PO Box 7545
Madison, Wisconsin 53707-7545

Study Director
Peter J. Thomford, PhD
Covance Laboratories Inc.
Telephone No.: 608.241.7207
Facsimile No.: 608.242.2736

Toxicologist
Thomas E. Ryan, BS
Covance Laboratories Inc.

Proposed Study Timetable
In-Life Start Date: April 6, 1998
In-Life End Date: April 5, 2000

Regulatory Compliance
This study will be conducted in compliance with the Food and Drug Administration Good Laboratory Practice Regulations as set forth in Title 21 of the US Code of Federal Regulations, Part 58, issued December 22, 1978 (effective June 20, 1979), and with any applicable amendments.

Animal Care and Use Statement
All procedures in this protocol are in compliance with the Animal Welfare Act Regulations, 9 CFR 1-4. In the opinion of the Sponsor and study director, the study does not unnecessarily duplicate any previous work.

Quality Assurance
The protocol, study conduct, and final report will be audited by the Covance Quality Assurance Unit (QAU). The proliferation cell nuclear antigen evaluation, data, and report will be audited by the QAU of Pathology Associates International.

Test Material

Identification
T-6316 (Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol, NEtFOSE)

Lot Numbers
The lot numbers will be maintained in the raw data.

Purity
98.1% NEtFOSE (w/w)

Stability
Responsibility of the Sponsor

Storage Conditions
At room temperature

Characteristics
Information on synthesis methods, composition, or other characteristics that define the test material is on file with the Sponsor.

Reserve (Archive) Samples
A reserve sample (approximately 5 g) will be taken and stored at room temperature. This sample will be transferred to the Sponsor after completion of the in-life phase to be retained in accordance with 21 CFR 58.195.

Disposition of Test Material
After authorization from the Sponsor, any remaining test material will be returned to:

Andrew M. Seacat, PhD
3M
Toxicology Services
Building 220-2E-02, 3M Center
St. Paul, Minnesota 55144-1000
Telephone No.: 612.575.3161
Facsimile No.: 612.733.1733

Animals

Species
Rat

Strain
Crl:CD®(SD)IGS BR

Source
Charles River Laboratories, Inc., Raleigh, North Carolina

Age at Initiation of Treatment
Preferably 6 weeks of age, but not more than 8 weeks of age

Weight at Initiation of Treatment
100 to 300 g

Number and Sex
140 males and 140 females

Identification
Implantable microchip identification device

Husbandry

Housing
Individual (may be group-housed during acclimation)

Diet
Certified Rodent Diet #5002, meal, (PMI Nutrition International) *ad libitum,* unless otherwise specified. The diet is routinely analyzed by the manufacturer for nutritional components and environmental contaminants. Results of specified nutrient and contaminant analyses are on file at Covance-Madison.

Water
Ad libitum. Samples of the water are routinely analyzed for specified microorganisms and environmental contaminants. The results are on file at Covance-Madison.

Contaminants
There are no known contaminants in the diet or water at levels that might interfere with this study.

Environment
Environmental controls for the animal room will be set to maintain 18 to 26°C, a relative humidity of 30 to 70%, a 12-hour light/12-hour dark cycle, and a minimum of 10 room air changes/hour. The light/dark cycle may be interrupted to accommodate study-related activities.

Acclimation

At least 1 week

Randomization

Selection of animals for the study will be based on body weights, clinical observations, and other data as appropriate. Animals will be assigned to treatment groups using a computerized blocking procedure designed to achieve body weight balance with respect to treatment groups. At the time of randomization, the weight variation of the animals of each sex used will not exceed ±2 standard deviations of the mean weight, and the mean body weight for each group of each sex will not be statistically different at the 5.0% probability level.

Justification

Rats historically have been used in safety evaluation studies and are recommended by appropriate regulatory agencies.

Group Designations and Dietary Levels

Group[a]	Number of Animals Male	Number of Animals Female	Dietary Levels (ppm NEtFOSE)[b]
8 (Control)[c,d,e]	70	70	0
9 (Treated)[d,e]	70	70	1

a Groups will be designated as Groups 8 and 9. This study will be located in the same animal room as Covance 6329-212.
b T-6316 is 98.1% n-ethyl perfluorooctanesulfonamido ethanol (NEtFOSE); dose levels are expressed as ppm of NEtFOSE.
c The control animals will receive the basal diet only.
d Five animals/sex/group will be sacrificed during Weeks 4 and 14 for hepatocellular proliferation rate measurements and biochemical analyses (palmitoyl-CoA oxidation).
e Ten animals/sex/group will be designated as interim sacrifice animals and will be sacrificed after at least 78 weeks of treatment.

Dosing Procedures

Method of Administration

Dietary. Animals will receive test diet for at least 104 weeks.

Reason for Dosing Route

The potential human exposure is by the oral route.

Dose Preparation

Dose preparations will be mixed at least once every 4 weeks according to the study-specific mixing procedure developed by Covance. Dose concentrations will be based on the NEtFOSE content as supplied; the Sponsor has stated the T-6316 is 98.1% NEtFOSE (w/w) as supplied. All dose preparations will be stored at room temperature.

Retention Samples

Samples (approximately 100 g) will be taken from each dose preparation and stored at room temperature. Unless used for analyses, these samples will be discarded at least 1 month after completion of the in-life phase.

Dose Analyses

By Covance using a method supplied by the Sponsor and validated by Covance

Homogeneity

Homogeneity will be determined for the 1-ppm diet preparation mixed for Week 1. One sample (approximately 100 g) each from the top, middle, and bottom of the diet preparation will be collected, divided into three subsamples for extraction and analysis, and analyzed for test material content. All samples will be stored at room temperature until analyzed within 7 days of mixing. Homogeneity analysis will be repeated if the batch size changes by more than 30%.

Stability

The middle homogeneity sample will be analyzed on the day of mixing and serve as stability time 0. Two additional samples (approximately 100 g each) will be taken from the 1-ppm diet preparation mixed for Week 1 to establish stability. One sample will be stored at room temperature for at least 19 days, then analyzed. The other sample will be stored at room temperature after at least 32 days, then analyzed.

Dose Confirmation

Samples (approximately 100 g each) will be collected from all dose preparations and analyzed in duplicate. Homogeneity samples collected from the middle of the dose preparation for Week 1 will be used for dose confirmation results. All samples will be stored at room temperature until analyzed.

Observation of Animals

Clinical Observations

Each animal will be observed twice daily (a.m. and p.m.) for mortality and moribundity, findings will be recorded as they are observed.

Once prior to treatment and weekly thereafter, each animal will be removed from its cage and examined; abnormal findings or an indication of normal will be recorded. The following information on each grossly visible or palpable mass will be recorded.

time of onset
location
size (small or large)
appearance
progression

Body Weights

Prior to treatment (at randomization), weekly for Weeks 1 through 17, once every 4 weeks thereafter, and at Week 105

Food Consumption

Weekly for Weeks 1 through 16 and once every 4 weeks thereafter

Clinical Pathology

Frequency and Number of Animals

Unscheduled Collection

When possible, a blood film will be made and held for possible future examination from animals sacrificed at unscheduled intervals.

Scheduled Collections

Hematology, clinical chemistry, urinalysis, and urine chemistry will be done on 10 animals/sex/group during Weeks 14, 27, and 53.

A blood film will be made and held for possible future examination from animals at scheduled sacrifices after at least 78 and 104 weeks of treatment.

Method of Collection

Hematology, Clinical Chemistry, Urinalyses, and Urine Chemistry

Animals will be fasted overnight; blood will be collected from a jugular vein. The anticoagulant will be potassium EDTA for hematology tests. Samples for clinical chemistry will be collected without anticoagulant. Urine will be collected chilled overnight (approximately 16 hours).

Blood Films

Blood films will be taken as part of the necropsy procedure.

Tests

Hematology

red blood cell (erythrocyte) count
hemoglobin
hematocrit
mean corpuscular volume
mean corpuscular hemoglobin
mean corpuscular hemoglobin concentration
platelet count
white blood cell (leukocyte) count
differential blood cell count
blood cell morphology
reticulocyte smear (made, but not examined)

Clinical Chemistry

glucose
urea nitrogen
creatinine
total protein
albumin
globulin
cholesterol
total bilirubin
alanine aminotransferase
gamma glutamyltransferase
aspartate aminotransferase
calcium
inorganic phosphorus
sodium
potassium
chloride

Urinalysis

appearance	glucose
volume	ketones
specific gravity	bilirubin
pH	blood
protein	microscopic examination of sediment
urobilinogen	

Urine Chemistry

sodium	16 hour excretion of:
potassium	sodium
	potassium

Serum Analyses

Frequency and Number of Animals

Five animals/sex/group during Weeks 4 and 14 (animals selected for hepatocellular proliferation and biochemical analyses), five animals/sex/group (from animals selected for interim sacrifice) after at least 78 weeks of treatment, and five animals/sex/group at the terminal sacrifice

Method of Collection

Animals will be fasted overnight; blood (approximately 2 mL) will be collected from a jugular vein. Samples will be collected without anticoagulant.

Sample Handling

Blood samples will be allowed to clot at room temperature and centrifuged. Serum samples will be harvested and stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to:

Kris J. Hansen, PhD
3M Environmental Technology and Safety Services
935 Bush Avenue
Building 2-3E-09
St. Paul, Minnesota 55133-3331
Telephone No.: 612.778.6018
Facsimile No.: 612.778.6176

Samples will be retained by the Sponsor for possible future analysis.

Termination

Unscheduled Sacrifices and Deaths

Necropsies will be done. Animals to be sacrificed will be anesthetized with sodium pentobarbital, weighed, and exsanguinated. A blood film will be taken as part of the necropsy procedure for sacrificed animals.

Scheduled Sacrifices

Interim Sacrifices

During Week 4, five animals/sex/group will be fasted overnight, bled for serum samples, anesthetized with sodium pentobarbital, weighed, and exsanguinated. The abdominal cavity of each animal will be opened, the liver will be removed and weighed, and liver samples will be collected. Animals will be discarded after liver collection.

During Week 14, five animals/sex/group will be fasted overnight, bled for serum samples, anesthetized with sodium pentobarbital, weighed, and exsanguinated. The abdominal cavity of each animal will be opened, the liver will be removed, weighed, and liver samples collected. Animals will be discarded after liver collection.

After at least 78 weeks of treatment, 10 animals/sex/group will be fasted overnight, bled for serum samples (five animals/sex/group), anesthetized with sodium pentobarbital, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Terminal Sacrifice

After at least 104 weeks of treatment, the remaining animals will be fasted overnight, bled for serum samples (five animals/sex/group), anesthetized with sodium pentobarbital, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Postmortem Procedures

Necropsy

The necropsy will include an examination of the external features of the carcass; all external body orifices; the abdominal, thoracic, and cranial cavities; organs; and tissues.

Cell Proliferation Tissue Collection and Immunohistochemical Evaluation

At the Week 4 and 14 interim sacrifices, representative samples of the left lateral lobe of the liver and any macroscopic lesions of the liver will be collected and preserved in zinc formalin.

After fixation, each sample of liver will be embedded in paraffin, and the paraffin blocks will be shipped to:

Sandra R. Eldridge, PhD
Pathology Associates International
15 Worman's Mill Court, Suite I
Frederick, Maryland 21701
Telephone No.: 301.663.1644, ext. 2201
Facsimile No: 301.663.8994

Proliferation cell nuclear antigen (PCNA) evaluation will be done on the samples. In addition, liver sections will be stained with hematoxylin and eosin and examined microscopically. Results will be provided for inclusion in the final report.

Palmitoyl-CoA Oxidase Tissue Collection and Analyses

At the Week 4 and 14 interim sacrifices, a sample (approximately 500 mg) of the right lateral lobe of the liver will also be collected from each animal and flash-frozen in liquid nitrogen. The liver tissue will be stored in a freezer set to maintain -60 to -80°C until analyzed by Covance for palmitoyl-CoA oxidase activity.

Organ Weights

At the Week 79 and terminal sacrifices, the following organs (when present) will be weighed; paired organs will be weighed separately:

adrenal (2)
brain
kidney (2)
liver
lung
ovary (2)
spleen
testis (2)
thyroid (2) with parathyroid
uterus with cervix

Organ-to-body weight percentages and organ-to-brain weight ratios will be calculated.

Bone Marrow Smear

From the femur of each animal at the Week 79 and terminal sacrifices only; made but not examined

Additional Liver Sample Collection

A portion of the liver will be collected from five animals/sex/group at the interim and terminal sacrifices and stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

Tissue Preservation

The following tissues (when present) from each animal will be preserved in 10% neutral-buffered formalin:

adrenal (2)
brain
cecum
cervix
colon
duodenum
epididymis (2)
esophagus
eye (2)
femur with bone marrow (articular surface of the distal end)
Harderian gland
heart
ileum
jejunum
kidney (2)
lesions
liver
lung with mainstem bronchi
lymph node (mesenteric)
mammary gland (females only)
ovary (2)
pancreas
pituitary
prostate
rectum
salivary gland [mandibular (2)]
sciatic nerve
seminal vesicle (2)
skeletal muscle (thigh)

skin
spinal cord (cervical, thoracic, and lumbar)
spleen
sternum with bone marrow
stomach
testis (2)
thymus
thyroid (2) with parathyroid
trachea
urinary bladder
uterus
vagina

Histopathology
Tissues will be held for possible future examination.

Reports
One copy of each draft report will be sent to the Sponsor. The report for this study will be included as an appendix to the report for Covance 6329-212. The report will include the following information:

Experimental Design and Methods

Results
dose analyses
mortality
clinical observations
body weights
body weight changes
food consumption
test material consumption
clinical pathology results
palmitoyl CoA oxidase activities
macroscopic observations
cell proliferation assessments (provided by the Sponsor's designee)

Statistical Evaluation
body weights
body weight changes
food consumption
survival rates
clinical pathology values
palmitoyl CoA oxidase activities

Statistical methods will be those presented in Attachments Nos. 1 and 2. For each sex, Group 9 will be compared to Group 8 (Control).

At the end of 1 year after issuance of the audited draft report, if no requested revisions or instructions to finalize have been communicated by the Sponsor, then the audited draft report will be considered 'final' and issued as the final report, signed by the study director, and submitted to the Sponsor.

Any modifications or changes to the audited draft report requested 1 year after issuance will be performed at additional cost to the Sponsor.

Record Retention

All raw data, documentation, records, protocol, specimens, and final report generated as a result of this study, including those items listed below, will be archived in the storage facilities of Covance-Madison for a period of 1 year following submission of the final report to the Sponsor. All raw data stored on magnetic media, the protocol and protocol amendments, study correspondence, and the original report will be retained by Covance. One year after submission of the final report, all of the aforementioned materials will be sent to the Sponsor, and a return fee will be charged. The Sponsor may elect to have the materials retained in the Covance archives for an additional period of time, and Covance will charge a storage fee. If the Sponsor chooses to have Covance dispose of the materials, a disposal fee will be charged.

protocol and protocol amendments
dose preparation records
in-life records
  animal receipt
  acclimation
  animal room maintenance
  randomizations
  dose administration
  clinical observations
  body weights
  food consumption
  sample collection
clinical pathology records
anatomical pathology records
statistical analyses
study correspondence
tissue specimens (wet)
blood and bone marrow slides
final report (original signed copy)

The following supporting records will be retained at Covance-Madison but will not be archived with the study data.

feed analysis records
water analysis records
animal room environment records
refrigerator and freezer temperature records
room temperature records for test material storage
instrument calibration and maintenance records

PCNA evaluation data and paraffin blocks and tissue slides will be retained by Pathology Associates International.

Serum and liver samples sent to the Sponsor will be retained by the Sponsor

PROTOCOL APPROVAL

Andrew M. Seacat

Andrew M. Seacat, PhD
Study Monitor
3M

4/17/98
Date

Peter J. Thomford

Peter J. Thomford, PhD
Study Director
Covance Laboratories Inc.

4/1/98
Date

Attachment No. 1

Statistical Analyses

Levene's test will be done to test for variance homogeneity. In the case of heterogeneity of variance at $p \leq 0.05$, transformations will be used to stabilize the variance. Comparison tests will take variance heterogeneity into consideration.

One-way analysis of variance (ANOVA) will be used (if applicable) to analyze body weights, body weight changes, food consumption, continuous clinical pathology values, and organ weight data. If the ANOVA is significant, Student's t-test will be used for control versus treated group comparisons.

If the ANOVA shows significance for body weights at Week 1, one-way analysis of covariance (ANCOVA) will be used to analyze body weights, with initial body weights as the covariate. If the ANCOVA is significant, covariate-adjusted means will be used for control versus treated group comparisons.

Group comparisons (Group 9 versus Group 8) will be evaluated at the 5.0% two-tailed probability level. Only data collected on or after the first day of treatment will be analyzed statistically.

Attachment No. 2

Adjusted survival data are analyzed by the National Cancer Institute (NCI) lifetable package. The tests include: Graphical (Kaplan-Meier product-limit estimation curves), Cox-Tarone binary regression methods for trend and heterogeneity, and Gehan-Breslow nonparametric methods for trend and heterogeneity.

COVANCE
THE DEVELOPMENT SERVICES COMPANY

PROTOCOL AMENDMENT NO. 1

Covance 6329-228

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Sponsor:	3M, St. Paul, Minnesota
Study Monitor:	Andrew M. Seacat, PhD
Testing Facility:	Covance Laboratories Inc., Madison, Wisconsin
Study Director:	Peter J. Thomford, PhD

This amendment modifies the following portions of the protocol:

Effective April 23, 1998

1. **Page 11, Termination.** To reflect the decision to anesthetize animals to be sacrificed with carbon dioxide, delete the text in this section and replace with the following:

   Unscheduled Sacrifices and Deaths
   Necropsies will be done. Animals to be sacrificed will be anesthetized with carbon dioxide, weighed, and exsanguinated. A blood film will be taken as part of the necropsy procedure for sacrificed animals.

Scheduled Sacrifices

Interim Sacrifices
During Week 4, five animals/sex/group will be fasted overnight, bled for serum samples, anesthetized with carbon dioxide, weighed, and exsanguinated. The abdominal cavity of each animal will be opened, the liver will be removed and weighed, and liver samples will be collected. Animals will be discarded after liver collection.

During Week 14, five animals/sex/group will be fasted overnight, bled for serum samples, anesthetized with carbon dioxide, weighed, and exsanguinated. The abdominal cavity of each animal will be opened, the liver will be removed and weighed, and liver samples will be collected. Animals will be discarded after liver collection.

After at least 78 weeks of treatment, 10 animals/sex/group will be fasted overnight, bled for serum samples (five animals/sex/group), anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

Terminal Sacrifice
After at least 104 weeks of treatment, the remaining animals will be fasted overnight, bled for serum samples (five animals/sex/group), anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

2 **Page 12, Postmortem Procedures, Cell Proliferation Tissue Collection and Immunohistochemical Evaluation, Paragraph 1.** To reflect the decision to collect liver samples for proliferation cell nuclear antigen evaluation at Week 14 only, delete the text in this paragraph and replace with the following:

At the Week 14 interim sacrifice, representative samples of the left lateral lobe of the liver and any macroscopic lesions of the liver will be collected and preserved in zinc formalin.

Effective April 28, 1998

3. **Page 2, Alternate Study Monitor.** To include the alternate study monitor in the protocol, add the following after "Study Monitor:"

   Alternate Study Monitor
   Marvin T. Case, DVM, PhD
   3M Toxicology Services
   Telephone No.: 612.733.5180
   Facsimile No.: 612.733.1773

4. **Page 6, Group Designations and Dietary Levels, Footnote e.** To reflect the decision to change the Week 79 interim sacrifice to Week 53, delete the text in this footnote and replace with the following:

   Ten animals/sex/group will be designated as interim sacrifice animals and will be sacrificed after at least 52 weeks of treatment.

5. **Page 9, Clinical Pathology, Frequency and Number of Animals, Scheduled Collections, Paragraph 2.** The Week 79 interim sacrifice will be moved to Week 53, therefore blood films will be prepared from animals sacrificed after 52 weeks of treatment. To reflect this decision, delete the text in this paragraph and replace with the following:

   A blood film will be made and held for possible future examination from animals at scheduled sacrifices after at least 52 and 104 weeks of treatment.

6. **Page 10, Serum Analyses, Frequency and Number of Animals.** The Week 79 interim sacrifice will be moved to Week 53, therefore serum samples will be collected from animals sacrificed after 52 weeks of treatment. To reflect this decision, delete the text in this section and replace with the following:

Five animals/sex/group during Weeks 4 and 14 (animals selected for hepatocellular proliferation and biochemical analyses), five animals/sex/group (from animals selected for interim sacrifice) after at least 52 weeks of treatment, and five animals/sex/group at the terminal sacrifice

7. **Page 11, Termination, Scheduled Sacrifices, Interim Sacrifices, Paragraph 3.** To reflect the decision to change the Week 79 interim sacrifice to Week 53, delete the text in this paragraph and replace with the following:

   After at least 52 weeks of treatment, 10 animals/sex/group will be fasted overnight, bled for serum samples (five animals/sex/group), anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

8. **Page 12, Postmortem Procedures, Organ Weights.** To reflect the decision to record organ weights at the Week 53 interim sacrifice only, delete the text in this section and replace with the following:

   At the Week 53 interim sacrifice, the following organs (when present) will be weighed; paired organs will be weighed separately:

adrenal (2)	ovary (2)
brain	spleen
kidney (2)	testis (2)
liver	thyroid (2) with parathyroid
lung	uterus with cervix

   Organ-to-body weight percentages and organ-to-brain weight ratios will be calculated.

9. **Page 13, Postmortem Procedures, Bone Marrow Smear.** The Week 79 interim sacrifice will be moved to Week 53, therefore bone marrow smears will be collected from animals sacrificed after 52 weeks of treatment. To reflect this decision, delete the text in this section and replace with the following:

From the femur of each animal at the Week 53 and terminal sacrifices only; made but not examined

10. **Page 14, Reports, Paragraph 1.** To reflect the decision to provide unaudited summary reports after the Week 14 and 53 interim sacrifices, delete the text in this paragraph and replace with the following:

After the Week 14 and 53 interim sacrifices, unaudited summary reports will be sent to the Sponsor in conjunction with the summary reports for Covance 6329-212. The summary reports will include a brief description of methods and results and summary tables of in-life data, clinical pathology data, and anatomical pathology data. After completion of the study, one copy of each draft report will be sent to the Sponsor. The report for this study will be included as an appendix to the report for Covance 6329-212. The report will include the following information:

Effective May 15, 1998

11. **Page 10, Serum Analyses, Method of Collection.** To reflect the decision to increase the volume collected for serum analyses at the Week 53 and 105 sacrifices, delete the text in this section and replace with following:

Animals will be fasted overnight; blood (approximately 2 mL at the Week 4 and 14 sacrifices, approximately 3 mL at the Week 53 and 105 sacrifices) will be collected from a jugular vein. Samples will be collected without anticoagulant.

AMENDMENT APPROVAL

Andrew M. Seacat, PhD
Study Monitor
3M

Date 6/8/98

Peter J. Thornford, PhD
Study Director
Covance Laboratories Inc.

Date 6/2/98

COVANCE
THE DEVELOPMENT SERVICES COMPANY

PROTOCOL AMENDMENT NO. 2

Covance 6329-228

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Sponsor:	3M, St. Paul, Minnesota
Study Monitor:	Andrew M. Seacat, PhD
Testing Facility:	Covance Laboratories Inc., Madison, Wisconsin
Study Director:	Peter J. Thomford, PhD

This amendment modifies the following portions of the protocol:

Effective April 1, 1998

1. **Page 4.** To include the vehicle used to dissolve the test material before mixing with the diet, add the following section.

Vehicle

Identification
Acetone

Lot Numbers
The lot numbers will be maintained in the raw data.

Purity
On file with the manufacturer

Stability
On file with the manufacturer

Storage Conditions
At room temperature

Characteristics
Information on synthesis methods, composition, or other characteristics that define the vehicle is on file with the manufacturer.

Effective June 30, 1998

2. **Page 7, Dosing Procedure, Retention Samples.** To reflect the decision to send retention samples of control diet to the Sponsor, delete the text in this section and replace with the following:

Samples (approximately 100 g) will be taken from each dose preparation during the in-life phase and stored at room temperature. Unless used for analyses, retention samples from the treated groups will be discarded at least 1 month after completion of the in-life phase. Samples from control diets prepared for Weeks 14 and before will be shipped to:

Kris J. Hansen, PhD
3M Environmental Technology and Safety Services
935 Bush Avenue
Building 2-3E-09
St. Paul, Minnesota 55133-3331
Telephone No.: 651.778.6018
Facsimile No.: 651.778.6176

Effective July 20, 1998

3. **Page 2, Study Monitor and Alternate Study Monitor.** To indicate the change in the area code, delete the text in these sections and replace with the following:

Study Monitor
Andrew M. Seacat, PhD
3M
Telephone No.: 651.575.3161
Facsimile No.: 651.733.1773

Alternate Study Monitor
Marvin T. Case, DVM, PhD
3M Toxicology Services
Telephone No.: 651.733.5180
Facsimile No.: 651.733.1773

4. **Page 4, Disposition of Test Material.** To indicate the change in the area code, delete the text in these section and replace with the following:

After authorization from the Sponsor, any remaining test material will be returned to:

Andrew M. Seacat, PhD
3M
Toxicology Services
Building 220-2E-02, 3M Center
St. Paul, Minnesota 55144-1000
Telephone No.: 651.575.3161
Facsimile No.: 651.733.1733

5. **Page 10, Serum Analyses, Sample Handling.** To indicate the change in the area code for the Sponsor, delete the text in these section and replace with the following:

Blood samples will be allowed to clot at room temperature and centrifuged. Serum samples will be harvested and stored in a freezer set to maintain -60 to -80 C. Samples will be packed on dry ice and shipped to:

Kris J. Hansen, PhD
3M Environmental Technology and Safety Services
935 Bush Avenue
Building 2-3E-09
St. Paul, Minnesota 55133-3331
Telephone No.: 651.778.6018
Facsimile No.: 651.778.6176

Samples will be retained by the Sponsor for possible future analysis.

Effective April 9, 1999

6. **Page 9, Clinical Pathology, Frequency and Number of Animals, Scheduled Collections, Paragraph 2.** Hematology tests will be done for animals sacrificed at Week 53 and blood films will not be necessary. Therefore, delete this paragraph and replace with the following.

   A blood film will be made and held for possible future examination from animals at scheduled sacrifice after at least 104 weeks of treatment.

7. **Page 11, Termination, Scheduled Sacrifices, Interim Sacrifices, Paragraph 3.** To reflect the decision to collect clinical pathology samples from animals at the Week 53 interim sacrifice, delete the text in this paragraph and replace with the following:

   After at least 52 weeks of treatment, 10 animals/sex/group will be fasted overnight, bled for clinical pathology tests (all animals) and serum samples (five animals/sex/group), anesthetized with carbon dioxide, weighed, exsanguinated, and necropsied.

AMENDMENT APPROVAL

Andrew M. Seacat, PhD
Study Monitor
3M

Date 6/14/99

Peter J. Thomford, PhD
Study Director
Covance Laboratories Inc.

Date 02 June 1999

COVANCE
THE DEVELOPMENT SERVICES COMPANY

PROTOCOL AMENDMENT NO. 3

Covance 6329-228

104-Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

Sponsor:	3M, St. Paul, Minnesota
Study Monitor:	Andrew M. Seacat, PhD
Testing Facility:	Covance Laboratories Inc., Madison, Wisconsin
Study Director:	Peter J. Thomford, PhD

This amendment modifies the following portion of the protocol.

Effective January 26, 2000

1. **Page 14, Postmortem Procedures, Histopathology.** The Sponsor has requested that all tissues from animals in Group 8 and selected tissues from animals in Group 9 be examined microscopically. To reflect this decision, delete the text in this section and replace with the following.

Tissues (as appropriate) from each animal in Group 8 will be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically.

Lesions, liver, lungs, kidneys, pancreas, thyroid, testes, and mammary glands (females) from each animal in Group 9 will be embedded in paraffin, sectioned, stained with hematoxylin and eosin, and examined microscopically. Parathyroids will be processed with the thyroids, but will not be examined.

Effective February 18, 2000

2. **Page 10, Serum Analyses, Frequency and Number of Animals.** To reflect the decision to collect serum samples from 10 animals/sex/group at termination, delete the text in this section and replace with the following:

   Five animals/sex/group during Weeks 4 and 14 (animals selected for hepatocellular proliferation and biochemical analyses), five animals/sex/group (from animals selected for interim sacrifice) after at least 52 weeks of treatment, and 10 animals/sex/group at the terminal sacrifice

3. **Page 11, Termination, Scheduled Sacrifices, Terminal Sacrifice.** To reflect the decision to collect serum samples from 10 animals/sex/group at termination, delete the text in this section and replace with the following:

   After at least 104 weeks of treatment, the remaining animals will be fasted overnight, bled for serum samples (10 animals/sex/group), anesthetized with sodium pentobarbital, weighed, exsanguinated, and necropsied. A blood film will be taken as part of the necropsy procedure.

4. **Page 13, Postmortem Procedures, Additional Liver Sample Collection.** decision to collect liver samples from 10 animals/sex/group at termination, delete the text in this section and replace with the following:

   A portion of the liver will be collected from five animals/sex/group at the interim sacrifice and 10 animals/sex/group at the terminal sacrifice and stored in a freezer set to maintain -60 to -80°C. Samples will be packed on dry ice and shipped to Kris J. Hansen, PhD, 3M Environmental Technology and Safety Services. Samples will be retained by the Sponsor for possible future analysis.

AMENDMENT APPROVAL

Andrew M. Seacat, PhD
Study Monitor
3M

Date 2/25/2000

Peter J. Thomford, PhD
Study Director
Covance Laboratories Inc.

Date 23 FEB 2000

3M Environmental Laboratory

Protocol - Analytical Study Phase

104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

In-Vivo Study Reference Number: Covance # 6329-228
(RE) LAC 7/27/99
Study Number: FACT-~~051498.1~~ 050598.2
Test Material: T-6316

Name and Address of Sponsor: 3M Toxicology Services
Building 220-2E-02, 3M Center
St. Paul, MN 55144-1000

Name and Address of Testing Facility: 3M Environmental Technology and Services
935 Bush Avenue
St. Paul, MN 55106

Proposed Experimental Start Date: May 25, 1998
Proposed Experimental Termination Date: December 30, 2001

Method Numbers and Revisions
FACT-M-1.0, Extraction of Perfluorooctanesulfonate from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry
FACT-M-2.0, Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry
FACT-M-3.0, Extraction of Perfluorooctanesulfonate from Sera for Analysis using HPLC-Electrospray/Mass Spectrometry
FACT-M-4.0, Analysis of Sera Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry

Author: Lisa Clemen

Kris J. Hansen 5/22/98
Kris J. Hansen, PhD Date
Study Director

D J Bacon 7/15/99
Dale Bacon Date
Study Director Management

Andrew M. Seacat 5/25/99
Andrew M. Seacat, PhD Date
Sponsor Representative

1.0 PURPOSE

1.1 According to this analytical protocol, the 3M Environmental Laboratory will analyze the tissue and fluid samples from the Covance study number 6329-228, "104-Week Dietary Carcinogenicity with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats." The collected data will be provided to the sponsor for use in the assessment of toxicological effects of the test material when administered in the diets of rats for at least 104 weeks.

1.2 Data collected in the Environmental Laboratory will be considered non-quantitative screening data until future studies have been conducted to determine absolute recoveries of specific or general fluorochemical compounds.

2.0 REGULATORY COMPLIANCE

2.1 This analytical phase of the study will be conducted in accordance with the FDA Good Laboratory Practices Regulations 21 CFR 58, with the following exceptions:

2.1.1 The analytical phase is being conducted as a separate study and therefore has a separate Study Director, protocol, and final report, from those listed in the Covance protocol 6329-228.

2.1.2 The characterization of the reference material, including purity, identity, and stability, are the responsibility of the sponsor.

2.1.3 Sample storage stability will not be determined.

3.0 TEST MATERIALS

3.1 Control, and reference Materials and Matrices

3.1.1 Analytical Reference Material: T-6316, from 3M ICP/PCP Division

3.1.2 Analytical Reference Matrix: Rat liver, from Covance and rat serum, from Sigma Chemical Company.

3.1.3 Analytical Control Material: None.

3.1.4 Analytical Control Matrix: Rat liver, from Covance and rat serum, from Sigma Chemical Company.

3.2 Number of Test and Control Samples: Throughout the course of the study, liver and serum from 140 test animals and 140 control animals will be made available. Samples will be analyzed as requested by the sponsor or the study director. Other biological tissues (kidney, bile, dermal application site, and cellular fraction) will be available for analysis if deemed appropriate.

3.3 Identification of Test and Control Samples: The samples will be identified using the Covance animal identification number that consists of a letter and five-digit number, plus the tissue identity and day identity (serum).

3.4 Purity and Identity of Reference Material: To be determined by Sponsor.

3.5 Stability of Reference Material: To be determined by Sponsor.

3.6 Storage Conditions for Reference Materials: Reference materials will be stored at room temperature (3.1.1), and samples will be stored at -20 ± 10∞C (3.1.2, 3.1.4). Test and Control samples will be received according to AMDT-S-10-0.

3.7 Disposition of Specimens: Biological tissues and fluids will be retained per GLP Regulation for the time period required for studies longer than 28 days.

3.8 Safety Precautions: Refer to appropriate MSDS. Wear appropriate laboratory attire. Use caution when handling knives for cutting tissue samples.

4.0 EXPERIMENTAL - Overview

4.1 The tissues from animals dosed as described (Covance# 6329-228), will be available for analysis for fluorine-containing compounds. At the discretion of the Study Director, a series of analytical tests can be performed. All high dose and control sera and livers will be analyzed initially using HPLC-electrospray mass spectrometry to identify fluorine-containing compounds of interest present in the sera and liver (if any). The screening for organic fluoride in liver via combustion may be performed to present definitive data for fluorine in the liver. Based on the findings from these analyses, additional samples, tissues, or fluids may be analyzed at the discretion of the Study Director to determine the presence of fluorochemicals in these matrices.

5.0 EXPERIMENTAL - Methods

5.1 Methods (attached):

- **5.1.1 FACT-M-1.0,** "Extraction of Perfluorooctanesulfonate from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry"
- **5.1.2 FACT-M-2.0,** "Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry"
- **5.1.3 FACT-M-3.0,** "Extraction of Perfluorooctanesulfonate from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry"
- **5.1.4 FACT-M-4.0,** "Analysis of Serum Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry"

6.0 DATA ANALYSIS

6.1 Quality Control: Matrix spikes will be extracted and analyzed to determine accuracy of the method. Also, continuing calibration checks will be analyzed to determine response bias.

6.2 Transformations: Any transformations performed on data collected during the analytical phase of the study will be documented in the final report.

6.3 Statistics: At the discretion of the Study Director, statistics used may include regression analysis of serum concentrations with time, averages, and standard deviations of concentrations for the different dose groups. If necessary, simple tests such as the Student's t-test may be applied to determine statistical difference. Any statistical analysis performed will be documented in the final report.

6.4 Data Reporting: A final data package will be submitted to 3M Toxicology Services. The data package will include the following with additional data included as deemed appropriate.

6.4.1 A summary of individual sample results, reported as a concentration (weight/weight, weight/volume) of fluoride per tissue or fluid, or as the mass of a specific fluorochemical (HPLC-electrospray mass spectrometry) per unit of tissue or fluid.

6.4.2 A summary of quality control results (continuing calibration checks, method blanks, instrument blanks, matrix spikes, and matrix spike duplicates).

6.4.3 Certified copies or originals of the written validated methods.

6.4.4 Certified copies or originals of sample identification sheets sent from Covance.

6.4.5 Certified copies or originals of study specific raw data.

6.4.6 A summary of key personnel involved with the analytical phase of the study.

6.4.7 A signed QAU statement listing the dates of inspections and reports of findings to management and Study Director.

7.0 MAINTENANCE OF RAW DATA AND RECORDS

7.1 The following raw data and records (or certified copies thereof) will be maintained in the study folder in the archives according to appropriate SOPs.

7.1.1 Approved protocol
7.1.2 Approved methods
7.1.3 Data summaries
7.1.4 Study correspondence
7.1.5 Shipping records
7.1.6 Raw data
7.1.7 Electronic copies of data

7.2 Supporting records to be retained separately from the study folder in the archives according to 3M ET & SS SOPs, will include, but not necessarily be limited to the following:

7.2.1 Approved validation reports
7.2.2 Training records
7.2.3 Calibration records
7.2.4 Instrument maintenance logs
7.2.5 Standard operating procedures, equipment procedures, and methods
7.2.6 Appropriate specimens

8.0 REFERENCES

8.1 Approved AMDT standard operating procedures.

8.2 Approved ETS standard operating procedures.

9.0 ATTACHMENTS

9.1 **FACT-M-1.0,** Extraction of Perfluorooctanesulfonate from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry

9.2 **FACT-M-2.0,** Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry

9.3 **FACT-M-3.0,** Extraction of Perfluorooctanesulfonate from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry

9.4 **FACT-M-4.0,** Analysis of Serum Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry

Study Title

104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

PROTOCOL AMENDMENT NO. 1

Amendment Date:

20 January 2000

Performing Laboratory

3M Environmental Technology & Safety Services
3M Environmental Laboratory
935 Bush Avenue
St. Paul, MN 55106

Laboratory Project Identification

ET&SS LRN-U2104
FACT TOX-003
Covance Study: 6329-228
3M Medical Department Study: T-6316.1

3M Environmental Laboratory

This amendment modifies the following portion(s) of the protocol:

1. ***PROTOCOL READS:***
   The following methods will be used:
   FACT-M-1.0, Extraction of Perfluorooctanesulfonate from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry
   FACT-M-2.0, Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry
   FACT-M-3.0, Extraction of Perfluorooctanesulfonate from Sera for Analysis using HPLC-Electrospray/Mass Spectrometry
   FACT-M-4.0, Analysis of Sera Extracts for Fluorochemicals using HPLC-Electrospray/Mass Spectrometry
   AMEND TO READ:
   The following methods will be used:
   ETS-8-4.1, Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry
   ETS-8-5.1, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry
   ETS-8-6.0, Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry
   ETS-8-7.0, Analysis of Potassium Perfluorooctanesulfonate or Other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry
   REASON:
   The methods originally listed were superseded during the course of the study.

2. ***PROTOCOL READS:***
   There is no independent section of the protocol that addresses sample retention.
   AMEND TO READ:
   Specimens will be maintained in the 3M Environmental Laboratory specimen archives. Any specimens sent to sub-contract laboratories will be returned to the 3M Environmental Laboratory upon completion of analysis and submission of the sub-contract laboratory(s) final report. Specimens analyzed at sub-contract laboratories will be returned with the following documentation: the signed original chain of custody and records of storage conditions while at the sub-contract facility.
   REASON:
   To define in detail the appropriate disposition of specimens analyzed at subcontract laboratories.

3. ***PROTOCOL READS:***
Section 7 states that the following raw data and records will be retained in the study folder in the archives according to AMDT-S-8: Approved protocol and amendments; approved methods; data summaries; study correspondence; shipping records; raw data; and electronic copies of data. Additionally, Section 7 states that supporting records to be retained separately from the study folder in the archives according to AMDT-S-8 will include at least the following: Approved validation reports; training records; calibration records; instrument maintenance logs; Standard Operating Procedures, Equipment Procedures, and Methods; and appropriate specimens.
AMEND TO READ:
Section 7 states: "The original data, or copies thereof, will be available at the 3M Environmental Laboratory to facilitate audits of the study during its progress and before acceptance of the final report. When the final report is completed, all original paper data, including: approved protocol and amendments, study correspondence, shipping records, raw data, approved final report, and electronic copies of data will be retained in the archives of the 3M Environmental Laboratory. All corresponding training records, calibration records, instrument maintenance logs, standard operating procedures, equipment procedures, and methods will be retained in the archives of the facility performing each analysis."
REASON:
To direct subcontract laboratories in the disposition of the items listed above.

4. ***PROTOCOL READS:***
The study director for the present study was identified in the protocol as Kristen J. Hansen, Ph.D.
AMEND TO READ:
The role of study director for the present study was reassigned to John L. Butenhoff, Ph.D., as of 20 January 2000. The previous study director, Kristen Hansen, has been reassigned to the role of Principle Analytical Investigator.
REASON:
The role of study director was reassigned in an effort to ensure compliance with Good Laboratory Practice Standards that outline study personnel requirements (refer to 21 CFR Part 58).

5. ***PROTOCOL READS:***
The sponsor for the present study was identified as Andrew M. Seacat, Ph.D.
AMEND TO READ:
The role of sponsor for the present study was reassigned to Marvin T. Case, D.V.M., Ph.D., as of 20 January 2000.
REASON:
The change was made at the request of the sponsor.

Amendment Approval

Andrew Seacat 2/10/2000
Andrew M. Seacat, Ph.D., Outgoing Sponsor Representative Date

Kristen Hansen 11-Feb-2000
Kristen J. Hansen, Ph.D., Outgoing Study Director Date

Marvin T. Case 10 February 2000
Marvin T. Case, D.V.M., Ph.D., Incoming Sponsor Representative Date

John L. Butenhoff February 10, 2000
John L. Butenhoff, Ph.D., Incoming Study Director Date

3M Environmental Laboratory

Study Title
104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

PROTOCOL AMENDMENT NO. 3

Amendment Date:
May 4, 2001

Performing Laboratory
3M Environmental Technology & Safety Services
3M Environmental Laboratory
935 Bush Avenue
St. Paul, MN 55106

Project Identification
3M Medical Department Study: T-6316.1
Covance In-Life Study: 6329-228
Analytical Report: FACT TOX-003
3M Laboratory Request No. U2104

Exact Copy of Original
LAC 06/04/01
Initial Date

3M Environmental Laboratory

This amendment modifies the following portion(s) of the protocol:

1. ***PROTOCOL READS:***
   PURPOSE: The FACT TOX-003 protocol does not clearly identify the intended analytes for the study.

   AMEND TO READ:
   PURPOSE: Rat sera and liver samples will be analyzed for PFOS, PFOSA, PFOSAA, PFOSEA, EtFOSE-OH and M556.

   REASON:
   The target analytes will be clearly specified.

3M Environmental Laboratory

Amendment Approval

Marvin T Case — 25 May 2001

Marvin Case, D.M.V., Ph. D., Sponsor Representative — Date

John R. Butenhoff — June 5, 2001

John Butenhoff, Ph.D., Study Director — Date

Study Title

104-Week Dietary Carcinogenicity Study with Narrow Range N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats

PROTOCOL AMENDMENT NO. 2

Amendment Date:

January 8, 2001

Performing Laboratory

3M Environmental Technology & Safety Services
3M Environmental Laboratory
935 Bush Avenue
St. Paul, MN 55106

Laboratory Project Identification

ET&SS LRN-U2104
FACT TOX-003
Covance 6329-228
3M Medical Department Study: T-6316.1

3M Environmental Laboratory

Protocol FACT TOX-003
Amendment No. 2

This amendment modifies the following portion(s) of the protocol:

1. PROTOCOL READS:

1.2 Data collected in the Environmental Laboratory will be considered non-quantitative screening data until future studies have been conducted to determine absolute recoveries of specific or general fluorochemical compounds.

AMEND TO READ:

1.2 If matrix spike studies provide accurate representation of recovery of endogenous levels of PFOS, PFOSA, PFOSAA, PFOSEA, M556, and EtFOSE-OH, the 3M Environmental Laboratory will provide semi-quantitative data for sera and liver samples collected from test animals.

REASON:

Due to improved analytical methods, the decision was made to change the purpose of the study results from non-quantitative to semi-quantitative.

3M Environmental Laboratory

Protocol FACT TOX-003
Amendment No. 2

Amendment Approval

Marvin T Case — 10 Jan 2001

Marvin T. Case, D.V.M., Ph.D., Sponsor Representative — Date

John L. Butenhoff — 10 Jan 2001

John L. Butenhoff, Ph.D., Study Director — Date

3M Environmental Laboratory

Record of Deviation

I. Identification

Study / Project No.
TOX023

Deviation Type (Check one)
☐ SOP ☒ Method ☐ Equipment Procedure
☐ Protocol ☐ Other:

Document Number
ETS 8-7.0

Date(s) of occurrence
6-29-98

II. Description:

Required Procedure/process:
A curve is to be run prior to and following the sample extracts. The average of the two curves will be plotted by linear regression.

Actual Procedure/process:
The final curve was excluded since it was incomplete. The data is bracketed by the first curve and CCVs that met acceptance criteria.

III. Actions Taken:
(such as amendment issued, SOP revision, etc.)
A method deviation was written.

Recorded By
Marlene [illegible]

Date
5-8-01

IV. Impact on Study / Project

No adverse impact on the outcome of this study. LAC 05/11/01 [illegible] 05/22/01

Authorized By (Study Director / Project Lead)
John T. Butenhoff May 15, 2001

Date

Study Director: John Butenhoff Sponsor Representative: Marv Case Marvin Case 14 May 2001 RE LAC 06/01

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 14 May 2001
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. Tox003 Covance 6329-228

Deviation Type (Check one) ☐ SOP ☒ Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number: ETS-8-5.1

Date(s) of occurrence: 7-13-98

II. Description:

Required Procedure/process:

The CCVs must be within ±30% of the theoretical value.

Actual Procedure/process:

Data was entered even though the CCVS did not meet the acceptance criteria. Samples had been reanalyzed on HP3000 and the criteria was still not met.

III. Actions Taken:

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

Data was entered from the analysis from 7-13-98 and was flagged as not meeting criteria for CCV.

Recorded By: Marlene [signature] Date: 1-5-01

IV. Impact on Study / Project

The flagged data will not meet the accuracy parameters defined for the study.

KJR 01/05/01

Authorized By (Study Director / Project Lead): Marvin Case 10 Jan 2001 John Butenhoff 10 JAN 2001 Date

Sponsor Representative: Marvin Case Study Director: John Butenhoff

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 2
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-003 Covance 6329-228

Deviation Type (Check one) ☐ SOP X Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number(s): FACT-M-4.0, ETS-8-5.1

Date(s) of occurrence: 07/13/98, 04/21/00, 04/24/00, 08/15/00, 08/28/00, 08/30/00

II. Description:

Required Procedure/process:
Section 10.1.1 and 10.1.2 states: "Analyze a method blank and a matrix blank prior to each calibration curve."

Actual Procedure/process:
On several occasions, the blanks were either a) analyzed after the calibration curve or b) not analyzed at all for a particular MS analysis (08/30/00, Wk4, Serum).

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
This deviation was written.

Recorded By: Lisa A. Clemen Date: 05/03/01

IV. Impact on Study / Project

All blanks were analyzed at least once during the course of the study. When blanks were analyzed after the calibration curve, all control-of-bias practices were followed. In one analysis, blanks were not analyzed since these blanks had been run previously and did not require reanalysis. In this particular analysis (08/30/00, Wk 4, Serum) instrument blanks were used to determine limits of quantitation. This deviation has no adverse affect on these study data.

kjh 05/04/01

Authorized By *(Study Director / Project Lead)* John R. Butenhoff May 15, 2001 Date

Study Director: John Butenhoff
Sponsor Representative: Marv Case Marvin T Case 8 May 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 3
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No.
FACT-TOX-3 Covance 6329-228

Deviation Type (Check one): ☐ SOP ☒ Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number	Date(s) of occurrence
FACT-M-3.0 & ETS-8-4.1	7/15/98

II. Description:

Required Procedure/process:
Method requires 1.0ml of serum to be used in sample preparation.

Actual Procedure/process:
Initial sample submitted for analysis was less than 1.0ml. All of sample was used in sample prep - volumes ranged from 0.5 to 1.0ml (1 out of 20 samples at 1.0ml)

III. Actions Taken:
(such as amendment issued, SOP revision, etc.)

Submitted deviation

Recorded By	Date
R. Wynne	8/9/00

IV. Impact on Study / Project

Recent work indicates that sample volumes ≥0.5 mL do not impact results.

Authorized By (Study Director / Project Lead)	Date
Glen T. Butenhoff 9/15/00 — Kjh 08/28/00 — Marvin Case 15 Sept 2000	

Study Director: John Butenhoff Sponsor Rep: Marv Case

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 4
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No.tox003

Deviation Type (Check one) ☐ SOP √ Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number:FACT-M-2.0 Date(s) of occurrence: 08/07/1998

II. Description:

Required Procedure/process:
Section **10.2.1** Analyze a matrix spike and matrix spike duplicate with each analysis. With a minimum of 2 spikes per batch.

Actual Procedure/process: The matrix spike and duplicates were analyzed in an earlier run and were not re-run with this analysis.

III. Actions Taken:

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

This deviation from the stated method is noted.

Recorded By: Harold Johnson 5.4.01 Date May 4, 2001

IV. Impact on Study / Project

This will have no impact on the study.

hh 05/04/01

Authorized By *(Study Director / Project Lead)* Date

John T. Butenhoff May 15, 2001

Study Director: John Butenhoff
Sponsor Representative: Marv Case Marvin T Case 8 May 2001
3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 5
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No.
FACT-TOX-003 Covance 6329-228

Deviation Type (Check one)
☐ SOP ☐ Method ☐ Equipment Procedure
☐ Protocol ☒ Other:

Document Number	Date(s) of occurrence
21 CFR 58.120 (a)(11)	05/22/99

II. Description:

Required Procedure/process:
Sponsor Representative approval of analytical protocol prior to Study Director approval.

Actual Procedure/process:
The Study Director signed/approved the analytical protocol before the Sponsor Representative.

III. Actions Taken:
(such as amendment issued, SOP revision, etc.)

In the future, analytical protocols will be signed by the Sponsor Representative before the Study Director.

Recorded By	Date
Lisa A. Clemen	07/27/99

IV. Impact on Study / Project

This deviation will not adversely affect the outcome of this study

Authorized By (Study Director / Project Lead)	Date
John T. Buckenhoff May 15, 2001 Kitty Hon	7/28/99

Study Director: John Buckenhoff Sponsor Representative: Mary Case
Mary T Case 8 May 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 6
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-003 Covance 6329-228

Deviation Type (Check one) ☐ SOP X Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number(s): ETS-8-7.0 Date(s) of occurrence: 04/14/00, 04/17/00

II. Description:

Required Procedure/process:
Section 14.3.1 states, "The r^2 value for the calibration curve must be 0.980 or better."
Section 14.3.6 states, "A valid curve must contain at least five active points."

Actual Procedure/process:
The EtFOSE-OH calibration curve for the Week 104 liver generated on 04/14/00 had an r^2 value of 0.9687.
A four point curve was used to plot the EtFOSE-OH calibration curve for the Week 53 liver, generated on 04/17/00.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
These data were flagged in the raw data and this deviation was written.

Recorded By: Lisa A. Clemen Date: 05/03/01

IV. Impact on Study / Project

EtFOSE-OH data is flagged as qualitative, so these data will not be adversely affected.
kjr 05/04/01

Authorized By *(Study Director / Project Lead)*: John T. Butenhoff May 15, 2001 Date

Study Director: John Butenhoff
Sponsor Representative: Marv Case Marvin T Case 8 May 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 7
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No.
Fact - TOX-003 / Covance 6329-228

Deviation Type (Check one)
☐ SOP ☐ Method ☒ Equipment Procedure
☐ Protocol ☐ Other:

Document Number: ETS-9-27.0, Section 13.1

Date(s) of occurrence: June 30, 2000 & July 5, 2000

II. Description:

Required Procedure/process:

When a Bottle-top Dispenser is used on a GLP project, it must be calibrated before use daily at the volume the Bottle-top Dispenser will be used on the project, and by the operator who will be using the Bottle-top Dispenser on the project.

Actual Procedure/process:

The Calibration Check logbook indicated that March 1, 2000 was the last time the Bottle-top Dispenser was calibrated.

III. Actions Taken:

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

The Bottle-top Dispenser will be calibrated before use daily when GLP projects are conducted at the the volume used on the project and by the operator who will be using the Bottle-top Dispenser on the project. This deviation was written.

Recorded By: Kelly Kuehlwein

Date: 8/8/00

IV. Impact on Study / Project

Standards + samples were prepared identically, therefore no impact on the study.

kh 08/29/00

Authorized By *(Study Director / Project Lead)*: John T. Butenhoff 9/15/00 Marvin T. Case 15 Sept 2000

Date:

Study Director: John Butenhoff Sponsor Rep: Marv Case

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 8
(assigned by Study Director or Project Lead at the end of study or project)

Record of Deviation

I. Identification

Study / Project No. FACT-TOX-003 Covance 6329-228

Deviation Type (Check one) ☐ SOP X Method ☐ Equipment Procedure ☐ Protocol ☐ Other:

Document Number(s): ETS-8-5.1 Date(s) of occurrence: 08/30/00

II. Description:

Required Procedure/process:
A five point curve or better is used for plotting the calibration curve.

Actual Procedure/process:
A four point curve was used for plotting the PFOSAA calibration curve for the Week 4 serum curve, generated on 08/30/00.

III. Actions Taken:

(such as amendment issued, SOP revision, etc.)
These data were flagged in the raw data and this deviation was written.

Recorded By: Lisa A. Clemen Date: 05/03/01

IV. Impact on Study / Project

These data will not be adversely affected.
all other QC parameters were met. kjh 05/04/01

Authorized By *(Study Director / Project Lead)*: John T. Butenhoff May 15, 2001 Date

Study Director: John Butenhoff
Sponsor Representative: Marv Case Marvin Case 8 May 2001

3M Environmental Laboratory
Form ETS-4-8.0

Deviation No. 9
(assigned by Study Director or Project Lead at the end of study or project)

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Appendix C: Extraction and Analytical Methods

This appendix includes the following methods:

Preparatory Methods

FACT-M-1.0. Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry, (8 pages)

FACT-M-3.0, Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry, (8 pages)

ETS-8-4.1, Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Serum for Analysis using HPLC-Electrospray/Mass Spectrometry, (14 pages)

ETS-8-6.0, Extraction of Potassium Perfluorooctanesulfonate or other Fluorochemical Compounds from Liver for Analysis using HPLC-Electrospray/Mass Spectrometry, (14 pages)

Analytical Methods

FACT-M-2.0, Analysis of Liver Extracts for Fluorochemicals Using HPLC-Electrospray/Mass Spectrometry, (8 pages)

FACT-M-4.0, Analysis of Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry, (8 pages)

ETS-8-5.1, Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemicals in Serum Extracts Using HPLC-Electrospray/Mass Spectrometry, (9 pages)

ETS-8-7.0, Analysis of Potassium Perfluorooctanesulfonate or other Fluorochemicals in Liver Extracts Using HPLC-Electrospray/Mass Spectrometry, (10 pages)

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER ANIONIC FLUOROCHEMICAL SURFACTANTS FROM LIVER FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: FACT-M-1.0 **Adoption Date:** 5/26/98

Revision Date: N/A

Author: Lisa Clemen

Approved By:

Laboratory Manager 5/26/98 Date

Group Leader 5/26/98 Date

Technical Reviewer 5/27/98 Date

Exact Copy of Original
Initial BSD
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the extraction of Potassium Perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from liver.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds.

1.3 Matrices: Rabbit, rat, bovine, and monkey livers or other livers as designated in the validation report.

2.0 Summary of Method

2.1 This method describes how to extract potassium perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from liver using ion pairing reagent and 5.0 mLs of ethyl acetate. An ion pairing reagent is added to each sample and partitioned into ethyl acetate. Four mLs of extract is removed to a centrifuge tube and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 1.0 mL methanol then filtered through a 3 cc plastic syringe attached to a 0.2 µm filter into glass autovials.

3.0 Definitions

3.1 None.

4.0 Warnings and Cautions

4.1 Health and Safety Warnings:

4.1.1 Use universal precautions when handling animal livers, they may contain pathogens.

5.0 Interferences

5.1 There are no known interferences at this time.

6.0 Equipment

6.1 The following equipment is used while carrying out this method. Equivalent equipment is acceptable.

6.1.1 Ultra-Turrax T25 Grinder for grinding liver samples
6.1.2 Vortex mixer, VWR, Vortex Genie 2
6.1.3 Centrifuge, Mistral 1000 or IEC
6.1.4 Shaker, Eberbach or VWR
6.1.5 Nitrogen Evaporator, Organomation
6.1.6 Balance

7.0 Supplies and Materials

7.1 Gloves
7.2 Dissecting scalpels
7.3 Eppendorf or disposable pipettes
7.4 Nalgene bottles, capable of holding 250 mL and 1 L
7.5 Glass, type A, volumetric flasks
7.6 40 mL glass I-CHEM vials
7.7 Plastic sampule vials, Wheaton, 6 mL
7.8 Polypropylene centrifuge tubes, 15 mL
7.9 Labels

7.10 Syringes, capable of measuring 10 µL to 50 µL

7.11 Glass, type A, volumetric pipettes

7.12 Graduated pipettes

7.13 Electronic pipettor, Eppendorf or equivalent

7.14 Timer

7.15 Disposable plastic 3 cc syringes

7.16 Filters, nylon syringe filters, 0.2 µm, 25 mm

7.17 Crimp cap autovials

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 Reagents and Standards

8.1 Reagents

8.1.1 Sodium Hydroxide (J.T Baker or equivalent), (NaOH) 10N: weigh approximately 200 grams NaOH. Pour into a 1000 mL beaker containing 500 liters (L) Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L nalgene bottle.

8.1.2 Sodium Hydroxide (J.T Baker or equivalent), (NaOH) 1N. Dilute 10N 1:10. Measure 10 mL of the 10N NaOH solution into a 100 mL volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 mL nalgene bottle.

8.1.3 Tetrabutylammonium hydrogen sulfate (Kodak or equivalent), (TBA) 0.5M: Weigh approximately 169 grams of TBA into a 1 L volumetric containing 500 L Milli-Q™ water. Adjust to pH 10 using approximately 64 mL 10N NaOH and dilute to volume with Milli-Q™ water. Add NaOH slowly while adding the last 1 mL of NaOH because the pH changes abruptly. Store in a 1 L nalgene bottle.

8.1.3.1 TBA requires a check prior to each use to ensure pH = 10. Adjust as needed using 1N NaOH solution.

8.1.4 Sodium carbonate/Sodium Bicarbonate Buffer (J.T. Baker or equivalent), (Na_2CO_3/$NaHCO_3$) 0.25M: Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and dilute to volume with Milli-Q™ water. Store in a 1 L nalgene bottle.

8.1.5 PFOS (3M Specialty Chemical Division), molecular weight = 538.

8.1.6 Ethyl Acetate, Omnisolv, glass distilled or HPLC grade.

8.1.7 Methanol, Omnisolv, glass distilled or HPLC grade.

8.1.8 Liver and control liver, received frozen from testing laboratory.

8.1.9 Milli-Q™ water, all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus system.

8.2 Standards

8.2.1 Prepare PFOS standards for the standard curve.

8.2.2 Weigh approximately 100 mg of PFOS into a 100 mL volumetric flask and record the actual weight.

8.2.3 Bring to volume with methanol for a stock standard of approximately 1000 ppm (µg/mL).

8.2.4 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.2.5 Dilute the stock solution with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.2.6 Dilute the stock solution with methanol for a working standard 3 solution of approx. 0.50 ppm.

9.0 SAMPLE HANDLING

9.1 All livers are received frozen and must be kept frozen until the extraction is performed.

10.0 QUALITY CONTROL

10.1 Matrix Spikes

10.1.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.1.2 Prepare each spike using liver chosen by the analyst, usually a control liver.

10.1.3 Expected concentrations will fall in the mid-range of the initial calibration curve.

10.2 Continuing Calibration Checks

10.2.1 Prepare and analyze continuing calibration check samples to determine the continued linearity of the initial calibration curve.

10.2.2 One check is prepared per group of ten samples. For example, if a sample set = 34, four checks are prepared and extracted.

10.2.3 Prepare each continuing calibration check from the same liver homogenate used to prep the initial curve.

10.2.4 The expected concentration will fall within the mid-range of the initial calibration curve.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare Liver Homogenate to Use for Standards

11.1.1 Weigh approximately 40 g of liver into a 250 mL Nalgene bottle containing 200 mLs Milli-Q™ water. Grind to a homogeneous solution.

11.1.2 If 40 g is not available, use appropriate amounts of liver and water in keeping with a 1:5 ratio.

11.1.3 See section 13.0 to calculate the actual density of liver.

11.1.4 Add 1 mL of homogeneous solution to a 15 mL centrifuge tube. Re-suspend homogeneous solution by shaking between aliquots while preparing a total of sixteen 1 mL aliquots of homogeneous solution in 15 mL centrifuge tubes.

11.1.5 Two 1 mL aliquots serve as matrix blanks. Use the standard concentrations and spiking amounts listed in table 1 to spike, in duplicate, two standard curves for a total of fourteen samples.

Table 1
Approximate Spiking Amounts for Calibration Standards

Working Standard (Approx. Conc.)	µL	Approx. final conc. of PFOS in liver
-	-	Blank
0.50 ppm	4	0.010 ppm
0.50 ppm	20	0.050 ppm
0.50 ppm	40	0.100 ppm
5.0 ppm	10	0.250 ppm
5.0 ppm	20	0.500 ppm
5.0 ppm	30	0.750 ppm
50 ppm	4	1.000 ppm

11.1.1 See section 13.0 to calculate actual concentrations of PFOS in calibration standards.

11.2 Extract spiked liver homogenates following **12.14-12.24** of this method. Use these standards to establish each initial curve on the mass spectrometer.

12.0 PROCEDURES

12.1 Obtain frozen liver samples. In spent tissue, note that the liver has not been packaged with other tissues.

12.2 Cut approximately 1 g of liver using a dissecting scalpel.

12.3 Weigh the sample directly into a tared plastic sampule vial.

12.4 Record the liver weight in the study notebook.

12.5 Label the sampule vial with the study number, weight, liver ID, date and analyst initials.

12.6 Add 2.5 mLs of water to sampule vial.

12.7 Grind the sample. Put the grinder probe in the sample and grind for about 2 minutes, or until the sample is homogeneous.

12.8 Rinse the probe into the sample with 2.5 mLs water using a pipette.

12.9 Take the grinder apart and clean it with methanol after each sample. Follow AMDT-EP-22.

12.10 Cap the sample and vortex for 15 seconds.

12.11 Pipette 1 mL homogenate into a 15 mL polypropylene centrifuge tube. Label the centrifuge tube with the identical information as the sampule vial. (See Worksheet for documenting the remaining steps.)

12.12 Spike liver homogenates with the appropriate amount of PFOS standard as described in section 11.1 or Table 1.

12.13 Pipette two 1 mL aliquots of Milli-Q™ water to centrifuge tubes. These will serve as instrument blanks.

12.14 Add 1 mL 0.5 M TBA and 2 mL of the 0.25 M sodium carbonate/sodium bicarbonate buffer.

12.15 Using a volumetric pipette, add 5 mLs ethyl acetate.

12.16 Cap each sample and put on the shaker for 20 minutes.

12.17 Centrifuge for 20 to 25 minutes, until layers are well separated. Set power on the centrifuge to approximately 3500 rpm.

12.18 Remove 4 mLs of organic layer, using a 5 mL graduated glass pipette, to a clean 15 mL centrifuge tube. Label this fresh tube with the same information as in **12.5**.

12.19 Put each sample on the analytical nitrogen evaporator until dry, approximately 2 to 3 hours.

12.20 Add 1.0 mL of methanol to each centrifuge tube using a graduated pipette.

12.21 Vortex mix for 30 seconds.

12.22 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 mL glass autovial.

12.23 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, and analyst(s) who performed the extraction.

12.24 Cap and hold for electrospray mass spectrometry analysis.

12.25 Complete the worksheet and tape to page of study notebook.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.1 Calculate the density of liver (mg) in 1.0 mL homogenate using the following equation:

$$\frac{\text{g of Liver} \times \text{Average weight of ten 1 mL aliquots (mg)}}{(\text{g of Liver} + \text{g of Water})}$$

13.1.2 Calculate actual concentrations of PFOS in calibration standards using the following equation:

$$\frac{\mu L \text{ of Standard} \times \text{Concentration } (\mu g/mL)}{\text{mg Liver}^{*} / 1 \text{ mL homogenate}} = \text{Final Concentration } (\mu g/g \text{ or } mg/kg) \text{ of PFOS in Liver}$$

*Average weight of liver in solution as determined in **13.1.1**, by weighing ten 1 mL homogenates of approximately 40 mg liver in 200 mL of Milli-Q water.

14.0 METHOD PERFORMANCE

14.1 The method detection limit is equal to half the lowest standard in the calibration curve.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

16.0 RECORDS

16.1 Complete the extraction worksheet and tape into the study notebook.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 The validation report associated with this method is FACT-M-1.0 & 2.0-V-1.

18.0 REFERENCES

18.1 AMDT-EP-22, "Routine Maintenance of Ultra-Turrax T-25"

19.0 AFFECTED DOCUMENTS

19.1 FACT-M-2, "Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

Extraction Worksheet for FACT-M-1

Study #	Sample Number set #	PFOS approx. 0.5 ppm actual ppm #W_____	PFOS approx. 5 ppm actual ppm #W_____	PFOS approx. 50 ppm actual ppm #W_____	Date and Initials for Std.
-	H_2O Blank	-	-	-	
-	Liver Blank	-	-	-	
-					
-					
-					
-					
-					
-					
-					
-					
[1]		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
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		-	-	-	
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		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

[1] Study number where the original worksheet is located.

Blank Liver Homogenate: Std.# Liver amount = g

Liver Extraction Method : **Date & Initials**

Vortex 15 sec.

Pipette 1 mL of Liver Solution

Pipette 1 mL of †0.5 M TBA, pH 10. Std. #_____

Pipette 2 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ Buffer Std. #_____

Pipette 5 mL of Ethyl Acetate TN-A-_____

Shake 20 min.

Centrifuge 20-25 min. Centrifuge Speed

Remove a 4 mL aliquot of organic layer

Put on Nitrogen Evaporator to dryness Evaporator Temperature

Add 1.0 mL of Methanol TN-A-_____

Vortex 30 sec.

Filter using a 3cc B-D syringe with a 0.2µm SRI filter into a 1.5 mL autosample vial

MS/MSD/___ Cont. Checks: Spiked _____ uL of a _____ ppm std (_____) for a final concentration of _____ ppm. MS/MSD used sample _____. Cont. Checks used same homogenate as for std curve.

FACT-M-1.0 Page 8 of 8
Extraction of PFOS from Liver

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER ANIONIC FLUOROCHEMICAL SURFACTANTS FROM SERUM FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: FACT-M-3.0 **Adoption Date:** 4/22/98

Revision Date: N/A

Author: Lisa Clemen

Approved By:

Laboratory Manager 4/22/98 Date

Group Leader 4/22/98 Date

Technical Reviewer 4/22/98 Date

Exact Copy of Original
Initial RSO
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the extraction of potassium perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from serum.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds.

1.3 Matrices: Rabbit, rat, and bovine serum or other sera as designated in the validation report.

2.0 Summary of Method

2.1 This method describes how to extract potassium perfluorooctanesulfonate (PFOS) or other anionic fluorochemical surfactants from serum using an ion pairing reagent and 5.0 mL of ethyl acetate. An ion pairing reagent is added to the sample and the analyte ion pair is partitioned into ethyl acetate. Four mL of extract are removed and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 1.0 mL of methanol, then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

3.0 Definitions

3.1 None.

4.0 Warnings and Cautions

4.1 Health and Safety Warnings:

4.1.1 Use universal precautions, especially laboratory coats, goggles, and gloves when handling animal serum, it may contain pathogens.

5.0 Interferences

5.1 There are no known interferences at this time.

6.0 Equipment

6.1 The following equipment is used while carrying out this method. Equivalent equipment is acceptable.

6.1.1 Vortex mixer, VWR, Vortex Genie 2
6.1.2 Centrifuge, Mistral 1000 or IEC
6.1.3 Shaker, Eberbach or VWR
6.1.4 Nitrogen evaporator, Organomation
6.1.5 Balance, (± 0.100 gm)

7.0 Supplies and Materials

7.1 Gloves
7.2 Eppendorf or disposable pipettes
7.3 Nalgene bottles, capable of holding 250 mL and 1 L
7.4 Glass, type A, volumetric flasks
7.5 40 mL glass I-CHEM vials
7.6 Polypropylene centrifuge tubes, 15 mL
7.7 Labels
7.8 Syringes, capable of measuring 10 µL to 50 µL
7.9 Glass, type A, volumetric pipettes
7.10 Graduated pipettes

7.11 Electronic pipettor, Eppendorf or equivalent

7.12 Timer

7.13 Disposable plastic 3 cc syringes

7.14 Filters, nylon syringe filters, 0.2 µm, 25 mm

7.15 Crimp cap autovials

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 Reagents and Standards

8.1 Reagents

8.1.1 Sodium hydroxide (J.T Baker or equivalent), (NaOH) 10N: weigh approximately 200 grams NaOH. Pour into a 1000 mL beaker containing 500 liters (L) Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L Nalgene bottle.

8.1.2 Sodium hydroxide (J.T Baker or equivalent), (NaOH) 1N. Dilute 10N 1:10. Measure 10 mL of 10N NaOH solution into a 100 mL volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 mL Nalgene bottle.

8.1.3 Tetrabutylammonium hydrogen sulfate (Kodak or equivalent), (TBA) 0.5M: Weigh approximately 169 grams of TBA into a 1 L volumetric containing 500 L Milli-Q™ water. Adjust to pH 10 using approximately 64 mL of 10N NaOH and dilute to volume with Milli-Q™ water. Add NaOH slowly while adding the last mL of NaOH because the pH changes abruptly. Store in a 1 L Nalgene bottle.

8.1.3.1 TBA requires a check prior to each use to ensure pH = 10. Adjust as needed using 1N NaOH solution.

8.1.4 Sodium carbonate/sodium bicarbonate buffer (J.T. Baker or equivalent), (Na_2CO_3/$NaHCO_3$) 0.25M: Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and bring to volume with Milli-Q™ water. Store in a 1 L nalgene bottle.

8.1.5 PFOS (3M Specialty Chemical Division), molecular weight = 538.

8.1.6 Other fluorochemicals, as appropriate.

8.1.7 Ethyl Acetate, Omnisolv, glass distilled or HPLC grade.

8.1.8 Methanol, Omnisolv, glass distilled or HPLC grade.

8.1.9 Serum, frozen liquid from Sigma.

8.1.10 Control serum received with each sample set.

8.1.11 Milli-Q™ water, all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus system.

8.2 Standards

8.2.1 Prepare PFOS standards for the standard curve.

8.2.2 Prepare other fluorochemical standards, as appropriate.

8.2.3 Weigh approximately 100 mg of PFOS into a 100 mL volumetric flask and record the actual weight.

8.2.4 Bring to volume with methanol for a stock standard of approximately 1000 ppm (µg/mL).

8.2.5 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.2.6 Dilute the stock solution with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.2.7 Dilute the stock solution with methanol for a working standard 3 solution of approx. 0.50 ppm.

9.0 SAMPLE HANDLING

9.1 All sera are received frozen and must be kept frozen until the extraction is performed.

10.0 QUALITY CONTROL

10.1 Matrix Blanks and Method Blanks

10.1.1 Two 1.0 mL aliquots of the serum are extracted following this procedure and used as matrix blanks. See section 11.1.2.

10.1.2 Two 1.0 mL aliquots of Milli-Q™ water are extracted following this procedure and used as method blanks.

10.2 Matrix Spikes

10.2.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.2.2 Prepare each spike using serum chosen by the analyst, usually control serum received with each sample set.

10.2.3 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spikes may be included and may fall in the low-range of the initial calibration curve.

10.3 Continuing Calibration Checks

10.3.1 Prepare and analyze continuing calibration check samples to determine the continued linearity of the initial calibration curve.

10.3.2 One check is prepared per group of ten samples. For example, if a sample set = 34, four checks are prepared and extracted.

10.3.3 Prepare each continuing calibration check from the same serum used to prep the initial curve.

10.3.4 The expected concentration will fall within the mid-range of the initial calibration curve.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare Serum Standards

11.1.1 Transfer 1 mL of serum to a 15 mL centrifuge tube.

11.1.2 If the majority of serum sample volumes are less than 1.0 mL, extract standards using serum volumes in the standards equal to the serum volumes in samples. Do not extract below 0.50 mL of serum. Record the serum volume on the extraction sheet.

11.1.3 Mix or shake between aliquots while preparing a total of sixteen aliquots of serum in 15 mL centrifuge tubes.

11.1.4 Two 1 mL or appropriate aliquots serve as matrix blanks. Typically use the standard concentrations and spiking amounts listed in table 1 to spike, in duplicate, two standard curves for a total of fourteen samples.

11.1.5 Refer to the validation report FACT-M-3.0-V-1 and FACT-M-4.0-V-1 which lists the working ranges for calibration curves.

Table 1
Approximate Spiking Amounts for Standards and Spikes Using 1.0 mL of Serum

Working Standard (Approx. Conc.)	µL	Approx. final conc. of PFOS in serum
-	-	Blank
0.500 ppm	20	0.010 ppm
5.00 ppm	5	0.025 ppm
5.00 ppm	10	0.050 ppm
5.00 ppm	20	0.100 ppm
50.0 ppm	5	0.250 ppm
50.0 ppm	10	0.500 ppm
50.0 ppm	15	0.750 ppm

11.1.4 See section 13.0 to calculate actual concentrations of PFOS in calibration standards.

11.2 Extract spiked serum standards following **12.6-12.16** of this method. Use these standards to establish each initial curve on the mass spectrometer.

12.0 PROCEDURES

12.1 Obtain frozen serum samples and allow to thaw.

12.2 Vortex mix for 15 seconds then remove 1.0 mL or appropriate volume to a 15 mL polypropylene centrifuge tube.

12.3 Return serum samples to freezer after extraction amount has been removed.

12.4 Record the serum volume on the extraction worksheet. The final methanol volume will equal the initial serum volume.

12.5 Label the tube with the study number, serum ID, date and analyst initials. See attached worksheet for documenting the remaining steps.

12.6 Spike serum with the appropriate amount of PFOS standard as described in section 11.1 or Table I for the calibration curve standards. Also spike matrix spikes and continuing calibration standards.

12.7 Vortex mix the standard curve samples, matrix spike samples, and continuing calibration samples for 15 seconds.

12.8 Add 1 mL 0.5 M TBA and 2 mL of the 0.25 M sodium carbonate/sodium bicarbonate buffer.

12.9 Using a volumetric pipette, add 5 mL ethyl acetate.

12.10 Cap each sample and put on the shaker for 20 minutes.

12.11 Centrifuge for 20 to 25 minutes, until layers are well separated. Set power on the centrifuge to approximately 3500 rpm.

12.12 Transfer 4 mL of organic layer, using a 5 mL graduated glass pipette, to a clean 15 mL centrifuge tube. Label this fresh tube with the same information as in **12.5**.

12.13 Put each sample on the analytical nitrogen evaporator until dry, approximately 2 to 3 hours.

12.14 Add 1.0 mL or appropriate volume of methanol to each centrifuge tube using a graduated pipette. (This volume equals the initial volume of serum used for the extraction.)

12.15 Vortex mix for 30 seconds.

12.16 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 mL glass autovial.

12.17 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, and analyst(s) who performed the extraction.

12.18 Cap and hold for HPLC-electrospray/mass spectrometry analysis. Extracts may be stored at 4° C until analysis.

12.19 Complete the extraction worksheet, attached to this document, and tape to page of study notebook.

13.0 Data Analysis and Calculations

13.1 Calculations:

13.1.1 Calculate actual concentrations of PFOS, or other appropriate fluorochemical, in calibration standards using the following equation:

$$\frac{\text{mL of Standard x Concentration (µg /mL)}}{\text{mL of Standard + Initial Serum Volume (mL)}} = \text{Final Concentration (µg/mL) of PFOS in Serum}$$

14.0 Method Performance

14.1 The method detection limit is equal to half the lowest standard in the calibration curve.

15.0 Pollution Prevention and Waste Management

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

16.0 Records

16.1 Complete the extraction worksheet attached to this method, and tape into the study notebook.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 The validation report associated with this method is FACT-M-3.0 & 4.0-V-1.

18.0 References

18.1 None

19.0 Affected Documents

19.1 FACT-M-4, "Analysis of Serum Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

20.0 Revisions

Revision Number.	Reason For Revision	Revision Date

Extraction Worksheet for FACT-M-3

Study #	Sample Number set #	PFOS approx. 0.5 ppm actual ppm #W_____	PFOS approx. 5 ppm actual ppm #W_____	PFOS approx. 50 ppm actual ppm #W_____	Date and Initials for Std. or Comments
-	H_2O Blank	-	-	-	
-	Serum Blank	-	-	-	
-					
-					
-					
-					
-					
-					
-					
[1]		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
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		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

[1] Study number where the original worksheet is located.

Blank Serum Std # Serum amount = g

Serum Extraction Method : **Date & Initials**

Vortex 15 sec.

Pipette Serum Volume mL

Pipette 1 mL of 0.5 M TBA, pH 10. Std. #_____

Pipette 2 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ buffer Std. #_____

Pipette 5 mL of ethyl acetate TN-A-_____

Shake 20 min.

Centrifuge 20-25 min. Centrifuge speed:

Remove a 4 mL aliquot of organic layer

Put on Nitrogen Evaporator to dryness Evaporator #: Temperature:

Add methanol Volume_____ mL TN-A-_____

Vortex 30 sec.

Filter using a 3cc B-D syringe with a 0.2µm SRI filter into a 1.5 mL autosample vial

MS/MSD/___ Cont. Checks: Spiked _____ uL of a _____ ppm std (_____) for a final concentration of _____ ppm. MS/MSD used sample _____. Cont. Checks used same serum as for std curve.

FACT-M-3.0 Page 8 of 8
Extraction of PFOS from Serum

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICAL COMPOUNDS FROM SERUM FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-4.1 **Adoption Date:** 03/01/99

Revision Date: 4/27/99

Author: Lisa Clemen, Glenn Langenburg

Approved By:

[signature]	4/27/99
Laboratory Manager	Date
[signature]	4/26/99
Group Leader	Date
Lisa A Clemen	04/26/99
Technical Reviewer	Date

Exact Copy of Original
RSD
Initial
6/5/01
Date

1.0 SCOPE AND APPLICATION

1.1 **Scope:** This method is for the extraction of potassium perfluorooctanesulfonate (PFOS) or other fluorochemical compounds from serum.

1.2 **Applicable compounds:** Fluorochemical surfactants or other fluorinated compounds.

1.3 **Matrices:** Rabbit, rat, bovine, monkey, and human serum or other fluids as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the procedure for extracting potassium perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from serum, or other fluids, using an ion pairing reagent and methyl-*tert*-butyl ether (MtBE). In this method, seven fluorochemicals were extracted: PFOS, PFOSA, PFOSAA, EtFOSE-OH, PFOSEA, M556, and surrogate standard (see *3.0 **Definitions***). An ion pairing reagent is added to the sample and the analyte ion pair is partitioned into MtBE. The MtBE extract is removed and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 1.0 mL of methanol, then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

2.2 These sample extracts are analyzed following method ETS-8-5.1 or other appropriate methods.

3.0 Definitions

3.1 PFOS: perfluorooctanesulfonate (anion of potassium salt) $C_8F_{17}SO_3^-$

3.2 PFOSA: perfluorooctane sulfonylamide $C_8F_{17}SO_2NH_2$

3.3 PFOSAA: perfluorooctane sulfonylamido (ethyl)acetate $C_8F_{17}SO_2N(CH_2CH_3)CH_2CO_2^-$

3.4 EtFOSE-OH: 2(N-ethylperfluorooctane sulfonamido)-ethyl alcohol $C_8F_{17}SO_2N(CH_2CH_3)CH_2CH_2OH$

3.5 PFOSEA: perfluorooctane sulfonyl ethylamide $C_8F_{17}SO_2N(CH_2CH_3)H$

3.6 M556: $C_8F_{17}SO_2N(H)(CH_2COOH)$

3.7 Surrogate standard: 1H-1H-2H-2H perfluorooctane sulfonic acid

4.0 Warnings and Cautions

4.1 **Health and safety warnings**

4.1.1 Use universal precautions, especially laboratory coats, goggles, and gloves when handling animal tissue, which may contain pathogens.

5.0 Interferences

5.1 There are no interferences known at this time.

6.0 Equipment

6.1 The following equipment is used while performing this method. Equivalent equipment is acceptable.

6.1.1 Vortex mixer, VWR, Vortex Genie 2

6.1.2 Centrifuge, Mistral 1000 or IEC

6.1.3 Shaker, Eberbach or VWR

6.1.4 Nitrogen evaporator, Organomation

6.1.5 Balance (± 0.100 g)

7.0 Supplies and Materials

7.1 Gloves

7.2 Eppendorf or disposable pipettes

7.3 Nalgene bottles, capable of holding 250 mL and 1 L

7.4 Volumetric flasks, glass, type A

7.5 I-CHEM vials, glass, 40 mL glass

7.6 Centrifuge tubes, polypropylene, 15 mL

7.7 Labels

7.8 Oxford Dispenser – 3.0 to 10.0 mL

7.9 Syringes, capable of measuring 5 µL to 50 µL

7.10 Graduated pipettes

7.11 Syringes, disposable plastic, 3 cc

7.12 Syringe filters, nylon, 0.2 µm, 25 mm

7.13 Timer

7.14 Crimp cap autovials and caps

7.15 Crimpers

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 Reagents and Standards

8.1 Type I reagent grade water, Milli-Q™ or equivalent; all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus™ system

8.2 Sodium hydroxide (NaOH), J.T Baker or equivalent

8.3 Tetrabutylammonium hydrogen sulfate(TBA), Kodak or equivalent

8.4 Sodium carbonate (Na_2CO_3), J.T. Baker or equivalent

8.5 Sodium bicarbonate ($NaHCO_3$), J.T. Baker or equivalent

8.6 Methyl-T-Butyl Ether, Omnisolv, glass distilled or HPLC grade

8.7 Methanol, Omnisolv, glass distilled or HPLC grade

8.8 Serum or blood, frozen from supplier

8.9 **Fluorochemical standards**

8.9.1 PFOS (3M Specialty Chemical Division), molecular weight = 538

8.9.2 PFOSA (3M Specialty Chemical Division), molecular weight = 499

8.9.3 PFOSAA (3M Specialty Chemical Division), molecular weight = 585

8.9.4 EtFOSE-OH (3M Specialty Chemical Division), molecular weight = 570

8.9.5 PFOSEA (3M Specialty Chemical Division), molecular weight = 527

8.9.6 M556 (3M Specialty Chemical Division), molecular weight = 557

8.9.7 Surrogate standard: 4-H, perfluorooctane sulfonic acid (1-H,1-H, 2-H, 2-H $C_8F_{13}SO_3H$) molecular weight = 428

8.9.8 Other fluorochemicals, as appropriate

8.10 Reagent preparation

NOTE: When preparing larger volumes than listed in reagent, standard, or surrogate preparation, adjust accordingly.

8.10.1 10 N sodium hydroxide (NaOH): Weigh approximately 200 g NaOH. Pour into a 1000 mL beaker containing 500 mL Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L Nalgene bottle.

8.10.2 1 N sodium hydroxide (NaOH): Dilute 10 N NaOH 1:10. Measure 10 mL of 10 N NaOH solution into a 100 mL volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 mL Nalgene bottle.

8.10.3 0.5 M tetrabutylammonium hydrogen sulfate (TBA): Weigh approximately 169 g of TBA into a 1 L volumetric containing 500 mL Milli-Q™ water. Adjust to pH 10 using approximately 44 to 54 mL of 10 N NaOH (While adding the last mL of NaOH, add slowly because the pH changes abruptly). Dilute to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.10.3.1 TBA requires a check prior to each use to ensure pH = 10. Adjust as needed using 1 N NaOH solution.

8.10.4 0.25 M sodium carbonate/sodium bicarbonate buffer ($Na_2CO_3/NaHCO_3$): Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and bring to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.11 Standards preparation

8.11.1 Prepare PFOS standards for the standard curve.

8.11.2 Prepare other fluorochemical standards, as appropriate. Multicomponent fluorochemical standards are acceptable (for example, one working standard solution containing 1.00 ppm PFOS, 1.02 ppm PFOSA, 0.987 ppm PFOSAA, and 1.10 ppm EtFOSE-OH.)

8.11.3 Weigh approximately 100 mg of PFOS into a 100 mL volumetric flask and record the actual weight.

8.11.4 Bring to volume with methanol for a stock standard of approximately 1000 ppm (µg/mL).

8.11.5 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.11.6 Dilute working standard 1 with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.11.7 Dilute working standard 1 with methanol for a working standard 3 solution of approx. 0.50 ppm.

8.12 Surrogate stock standard preparation

8.12.1 Weigh approximately 50-60 mg of surrogate standard 1-H,1-H, 2-H, 2-H, $C_8F_{13}SO_3H$ into a 50 mL volumetric flask and record the actual weight.

8.12.2 Bring to volume with methanol for a surrogate stock of approximately 1000-1200 ppm.

8.12.3 Prepare a surrogate working standard. Transfer approximately 1 mL of surrogate stock to a 10 mL volumetric flask and bring to volume with methanol for a working standard of 100 ppm. Record the actual volume transferred.

9.0 SAMPLE HANDLING

9.1 All samples are received frozen and must be kept frozen until the extraction is performed.

9.2 Allow samples to thaw to room temperature prior to extraction.

10.0 QUALITY CONTROL

10.1 Solvent Blanks, Method blanks and matrix blanks

10.1.1 An aliquot of 1.0 mL methanol is used as a solvent blank.

10.1.2 Extract two 1.0 mL aliquots of Milli-Q™ water following this procedure and use as method blanks.

10.1.3 Extract two 1.0 mL aliquots of the serum following this procedure and use as matrix blanks. See **11.1.4**.

10.2 Matrix spikes

10.2.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.2.2 Prepare each spike using a sample chosen by the analyst, usually the control matrix received with each sample set.

10.2.3 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spikes may be included and may fall in the low-range of the initial calibration curve.

10.2.4 Prepare one matrix spike and matrix spike duplicate per 40 samples, with a minimum of 2 matrix spikes per batch.

10.3 Continuing calibration checks

10.3.1 Prepare continuing calibration check samples to ensure the accuracy of the initial calibration curve.

10.3.2 Prepare, at a minimum, one continuing check per group of 10 samples. For example, if a sample set = 34, four checks are prepared and extracted.

10.3.3 Prepare each continuing calibration check from the same matrix used to prepare the initial curve.

10.3.4 The expected concentrations will fall within the mid-range of the initial calibration curve. Additional spikes may be included that fall in the low-range of the initial calibration curve. This is necessary if the analyst must quantitate using only the low end of the calibration curve (for example, 5 ppb – 100 ppb, rather than
5 ppb – 1000 ppb).

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare matrix calibration standards

11.1.1 Transfer 1 mL of serum to a 15 mL centrifuge tube.

11.1.2 If most sample volumes are less than 1.0 mL, extract standards with matrix volumes equal to the sample volumes. Do not extract less than 0.50 mL of matrix. Record each sample volume on the extraction sheet.

11.1.3 While preparing a total of twenty aliquots in 15 mL centrifuge tubes, mix or shake between aliquots.

11.1.4 Two 1 mL aliquots, or other appropriate volume, serve as matrix blanks. Typically use the standard concentrations and spiking amounts listed in Table 1, at the end of this section, to spike, in duplicate, two standard curves, for a total of eighteen standards, two matrix blanks, and two method blanks.

11.1.5 Refer to validation report **ETS-8-4.0 & ETS-8-5.0-V-1,** which lists the working ranges and the Linear Calibration Range (LCR) for calibration curves.

11.1.6 Use Attachment D as an aid in calculating the concentrations of the working standards. See Section **13.0** to calculate actual concentrations of PFOS in calibration standards.

11.2 To each standard, blank, or continuing check, add appropriate amount of surrogate working standard for the concentration to fall within the calibration curve range 5 ppb - 1000 ppb.

11.3 Extract spiked matrix standards following **12.6-12.16** of this method. Use these standards to establish each initial curve on the mass spectrometer.

Table 1 Approximate spiking amounts for standards and spikes Using 1.0 mL of matrix		
Working standard (approx. conc.)	µL	Approx. final conc. of analyte in matrix
-	-	Blank
0.500 ppm	10	0.005 ppm
0.500 ppm	20	0.010 ppm
5.00 ppm	5	0.025 ppm
5.00 ppm	10	0.050 ppm
5.00 ppm	20	0.100 ppm
50.0 ppm	5	0.250 ppm
50.0 ppm	10	0.500 ppm
50.0 ppm	15	0.750 ppm
50.0 ppm	20	1.00 ppm

12.0 PROCEDURE

12.1 Obtain frozen samples and allow to thaw at room temperature or in a lukewarm waterbath.

12.2 Vortex mix for 15 seconds, then transfer 1.0 mL or other appropriate volume to a 15 mL polypropylene centrifuge tube.

12.3 Return unused samples to freezer after extraction amounts have been removed.

12.4 Record the initial volume on the extraction worksheet.

12.5 Label the tube with the study number, sample ID, date and analyst initials. See attached worksheet for documenting the remaining steps.

12.6 Spike all samples, including blanks and standards, ready for extraction with surrogate standard as described in **11.2**.

12.7 Spike each matrix with the appropriate amount of standard as described in **11.1**, or **Table 1** in that section, for the calibration curve standards. Also prepare matrix spikes and continuing calibration standards.

12.8 Vortex mix the standard curve samples, matrix spike samples, and continuing calibration samples for 15 seconds.

12.9 Check to ensure the 0.5 M TBA reagent is at pH 10. If not, adjust accordingly.

12.10 To each sample, add 1 mL 0.5 M TBA and 2 mL of 0.25M sodium carbonate/sodium bicarbonate buffer.

12.11 Using an Oxford Dispenser, add 5 mL methyl-*tert*-butyl ether.

12.12 Cap each sample and put on the shaker at a setting of 300 rpm, for 20 minutes.

12.13 Centrifuge for 20 to 25 minutes at a setting of 3500 rpm, or until layers are well separated.

12.14 Label a fresh 15 mL centrifuge tube with the same information as in **12.5**.

12.15 Remove 4.0 mL of the organic layer to this clean 15 mL centrifuge tube.

12.16 Put each sample on the analytical nitrogen evaporator until dry, approximately 1 to 2 hours.

12.17 Add 1.0 mL of methanol to each centrifuge tube using a graduated pipette.

12.18 Vortex mix for 30 seconds.

12.19 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 mL glass autovial or low-volume autovial when necessary.

12.20 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, and analyst(s) performing the extraction.

12.21 Cap and store extracts at room temperature or at approximately 4 °C until analysis.

12.22 Complete the extraction worksheet, attached to this document, and tape in the study notebook or include in study binder, as appropriate.

13.0 Data Analysis and Calculations

13.1 Calculations

13.1.1 Calculate actual concentrations of PFOS, or other applicable fluorochemical, in calibration standards using the following equation:

$$\frac{\text{mL of standard} \times \text{concentration of standard } (\mu g/mL)}{\text{mL of standard} + \text{mL of surrogate standard} + \text{initial matrix volume (mL)}} =$$

Final Concentration (µg/mL) of PFOS in matrix

14.0 Method Performance

14.1 The method detection limit (MDL) is analyte and matrix specific. Refer to MDL report for specific MDL and limit of quantitation (LOQ) values (see **Attachments B and C**).

14.2 The following quality control samples are extracted with each batch of samples to evaluate the quality of the extraction and analysis.

14.2.1 Method blanks and matrix blanks.

14.2.2 Matrix spike and matrix spike duplicate samples to determine accuracy and precision of the extraction.

14.2.3 Continuing calibration check samples to determine the continued accuracy of the initial calibration curve.

14.3 Refer to section 14 of ETS-8-5.1 for method performance criteria.

15.0 Pollution Prevention and Waste Management

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

16.0 RECORDS

16.1 Complete the extraction worksheet attached to this method, and tape in the study notebook or include in the 3-ring study binder, as appropriate.

17.0 ATTACHMENTS

17.1 Attachment A, Extraction worksheet

17.2 Attachment B, MDL/LOQ values and summary

17.3 Attachment C, Calibration standard concentration worksheet

18.0 REFERENCES

18.1 The validation report associated with this method is **ETS-8-4.0 & 5.0-V-1.**

18.2 FACT-M-3.1, "Analysis of Serum or Other Fluid Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

19.0 AFFECTED DOCUMENTS

19.1 ETS-8-5.1, "Analysis of Serum or Other Fluid Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

20.0 REVISIONS

Revision Number	Reason For Revision	Revision Date
1	Section 12.21 Changed to include sample storage at room temperature. Section 12.13 Added the shaker speed. Section 12.17 Final volume is 1.0 mL; not adjusted for initial volumes less than 1.0 mL.	04/02/99

Extraction Worksheet ETS-8-4.1

Study #_____ Matrix_____ Box #_____ Wk/Day_____ DateSpiked/Analyst	Surrogate Std approx. ppm actual ppm #_____	FC-Mix approx. 0.5 pm actual ppm #_____	FC-Mix approx. 5 ppm actual ppm #_____	FC-Mix approx. 50 ppm actual ppm #_____	Comments
CCV					
MS					
MSD					
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

Blank	Std #	amount =	mL
Serum Extraction Method	:		**Date & Initials**
Vortex 15 sec.			
Pipette Matrix	Volume	mL	
Pipette 1 mL of 0.5 M TBA, pH 10. pH = _____	Std. #_____		
Pipette 2 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ buffer	Std. #_____		
Dispense 5 mL of methyl-t-butyl ether	TN-A-_____		
Shake 20 min.	Shaker speed:		
Centrifuge 20-25 min.	Centrifuge speed:		
Remove a 4 mL aliquot of organic layer			
Put on Nitrogen Evaporator to dryness	Temperature:		
Add methanol	Volume_____ mL	TN-A-_____	
Vortex 30 sec.			
Filter using a 3cc B-D syringe with a 0.2µm filter into a 1.5 mL autosample vial			

Cont. Cal. Verifications used same matrix as for std curve.

MDL/LOQ values for rabbit serum

Compound	MDL (ppb)	LOQ (ppb)	Linear Calibration Range (LCR) Approximate concentrations to be used for preparing the Standard Calibration Curve
PFOS	1.74	5.55	5 ppb - 1000 ppb
PFOSA	1.51	4.79	5 ppb - 1000 ppb
PFOSAA	3.46	20.5	5 ppb - 1000 ppb
EtFOSE-OH	11.4	36.2	5 ppb - 1000 ppb
M556	6.03	19.2	5 ppb - 1000 ppb
PFOSEA	5.71	18.2	5 ppb - 1000 ppb

MDL/LOQ values in rat, bovine, monkey, and human serum, and monkey plasma were not statistically determined. Two curves in each of these matrices were extracted and analyzed with the rabbit serum curves to determine equivalence. Responses in the rat, bovine, monkey, and human were equivalent to the rabbit responses, therefore, their MDL and LOQ will be the same values as determined in rabbit serum.

Please see LOQ Summary and MDL study in ETS-8-4.0 & 5.0-V-1 for further information.

Compound: PFOS

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.995 - 978	24.8 - 978	83-108	4.67-11.0
Low Curve	4.94 - 248	4.94 - 248	85-104	5.34-12.0
High curve	97.8 - 978	97.8 – 978	85-106	4.84-9.80
1/X	0.995 - 978	4.94 - 978	94-111	4.60-10.5

Compound: PFOSA

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.993 - 976	4.93 - 976	88-103	5.10-14.7
Low Curve	4.93 - 97.6	4.93 - 97.6	87-105	9.85-14.7
High curve	24.8 - 976	24.8 - 978	93-102	5.08-13.9
1/X	0.993 - 976	4.93 - 976	94-103	5.10-14.5

Compound: PFOSAA

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.991 - 974	24.7 - 974	81-111	4.18-10.6
Low Curve	4.92 - 247	9.74 - 247	97-107	6.38-21.8
High curve	49.2 - 974	97.4 - 974	85-108	4.33-12.5
1/X	0.991 - 974	9.74 - 974	95-115	4.11-23.2

Compound: EtFOSE-OH

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.993 - 976	49.3 - 976	77-110	11.2-25.5
Low Curve	4.93 – 97.6	9.76 – 97.6	97-107	14.1-21.3
High curve	49.3 - 976	97.6 - 976	90-109	11.5-19.6
1/X	0.993 - 493	9.76 - 976	86-111	11.1-21.2

Compound: PFOSEA

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.993 - 976	24.8 - 976	96-106	10.1-16.2
Low Curve	4.93 - 248	9.76 - 248	91-110	11.8-19.5
High curve	49.3 - 976	49.3 - 976	86-106	10.2-18.2
1/X	0.993 - 976	9.76 - 976	95-117	10.1-19.1

Compound: M556

Rabbit Serum	Prepared range of standards (ppb) (ng/mL)	LCR from curve (ppb) (ng/mL)	% Recovery Range	RSD Range
Full Range	0.993 - 976	24.8 - 976	88-106	4.82-17.9
Low Curve	4.93 - 97.6	9.76 – 97.6	100-105	5.95-18.2
High curve	97.6 - 976	97.6 - 976	81-111	5.11-9.74
1/X	0.993 - 976	9.76 - 976	97-110	4.77-19.5

Ion Pair Standard Curves – Fluids

Prep date(s):
Analyte(s):
Sample matrix:

Standard number:
Equipment number:
Final solvent and TN:
Blank fluid/identifier:

Method/revision:
Target analyte(s):
FC mix std approx. 0.500 ppm:
FC mix std approx. 5.00 ppm:
FC mix std approx. 50.0 ppm:
Surrogate std approx. 100 ppm:

Actual concentrations of standards in the FC mix

PFOS Std conc ug/mL	PFOSA Std conc ug/mL	PFOSAA Std conc ug/mL	EtFOSE Std conc ug/mL	PFOSEA Std conc ug/mL	M556 Std conc ug/mL	All Am't spiked mL	All Final vol mL
0.500	0.507	0.532	0.501	0.521	0.501	0.010	1.015
0.500	0.507	0.532	0.501	0.521	0.501	0.020	1.025
5.00	5.07	5.32	5.01	5.21	5.01	0.005	1.010
5.00	5.07	5.32	5.01	5.21	5.01	0.010	1.015
5.00	5.07	5.32	5.01	5.21	5.01	0.020	1.025
50.0	50.1	53.2	50.1	52.1	50.1	0.005	1.010
50.0	50.1	53.2	50.1	52.1	50.1	0.010	1.015
50.0	50.1	53.2	50.1	52.1	50.1	0.015	1.020
50.0	50.1	53.2	50.1	52.1	50.1	0.020	1.025

Calculated concentrations of standards in the sample matrix

PFOS Final conc ng/mL	PFOSA Final conc ng/mL	PFOSAA Final conc ng/mL	EtFOSE Final conc ng/mL	PFOSEA Final conc ng/mL	M556 Final conc ng/mL	Surrogate Std conc ng/mL	All Am't spiked mL
4.93	5.00	5.24	4.94	5.01	5.13	100	0.005
9.76	9.89	10.4	9.78	9.93	10.2		
24.8	25.1	26.3	24.8	25.2	25.8	**Surrogate**	
49.3	50.0	52.4	49.4	50.1	51.3	Final conc	
97.6	98.9	104	97.8	99.3	102	ng/mL	
248	251	263	248	252	258	500	
493	500	524	494	501	513		
735	746	782	737	749	766		
976	989	1038	978	993	1017		

Validated ranges – approximate concentrations

Serum	PFOS	PFOSA	PFOSAA	EtFOSE-OH	PFOSEA	M556
Rabbit	5.00-1000	5.00-1000	5.00-1000	5.00-1000	5.00-1000	5.00-1000
Bovine	Estimates only. Use values for rabbit.					
Rat	Estimates only. Use values for rabbit.					
Monkey & Plasma	Estimates only. Use values for rabbit.					
Human	Estimates only. Use values for rabbit.					

3M ENVIRONMENTAL LABORATORY

METHOD

EXTRACTION OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICAL COMPOUNDS FROM LIVER FOR ANALYSIS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-6.0 **Adoption Date:** 07/22/99

Revision Date: NA

Author: Lisa Clemen, Robert Wynne

Approved By:

Laboratory Manager — Date 7/22/99

Group Leader — Date 7/14/99

Technical Reviewer — Date 07/14/99

Exact Copy of Original
Initial RSO
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the extraction of potassium perfluorooctanesulfonate (PFOS) or other fluorochemical compounds from liver.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds.

1.3 Matrices: Rabbit, rat, bovine, and monkey livers or other tissues as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the procedure for extracting potassium perfluorooctanesulfonate (PFOS) or other fluorochemical surfactants from liver, or other tissues, using an ion pairing reagent and methyl-*tert*-butyl ether (MtBE). In this method, seven fluorochemicals can be extracted: PFOS, PFOSA, PFOSAA, EtFOSE-OH, PFOSEA, M556, and surrogate standard. An ion pairing reagent is added to the sample and the analyte ion pair is partitioned into MtBE. The MtBE extract is transferred to a centrifuge tube and put onto a nitrogen evaporator until dry. Each extract is reconstituted in 1.0 mL methanol then filtered through a 3 cc plastic syringe attached to a 0.2 µm nylon filter into glass autovials.

2.2 These sample extracts are analyzed following method ETS-8-7.0 or other appropriate methods.

3.0 Definitions

3.1 PFOS: perfluorooctanesulfonate (anion of potassium salt) $C_8F_{17}SO_3$

3.2 PFOSA: perfluorooctane sulfonylamide $C_8F_{17}SO_2NH_2$

3.3 PFOSAA: perfluorooctane sulfonylamido (ethyl)acetate $C_8F_{17}SO_2N(CH_2CH_3)CH_2CO_2$

3.4 EtFOSE-OH: 2(N-ethylperfluorooctane sulfonamido)-ethyl alcohol $C_8F_{17}SO_2N(CH_2CH_3)CH_2CH_2OH$

3.5 PFOSEA: perfluorooctane sulfonyl ethylamide $C_8F_{17}SO_2N(CH_2CH_3)H$

3.6 M556: $C_8F_{17}SO_2N(H)(CH_2COOH)$

3.7 Surrogate standard: 1H-1H-2H-2H perfluorooctane sulfonic acid

4.0 Warnings and Cautions

4.1 **Health and Safety Warnings:**

4.1.1 Use universal precautions, especially laboratory coats, goggles, and gloves when handling animal tissue, which may contain pathogens.

5.0 Interferences

5.1 There are no interferences known at this time.

6.0 Equipment

6.1 The following equipment is used while performing this method. Equivalent equipment is acceptable.

6.1.1 Ultra-Turrax T25 Grinder for grinding liver samples

6.1.2 Vortex mixer, VWR, Vortex Genie 2

6.1.3 Centrifuge, Mistral 1000 or IEC

6.1.4 Shaker, Eberbach or VWR

6.1.5 Nitrogen Evaporator, Organomation

6.1.6 Balance (sensitivity to 0.100 g)

7.0 Supplies and Materials

7.1 Gloves

7.2 Dissecting scalpels

7.3 Eppendorf or disposable pipettes

7.4 Nalgene bottles, capable of holding 250 mL and 1 L

7.5 Volumetric flasks, glass, type A

7.6 I-CHEM vials, 40 mL glass

7.7 Plastic sampule vials, Wheaton, 6 mL (or appropriate size)

7.8 Centrifuge tubes, polypropylene, 15 mL

7.9 Labels

7.10 Oxford Dispensor – 3.0 to 10.0 ml

7.11 Syringes, capable of measuring 5 µL to 50 µL

7.12 Graduated pipettes

7.13 Syringes, disposable plastic, 3 cc

7.14 Syringe filters, nylon, 0.2 µm, 25 mm

7.15 Timer

7.16 Crimp cap autovials and caps

7.17 Crimpers

Note: Prior to using glassware and bottles, rinse 3 times with methanol and 3 times with Milli-Q™ water. Rinse syringes a minimum of 9 times with methanol, 3 rinses from 3 separate vials.

8.0 Reagents and Standards

8.1 Type I reagent grade water, Milli-Q™ or equivalent; all water used in this method should be Milli-Q™ water and be provided by a Milli-Q TOC Plus™ system

8.2 Sodium hydroxide (NaOH), J.T Baker or equivalent

8.3 Tetrabutylammonium hydrogen sulfate(TBA), Kodak or equivalent

8.4 Sodium carbonate (Na_2CO_3), J.T. Baker or equivalent

8.5 Sodium bicarbonate ($NaHCO_3$), J.T. Baker or equivalent

8.6 Methyl-*tert*-butyl ether, Omnisolv, glass distilled or HPLC grade

8.7 Methanol, Omnisolv, glass distilled or HPLC grade

8.8 Liver, frozen from supplier

8.9 Dry ice from supplier

8.10 **Fluorochemical standards**

8.10.1 PFOS (3M Specialty Chemical Division), molecular weight = 538

8.10.2 PFOSA (3M Specialty Chemical Division), molecular weight = 499

8.10.3 PFOSAA (3M Specialty Chemical Division), molecular weight = 585

8.10.4 EtFOSE-OH (3M Specialty Chemical Division), molecular weight = 570

8.10.5 PFOSEA (3M Specialty Chemical Division), molecular weight = 527

8.10.6 M556 (3M Specialty Chemical Division), molecular weight = 557

8.10.7 Surrogate standard: 4-H, perfluorooctane sulfonic acid (1-H,1-H, 2-H, 2-H $C_8F_{13}SO_3H$) molecular weight = 428

8.10.8 Other fluorochemicals, as appropriate

8.11 Reagent preparation

NOTE: When preparing larger volumes than listed in reagent, standard, or surrogate preparation, adjust accordingly.

8.11.1 10 N sodium hydroxide (NaOH): Weigh approximately 200 g NaOH. Pour into a 1000 mL beaker containing 500 mL Milli-Q™ water, mix until all solids are dissolved. Store in a 1 L Nalgene bottle.

8.11.2 1 N sodium hydroxide (NaOH): Dilute 10 N NaOH 1:10. Measure 10 mL of 10 N NaOH solution into a 100 mL volumetric flask and dilute to volume using Milli-Q™ water. Store in a 125 mL Nalgene bottle.

8.11.3 0.5 M tetrabutylammonium hydrogen sulfate (TBA): Weigh approximately 169 g of TBA into a 1 L volumetric containing 500 mL Milli-Q™ water. Adjust to pH 10 using approximately 44 to 54 mL of 10 N NaOH (While adding the last mL of NaOH, add slowly because the pH changes abruptly). Dilute to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.11.3.1 TBA requires a check prior to each use to ensure pH = 10. Adjust as needed using 1 N NaOH solution.

8.11.4 0.25 M sodium carbonate/sodium bicarbonate buffer ($Na_2CO_3/NaHCO_3$): Weigh approximately 26.5 g of sodium carbonate (Na_2CO_3) and 21.0 g of sodium bicarbonate ($NaHCO_3$) into a 1 L volumetric flask and bring to volume with Milli-Q™ water. Store in a 1 L Nalgene bottle.

8.12 Standards preparation

8.12.1 Prepare PFOS standards for the standard curve.

8.12.2 Prepare other fluorochemical standards, as appropriate. Multicomponent fluorochemical standards are acceptable (for example, one working standard solution containing 1.00 ppm PFOS, 1.02 ppm PFOSA, 0.987 ppm PFOSAA, and 1.10 ppm EtFOSE-OH.)

8.12.3 Weigh approximately 100 mg of PFOS into a 100 mL volumetric flask and record the actual weight.

8.12.4 Bring to volume with methanol for a stock standard of approximately 1000 ppm (μg/mL).

8.12.5 Dilute the stock solution with methanol for a working standard 1 solution of approximately 50 ppm.

8.12.6 Dilute the stock solution with methanol for a working standard 2 solution of approx. 5.0 ppm.

8.12.7 Dilute the stock solution with methanol for a working standard 3 solution of approx. 0.50 ppm.

8.13 Surrogate stock standard preparation

8.13.1 Weigh approximately 50-60 mg of surrogate standard 1-H,1-H, 2-H, 2-H, $C_8F_{13}SO_3H$ into a 50 ml volumetric flask and record the actual weight.

8.13.2 Bring to volume with methanol for a surrogate stock of approximately 1000-1200 ppm.

8.13.3 Prepare a surrogate working standard. Transfer approximately 1.0 ml of surrogate stock to a 10 ml volumetric flask and bring to volume with methanol for a working standard of 10-20 ppm. Record the actual volume transferred.

9.0 SAMPLE HANDLING

9.1 All samples are received frozen and must be kept frozen until the extraction is performed.

10.0 QUALITY CONTROL

10.1 Matrix blanks and method blanks

10.1.1 An aliquot of 1.0 mL methanol is used as a solvent blank.

10.1.2 Extract two 1.0 mL aliquots of Milli-Q™ water following this procedure and use as method blanks.

10.1.3 Extract two 1.0 mL aliquots of liver homogenate following this procedure and use as matrix blanks. Refer to **11.1.6**.

10.2 Matrix spikes

10.2.1 Prepare and analyze matrix spike and matrix spike duplicate samples to determine the accuracy of the extraction.

10.2.2 Prepare each spike using a sample chosen by the analyst, usually a control liver received with each sample set.

10.2.3 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spikes may be included and may fall in the low-range of the initial calibration curve.

10.2.4 Prepare one matrix spike and matrix spike duplicate per 40 samples, with a minimum of 2 matrix spikes per batch.

10.3 Continuing calibration verifications

10.3.1 Prepare continuing calibration verification samples to ensure the accuracy of the initial calibration curve.

10.3.2 Prepare, at a minimum, one continuing calibration verification sample per group of 10 samples. For example, if a sample set = 34, four verifications are prepared and extracted.

10.3.3 Prepare each continuing calibration verification from the same matrix used to prepare the initial curve.

10.3.4 The expected concentrations will fall within the mid-range of the initial calibration curve. Additional spikes may be included that fall in the low-range of the initial calibration curve. This is necessary if the analyst must quantitate using only the low end of the calibration curve (for example, 5 ppb – 100 ppb, rather than 5 ppb – 1000 ppb).

11.0 CALIBRATION AND STANDARDIZATION

11.1 Prepare matrix calibration standards

11.1.1 Weigh approximately 40 g of liver into a 250 mL Nalgene bottle containing 200 mLs Milli-Q™ water. Grind to a homogeneous solution.

11.1.2 If 40 g is not available, use appropriate amounts of liver and water to ensure a 1:5 ratio.

11.1.3 Refer to **13.0** to calculate the actual density of liver homogenate and the concentration of solid liver tissue dispersed in 1.0 mL of homogenate solution.

11.1.5 Add 1 mL of homogenate to a 15 mL centrifuge tube. Re-suspend solution by shaking between aliquots while preparing a total of eighteen 1 mL aliquots of homogeneous solution in 15 mL centrifuge tubes.

11.1.6 Two 1 mL aliquots, or other appropriate volume, serve as matrix blanks.

11.1.7 Typically use the standard concentrations and spiking amounts listed in Table 1, at the end of this section, to spike, in duplicate, two standard curves, for a total of eighteen samples, two matrix blanks, and two method blanks.

11.1.8 Refer to validation reports **ETS-8-6.0 and ETS-8-7.0-V-1** or **Attachment B,** which lists the working ranges and the Linear Calibration Range (LCR) for calibration curves.

11.1.9 Use **Attachment C** as an aid in calculating the concentrations of the working standards. Refer to **13.0** to calculate actual concentrations of PFOS in calibration standards.

11.2 To each working standard, blank, or continuing verification, add appropriate amount of surrogate working standard for the concentration to fall within the calibration curve range 5 ppb – 1000ppb.

11.3 Extract spiked liver homogenates following **12.14-12.25** of this method. Use these standards to establish each initial curve on the mass spectrometer.

Table 1
Approximate Spiking Amounts for Calibration Standards

Working Standard (Approx. Conc.)	µl	Approx. final conc. of PFOS in liver
-	-	Blank
0.50 ppm	2	0.005 ppm
0.50 ppm	4	0.010 ppm
0.50 ppm	10	0.025 ppm
0.50 ppm	20	0.050 ppm
0.50 ppm	40	0.100 ppm
5.0 ppm	10	0.250 ppm
5.0 ppm	20	0.500 ppm
5.0 ppm	30	0.750 ppm
50 ppm	4	1.00 ppm

12.0 PROCEDURE

12.1 Obtain frozen liver samples.

12.2 Cut approximately 1 g of liver using a dissecting scalpel. This part of the procedure is best performed quickly, not allowing the liver to thaw.

12.3 Weigh the sample directly into a tared plastic sampule vial.

12.4 Record the liver weight in the study notebook.

12.5 Return unused liver portions to freezer.

12.6 Add 2.5 mLs of water to sampule vial.

12.7 Grind the sample. Put the grinder probe in the sample and grind for about 2 minutes, or until the sample is homogeneous.

12.8 Rinse the probe into the sample with 2.5 mLs water using a pipette.

12.9 Take the grinder apart and clean it with methanol after each sample. Refer to AMDT-EP-22.

12.10 Cap the sample and vortex for 15 seconds. Label the sampule vial with the study number, weight, liver ID, date and analyst initials.

12.11 Pipette 1.0 mL, or other appropriate volume, of homogenate into a 15 mL polypropylene centrifuge tube. Label the centrifuge tube with the identical information as the sampule vial. Refer to attached worksheet for documenting the remaining steps.

12.12 Pipette two 1 mL aliquots of Milli-Q™ water to centrifuge tubes. These will serve as method blanks.

12.13 Spike all samples, including blanks and standards ready for extraction with surrogate standard as described in section **11.2**.

12.14 Spike each matirx with the appropriate amount of standard as described in 11.1, or Table 1 of that section, for the calibration curve standards. Also prepare matrix spikes and continuing calibration standards.

12.15 Vortex mix the standard curve samples, matrix spike samples, and continuing calibration samples for 15 seconds.

12.16 Check to ensure 0.5 M TBA reagent is at pH 10. If not, adjust accordingly.

12.17 To each sample, add 1 mL 0.5 M TBA and 2 mL of the 0.25 M sodium carbonate/sodium bicarbonate buffer.

12.18 Using an Oxford Dispenser, add 5 mL methyl-*tert*-butyl ether.

12.19 Cap each sample and put on the shaker at a setting of 300 rpm, for 20 minutes.

12.20 Centrifuge for 20 to 25 minutes at a setting of 3500 rpm, or until layers are well separated.

12.21 Label a fresh 15 mL centrifuge tube with the same information as in **12.10**.

12.22 Remove 4.0 mL of the organic layer to the fresh 15 mL centrifuge tube.

12.23 Put each sample on the analytical nitrogen evaporator until dry, approximately 1 to 2 hours.

12.24 Add 1.0 mL to each centrifuge tube using a graduated pipette.

12.25 Vortex mix for 30 seconds.

12.26 Attach a 0.2 µm nylon mesh filter to a 3 cc syringe and transfer the sample to this syringe. Filter into a 1.5 mL glass autovial or low-volume autovial when necessary.

12.27 Label the autovial with the study number, animal number and gender, sample timepoint, matrix, final solvent, extraction date, and analyst(s) performing the extraction.

12.28 Cap and store extracts at room temperature or at approximately 4 °C until analysis.

12.29 Complete the extraction worksheet, attached to this document, and tape in study notebook or include in study binder, as appropriate.

13.0 Data Analysis and Calculations

13.1 Calculations:

13.1.1 Calculate the average density of the liver homogenate by recording each mass of ten separate 1.0 mL aliquots of homogenate.

$$\text{Average density (mg/mL)} = \frac{\text{Average mass (mg) of the aliquots}}{\text{1.0 mL aliquot}}$$

13.1.2 Calculate the amount of liver (mg) per 1.0 mL homogenate (or concentration of dispersed solid tissue per mL of homogenate suspension) using the following equation:

$$\frac{\text{g of Liver x Average density* of homogenate (mg/mL)}}{\text{(g of Liver + g of Water)}}$$

* refer to **13.1.1** for details.

13.1.3 Calculate actual concentrations of PFOS and other fluorochemicals in calibration standards using the following equation:

$$\frac{\text{µL of Standard x Concentration (µg /mL)}}{\text{mg Liver / 1 mL homogenate*}} = \text{Final Concentration (µg/g or mg/kg) of PFOS in Liver}$$

*refer to **13.1.2** for details.

14.0 Method Performance

14.1 The method detection limit (MDL) is analyte and matrix specific. Refer to MDL report for specific MDL and limit of quantitation (LOQ) values (refer to **Attachments B and C**).

14.2 The following quality control samples are extracted with each batch of samples to evaluate the quality of the extraction and analysis.

14.2.1 Method blanks and matrix blanks.

14.2.2 Matrix spike and matrix spike duplicate samples to determine accuracy and precision of the extraction.

14.2.3 Continuing calibration verification samples to determine the continued accuracy of the initial calibration curve.

14.3 Refer to section **14** of **ETS-8-7.0** for method performance criteria.

15.0 Pollution Prevention and Waste Management

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

16.0 RECORDS

16.1 Complete the extraction worksheet attached to this method, and tape in the study notebook or include in the 3-ring study binder, as appropriate.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 Attachment A, Extraction worksheet

17.2 Attachment B, MDL/LOQ values and summary

17.3 Attachment C, Calibration standard calculation and concentration worksheet

18.0 REFERENCES

18.1 The validation report associated with this method is **ETS-8-6.0 & 7.0-V-1.**

18.2 AMDT-EP-22, "Routine Maintenance of Ultra-Turrax T-25"

18.3 FACT-M-1.1, "Extraction of PFOS or Other Anionic Fluorochemical Surfactants from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry"

19.0 AFFECTED DOCUMENTS

19.1 ETS-8-7.0, "Analysis of Liver Extracts for Fluorochemicals using HPLC-Electrospray Mass Spectrometry"

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

Study #_____ Matrix_____ Box #_____ Wk/Day_____ Date Spiked/Analyst	Surrogate Std approx. ppm actual ppm #_____	FC Mix Std approx. 0.5 ppm actual ppm #_____	FC Mix Std approx. 5 ppm actual ppm #_____	FC Mix Std approx. 50 ppm actual ppm #_____	Comments
CCV					
MS					
MSD					
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	
		-	-	-	

Blank Liver Homogenate: Std # Liver amount = g

Liver Extraction Method :	**Date & Initials**
Spike surrogate and Standard mix, Vortex 15 sec.	
Pipette 1 mL of Liver Solution	
Pipette 1 mL of †0.5 M TBA, pH 10. pH = _____	Std. #_____
Pipette 2 mL of 0.25 Na_2CO_3/0.25M $NaHCO_3$ Buffer	Std. #_____
Dispense 5ml of Methyl-t-Butyl Ether	TN-A-_____
Shake 20 min.	Shaker Speed
Centrifuge 20-25 min.	Centrifuge Speed
Remove a 4 mL aliquot of organic layer	
Put on Nitrogen Evaporator to dryness	Evaporator Temperature
Add 1.0 mL of Methanol	TN-A-_____
Vortex 30 sec.	
Filter using a 3cc B-D syringe with a 0.2µm SRI filter into autosample vial	

Cont. Cal. Verifications used the same matrix as for the standard curve.

MDL/LOQ values for rabbit liver

Compound	MDL (ppb)	LOQ (ppb)	Linear Calibration Range (LCR) Approximate concentrations to be used for preparing the Standard Calibration Curve
PFOS	8.45	26.9	30 ppb – 1200 ppb
PFOSA	3.50	11.1	12 ppb – 1200 ppb
PFOSAA	24.6	78.3	30 ppb – 1200 ppb
EtFOSE-OH	108	345	60 ppb – 900 ppb*
M556	82.3	262	60 ppb – 1200 ppb
PFOSEA	33.9	108	30 ppb- 1200 ppb

MDL/LOQ values in rat, bovine, and monkey liver were not statistically determined. Two curves in each of these matrices were extracted and analyzed with the rabbit liver curves to determine equivalence. Responses in the rat, bovine, and monkey liver curves were equivalent to the rabbit responses, therefore, their MDL and LOQ will be assumed to be equivalent to those values as determined for the rabbit liver.

Refer to LOQ Summary and MDL study in ETS-8-6.0 & 7.0-V-1 for further information
* EtFOSE-OH estimates only for MDL and LOQ. Did not meet criteria for validation.

Compound: PFOS

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.19 - 1237	12 - 1200	12 - 1200	6 - 300	12 - 300	60 - 1200	60 - 1200

Compound: PFOSA

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.19 - 1237	12 - 1200	12 - 1200	12 - 300	12 - 300	60 - 1200	60 - 1200

Compound: PFOSAA

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.16 - 1232	12 - 1200	30 - 1200	30 - 900	60 - 900	N/A	N/A

Compound: EtFOSE-OH

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.17 - 1235	31 - 900	31 - 900	N/A	N/A	N/A	N/A

Compound: PFOSEA

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.17 - 1235	31 - 1200	31 - 1200	N/A	N/A	N/A	N/A

Compound: M556

Liver matrix	Prepared range of standards (ppb) (ng/mL)	Range of average curve (ppb) (ng/mL)	LCR from ave curve (ppb) (ng/mL)	Range of low std curve (ppb) (ng/mL)	LCR from low std curve (ppb) (ng/mL)	Range of high std curve (ppb) (ng/mL)	LCR from high std curve (ppb) (ng/mL)
Rabbit	6.17 - 1235	31 - 1200	60 - 1200	N/A	N/A	N/A	N/A

Ion Pair Standard Curves – Tissue

Prep date(s):
Analyte(s):
Sample matrix:

Standard number:
Equipment number:
Final solvent and TN:
Blank liver/identifier:

Method/revision:
Target analyte(s):
FC mix std approx. 0.500 ppm:
FC mix std approx. 5.00 ppm:
FC mix std approx. 50.0 ppm:
Surrogate std approx. 100 ppm:

Actual concentrations of standards in the FC mix

PFOS Std conc ug/mL	PFOSA Std conc ug/mL	PFOSAA Std conc ug/mL	EtFOSE Std conc ug/mL	PFOSEA Std conc ug/mL	M556 Std conc ug/mL	Std conc ug/mL	All Am't spiked mL	All Density g
0.500	0.500	0.500	0.500	0.500	0.500		0.002	0.167
0.500	0.500	0.500	0.500	0.500	0.500		0.004	0.167
0.500	0.500	0.500	0.500	0.500	0.500		0.010	0.167
0.500	0.500	0.500	0.500	0.500	0.500		0.020	0.167
0.500	0.500	0.500	0.500	0.500	0.500		0.040	0.167
5.00	5.00	5.00	5.00	5.00	5.00		0.010	0.167
5.00	5.00	5.00	5.00	5.00	5.00		0.020	0.167
5.00	5.00	5.00	5.00	5.00	5.00		0.030	0.167
50.0	50.0	50.0	50.0	50.0	50.0		0.004	0.167

Calculated concentrations of standards in the sample matrix

PFOS Final conc ng/g	PFOSA Final conc ng/g	PFOSAA Final conc ng/g	EtFOSE Final conc ng/g	PFOSEA Final conc ng/g	M556 Final conc ng/g	Std conc ng/g	Surrogate Std conc ng/mL	All Am't spiked mL
5.99	5.99	5.99	5.99	5.99	5.99		100	0.005
12.0	12.0	12.0	12.0	12.0	12.0			
29.9	29.9	29.9	29.9	29.9	29.9		**Surrogate**	
59.9	59.9	59.9	59.9	59.9	59.9		Final conc	
120	120	120	120	120	120		ng/mL	
299	299	299	299	299	299		0.500	
599	599	599	599	599	599			
898	898	898	898	898	898			
1198	1198	1198	1198	1198	1198			

Validated ranges – approximate concentrations

Liver	PFOS	PFOSA	PFOSAA	EtFOSE-OH	POAA	PFOSEA
Rabbit	5-1000 ppb	5-1000 ppb	5-1000 ppb	5-1000 ppb	5-1000 ppb	5-1000 ppb
Bovine	Estimates only, use rabbit values.					
Rat	Estimates only, use rabbit values.					
Monkey	Estimates only, use rabbit values.					

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF FLUOROCHEMICALS IN LIVER EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: FACT-M-2.0 **Adoption Date:** 5/26/98

Revision Date: N/A

Author: Lisa Clemen

Approved By:

Laboratory Manager 5/26/98
Date

Group Leader 5/26/98
Date

Technical Reviewer 5/27/98
Date

Initial Date

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the analysis of extracts of liver or other tissues for fluorochemical surfactants using HPLC-electrospray/mass spectrometry.

1.2 Applicable Compounds: Potassium perfluorooctanesulfonate, anionic fluorochemical surfactants, or other ionizable compounds.

1.3 Matrices: Rabbit, rat, bovine, and monkey livers or other livers as designated in the validation report.

2.0 SUMMARY OF METHOD

2.1 This method describes the analysis of fluorochemical surfactants extracted from liver using HPLC-electrospray/mass spectrometry. The analysis is performed by monitoring a single ion characteristic of a particular fluorochemical, such as the potassium perfluorooctanesulfonate (PFOS) anion, M/Z= 499. Samples may also be screened to verify compound identification.

3.0 DEFINITIONS

3.1 None.

4.0 WARNINGS AND CAUTIONS

4.1 **Health and Safety Warnings:**

4.1.1 Use caution with the voltage cable for the probe. When the voltage cable is plugged into the probe DO NOT TOUCH THE PROBE, there is risk of electrical shock.

4.2 **Cautions:**

4.2.1 Do not run solvent pumps above capacity of 400 bar (5800 psi). If pressure goes over 400 bar, the HP1100 will initiate automatic shutdown.

4.2.2 Do not run solvent pumps to dryness.

5.0 INTERFERENCES

5.1 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 changed in order to optimize the system.

6.1.1 Micromass Electrospray Mass Spectrometer

6.1.2 HP1100 low pulse solvent pumping system and autosampler.

7.0 SUPPLIES AND MATERIALS

7.1 **Supplies**

7.1.1 Nitrogen gas, refrigerated liquid, regulated to approximately 100 psi.

7.1.2 HPLC column, specifics to be determined by the analyst.

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, all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus system.

8.1.3 Ammonium acetate, HPLC grade or equivalent.

8.2 Standards

8.2.1 Typically one H_2O blank, one liver blank, and seven liver standards are prepared during the extraction procedure. See FACT-M-1.

9.0 Sample Handling

9.1 Fresh liver standards are prepared with each analysis. Extracted standards and samples are stored in capped autovials or capped 15 mL centrifuge tubes until analysis.

9.2 If analysis will be delayed, extracted standards and samples may be refrigerated until analysis can be performed.

10.0 Quality Control

10.1 Matrix Blanks and Method Blanks

10.1.1 Analyze a method blank and matrix blank prior to each calibration curve.

10.2 Matrix Spikes

10.2.1 Analyze a matrix spike and matrix spike duplicate with each analysis.

10.2.2 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spike concentrations may fall in the low-range of the initial calibration curve.

10.2.3 See section 13 to calculate percent recovery.

10.3 Continuing Calibration Checks

10.3.1 Analyze a mid-range calibration standard after every tenth sample. If a significant change (± 30%) in peak area occurs, relative to the initial standard curve, stop the run. Only those samples analyzed before the last acceptable calibration standard will be used. The remaining samples must be reanalyzed.

10.3.2 See section 13 to calculate percent difference.

10.4 System Suitability

10.4.1 System suitability (e.g. peak area, retention time and peak shape, etc.) will be assessed for each run.

11.0 Calibration and Standardization

11.1 Analyze the extracted liver standards prior to and following each set of extracts. The mean of two standard values, at each standard concentration, will be plotted by linear regression for the calibration curve using MassLynx or other suitable software.

11.2 The r^2 value for the data should be 0.98 or greater. Lower values may be acceptable at the discretion of the analyst.

11.3 If the curve does not meet requirements, perform routine maintenance or reextract the standard curve (if necessary) and reanalyze.

12.0 PROCEDURES

12.1 Acquisition Set up

12.1.1 Click on start button in the Acquisition Control Panel. Set up a sample list. Assign a filename using letter-MO-DAY-last digit of year-sample number, assign a method (MS) for acquiring, and type in sample descriptions.

12.1.2 To create a method click on scan button in the Acquisition control panel and select SIR. Set Ionization Mode as appropriate and mass to 499 or other appropriate masses. A scan is usually collected along with the SIRs. Save method.

12.1.3 Typically the sample list begins with the first set of liver standards and ends with the second set of standards.

12.1.4 Samples are analyzed with a continuing calibration check injected after every tenth sample. Solvent blanks should be analyzed periodically to monitor possible analyte carryover and are not considered samples but may be included as such.

12.2 Using the Autosampler

12.2.1 Set up sample tray according to the sample list prepared in section 12.1.1.

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

12.2.2.1 Sample size = 10 µL injection with a sample wash

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 15 minutes

12.2.2.4 Solvent ramp =

Time	MeOH	2.0 mM Ammonium acetate
0.00 min.	45%	55%
7.5 min.	90%	10%
11.0 min.	90%	10%
11.5 min.	45%	55%

Note: In this instrument configuration, the run must be set up on the electrospray software with a "Waiting for inlet start" message before the "Start" button is pressed on the HP Workstation.

12.2.2.5 Press the "Start" button.

12.3 Instrument Sep-up

12.3.1 Refer to AMDT-EP-31 for more details.

12.3.2 Check the solvent level in reservoirs and refill if necessary.

12.3.3 Check the stainless steel capillary at the end of the probe. Use an eye piece to check the tip. 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.

12.3.4 Set HPLC pump to "On". Set the flow to 10 - 500 uL/min or as appropriate. Observe droplets coming out of the tip of the probe. Allow to equilibrate for approximately 10 minutes.

12.3.5 Turn on the nitrogen. A fine mist should be expelled with no nitrogen leaking around the tip of the probe.

12.3.6 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:

12.3.6.1 Drying gas 250-400 liters/hour

12.3.6.2 ESI nebulizing gas 10-15 liters/hour

12.3.6.3 LC constant flow mode flow rate 10 – 500 uL/min

12.3.6.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the instrument is operating correctly.)

12.3.7 Carefully guide the probe into the opening. Insert probe until it will not go any further. Connect the voltage cables to the probe.

12.3.8 Record tune parameters in the instrument log.

12.3.9 Using the cross-flow counter electrode in the ES/MS source is recommended for the analysis of biological matrices.

12.3.10 Click on start button in the Acquisition Control Panel. Press the start button at top of sample list. Ensure start and end sample number includes 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 PFOS anion in total liver (mg):

$$\frac{\left(\dfrac{\text{ug PFOS anion calc. from std curve}}{\text{g of liver used for analysis}}\right)}{1000\ \text{ug}/1\ \text{mg}} \times \text{Total mass of liver (g)}$$

14.0 METHOD PERFORMANCE

14.1 The method detection limit is equal to at least three times the baseline noise in the matrix blank.

14.2 The practical quantitation limit is equal to the lowest standard in the calibration curve.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

16.0 RECORDS

16.1 Store chromatograms in the study folder. Each chromatogram should have the following information included either in the header or hand written on the chromatogram: study number, sample name, extraction date, and dilution factor (if applicable).

16.2 Plot calibration curve by linear regression and store in the study folder.

16.3 Print sample list from MassLynx and tape into the instrument runlog.

16.4 Print data integration summary from MassLynx and tape into the instrument runlog.

16.5 Copy instrument runlog pages, including instrument parameters and sample results, and tape into appropriate study notebook.

16.6 Summarize data using suitable software and store in the study folder.

16.7 Back up electronic data to appropriate media. Record in study notebook the file name and location of backup electronic data.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 Attachment A: FACT-M-2 Data reporting spreadsheet

17.2 The validation report associated with this method is FACT-M-1.0 & 2.0-V-1.

18.0 REFERENCES

18.1 AMDT-EP-31, "Operation of VG Platform Electrospray Mass Spectrometer"

19.0 AFFECTED DOCUMENTS

19.1 FACT-M-1.0, "Extraction of Potassium Perfluorooctanesulfonate from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry"

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

Laboratory Study

Study:
Test Material:
Matrix/Final Solvent:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y Intercept:
Date of Extraction/Analyst:
Date of Analysis/Analyst:

Group Dose	Sample#	Concentration ug/mL	Initial Vol. mL	Dilution Factor	Final Conc. ug/mL

Slope: Taken from linear regression equation.
Group/Dose: Taken from the study folder.
Sample#: Taken from the study folder.
Concentration (ug/mL): Taken from the MassLynx integration summary.
Initial Volume (mL): Taken from the study folder.
Dilution Factor: Taken from the study folder.
Final Conc. (ug/mL): Calculated by dividing the initial volume from the concentration

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF FLUOROCHEMICALS IN SERUM EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: FACT-M-4.0 **Adoption Date:** 4/22/98

Revision Date: N/A

Author: Lisa Clemen

Approved By:

Laboratory Manager 4/22/98 Date

Group Leader 4/22/98 Date

Technical Reviewer 4/14/98 Date

Initial RSD

Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the analysis of extracts of serum or tissue for fluorochemical surfactants using HPLC-electrospray/mass spectrometry.

1.2 Applicable Compounds: Potassium perfluorooctanesulfonate, anionic fluorochemical surfactants, or other ionizable compounds.

1.3 Matrices: Rabbit, rat, and bovine serum or other sera as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the analysis of fluorochemical surfactants extracted from serum using HPLC-electrospray/mass spectrometry. The analysis is performed by monitoring a single ion characteristic of a particular fluorochemical, such as the potassium perfluorooctanesulfonate (PFOS) anion, M/Z= 499. Samples may also be screened to verify compound identification.

3.0 Definitions

3.1 None.

4.0 Warnings and Cautions

4.1 Health and Safety Warnings:

4.1.1 Use caution with the voltage cable for the probe. When the voltage cable is plugged into the probe DO NOT TOUCH THE PROBE, there is risk of electrical shock.

4.2 Cautions:

4.2.1 Do not run solvent pumps above capacity of 400 bar (5800 psi). If pressure goes over 400 bar, the HP1100 will initiate automatic shutdown.

4.2.2 Do not run solvent pumps to dryness.

5.0 Interferences

5.1 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 changed in order to optimize the system.

6.1.1 Micromass Electrospray Mass Spectrometer

6.1.2 HP1100 low pulse solvent pumping system and autosampler.

7.0 Supplies and Materials

7.1 Supplies

7.1.1 Nitrogen gas, refrigerated liquid, regulated to approximately 100 psi.

7.1.2 HPLC column, specifics to be determined by the analyst.

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, all water used in this method should be Milli-Q™ water and may be provided by a Milli-Q TOC Plus system.

8.1.3 Ammonium acetate, HPLC grade or equivalent.

8.2 Standards

8.2.1 Typically one H_2O blank, one serum blank, and seven serum standards are prepared during the extraction procedure. See FACT-M-3.

9.0 SAMPLE HANDLING

9.1 Fresh serum standards are prepared with each analysis. Extracted standards and samples are stored in capped autovials or capped 15 mL centrifuge tubes until analysis.

9.2 If analysis will be delayed, extracted standards and samples may be refrigerated at 4° C until analysis can be performed.

10.0 QUALITY CONTROL

10.1 Matrix Blanks and Method Blanks

10.1.1 Analyze a method blank and a matrix blank prior to each calibration curve.

10.2 Matrix Spikes

10.2.1 Analyze a matrix spike and matrix spike duplicate with each analysis.

10.2.2 Expected concentrations will fall in the mid-range of the initial calibration curve. Additional spike concentrations may fall in the low-range of the initial calibration curve.

10.2.3 See section 13 to calculate percent recovery.

10.3 Continuing Calibration Checks

10.3.1 Analyze a mid-range calibration standard after every tenth sample. If a significant change (± 30%) in peak area occurs, relative to the initial standard curve, stop the run. Only those samples analyzed before the last acceptable calibration standard will be used. The remaining samples must be reanalyzed.

10.3.2 See section 13 to calculate percent difference.

10.4 System Suitability

10.4.1 System suitability (e.g., peak area, retention time, peak shape, etc.) will be assessed for each run.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Analyze the extracted serum standards prior to and following each set of extracts. The mean of two standard values, at each standard concentration, will be plotted by linear regression for the calibration curve using MassLynx or other suitable software.

11.2 The r^2 value for the data should be 0.98 or greater. Lower values may be acceptable at the discretion of the analyst.

11.3 If the curve does not meet requirements, perform routine maintenance or reextract the standard curve (if necessary) and reanalyze.

12.0 PROCEDURES

12.1 Acquisition Set up

12.1.1 Click on start button in the Acquisition Control Panel. Set up a sample list. Assign a filename using letter-MO-DAY-last digit of year-sample number, assign a method (MS) for acquiring, and type in sample descriptions.

12.1.2 To create a method click on scan button in the Acquisition control panel and select SIR (Single Ion Recording). Set Ionization Mode as appropriate and mass to 499 or other appropriate masses. A scan is usually collected along with the SIRs. Save method.

12.1.3 Typically the sample list begins with the first set of serum standards and ends with the second set of standards.

12.1.4 Samples are analyzed with a continuing calibration check injected after every tenth sample. Solvent blanks should be analyzed periodically to monitor possible analyte carryover and are not considered samples but may be included as such.

12.2 Using the Autosampler

12.2.1 Set up sample tray according to the sample list prepared in section 12.1.1.

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

12.2.2.1 Sample size = 10 µL injection with a sample wash

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 15 minutes

12.2.2.4 Solvent ramp =

Time	MeOH	2.0 mM Ammonium acetate
0.00 min.	45%	55%
7.5 min.	90%	10%
11.0 min.	90%	10%
11.5 min.	45%	55%

Note: In this instrument configuration, the run must be set up on the electrospray software with a "Waiting for inlet start" message before the "Start" button is pressed on the HP Workstation.

12.2.2.5 Press the "Start" button.

12.3 Instrument Set-up

12.3.1 Refer to AMDT-EP-31 for more details.

12.3.2 Check the solvent level in reservoirs and refill if necessary.

12.3.3 Check the stainless steel capillary at the end of the probe. Use an eye piece to check the tip. 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.

12.3.4 Set HPLC pump to "On". Set the flow to 10 - 500 uL/min or as appropriate. Observe droplets coming out of the tip of the probe. Allow to equilibrate for approximately 10 minutes.

12.3.5 Turn on the nitrogen. A fine mist should be expelled with no nitrogen leaking around the tip of the probe.

12.3.6 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:

12.3.6.1 Drying gas 250-400 liters/hour

12.3.6.2 ESI nebulizing gas 10-15 liters/hour

12.3.6.3 HPLC constant flow mode flow rate 10 – 500 µL/min

12.3.6.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the HPLC is operating correctly.)

12.3.7 Carefully guide the probe into the opening. Insert probe until it will not go any further. Connect the voltage cables to the probe.

12.3.8 Record tune parameters in the instrument log.

12.3.9 Using the cross-flow counter electrode in the ES/MS source is recommended for the analysis of biological matrices.

12.3.10 Click on start button in the Acquisition Control Panel. Press the start button at top of sample list. Ensure start and end sample number includes all samples to be analyzed.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.4 Calculate matrix spike percent recoveries using the following equation:

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

13.1.5 Calculate percent difference using the following equation:

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

13.1.6 Calculate actual concentration of PFOS, or other fluorochemical, anion in serum (µg/mL):

$$\frac{\text{µg of PFO calc. from std. Curve}}{\text{Initial Volume of serum (mL)}} \times \text{Dilution Factor} \times \text{Final Volume (mL)}$$

14.0 METHOD PERFORMANCE

14.1 The method detection limit is equal to half the lowest standard in the calibration curve.

14.2 The practical quantitation limit is equal to the lowest standard in the calibration curve.

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 Store chromatograms in the study folder. Each chromatogram must have the following information included either in the header or hand written on the chromatogram: study number, sample name, extraction date, and dilution factor (if applicable).

16.2 Plot calibration curve by linear regression and store in the study folder.

16.3 Print sample list from MassLynx and tape into the instrument runlog.

16.4 Print data integration summary from MassLynx and tape into the instrument runlog.

16.5 Copy instrument runlog pages, including instrument parameters and sample results, and tape into appropriate study notebook.

16.6 Summarize data using suitable software and store in the study folder.

16.7 Back up electronic data to appropriate medium. Record in study notebook the file name and location of backup electronic data.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 Attachment A: FACT-M-4 Data reporting spreadsheet

17.2 The validation report associated with this method is FACT-M-3.0 & 4.0-V-1.

18.0 REFERENCES

18.1 AMDT-EP-31, "Operation of VG Platform Electrospray Mass Spectrometer"

19.0 AFFECTED DOCUMENTS

19.1 FACT-M-3.0, "Extraction of Fluorochemical Anions from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry"

20.0 REVISIONS

Revision Number.	Reason For Revision	Revision Date

Laboratory Study

Study:
Test Material:
Matrix/Final Solvent:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y Intercept:
Date of Extraction/Analyst:
Date of Analysis/Analyst:

Group Dose	Sample#	Concentration ug/mL	Initial Vol. mL	Dilution Factor	Final Conc. ug/mL

Slope: Taken from linear regression equation.
Group/Dose: Taken from the study folder.
Sample#: Taken from the study folder.
Concentration (ug/mL): Taken from the MassLynx integration summary.
Initial Volume (mL): Taken from the study folder.
Dilution Factor: Taken from the study folder.
Final Conc. (ug/mL): Calculated by dividing the initial volume from the concentration

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICALS IN SERUM EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-5.1 **Adoption Date:** 03/01/99

Revision Date: 4/26/99

Author: Lisa Clemen, Robert Wynne

Approved By:

Laboratory Manager 4/26/

Group Leader 4/26/99

Lisa A Clemen 04/26/99
Technical Reviewer Date

Exact Copy of Original
Initial RSD
Date 6/5/01

1.0 SCOPE AND APPLICATION

1.1 Scope: This method describes the analysis of serum extracts for fluorochemical surfactants using HPLC-electrospray/mass spectrometry.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds, or other ionizable compounds.

1.3 Matrices: Rabbit, rat, bovine, monkey, and human serum, or other fluids as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the analysis of fluorochemical surfactants extracted from serum or other fluids, using HPLC-electrospray/mass spectrometry, or similar system as appropriate. The analysis is performed by monitoring a single ion characteristic of a particular fluorochemical, such as the perfluorooctanesulfonate (PFOS) anion, m/z= 499. Additionally, samples may be analyzed using a tandem mass spectrometer to further verify the identity of a compound by detecting daughter ions of the parent ion.

3.0 Definitions

3.1 **Atmospheric Pressure Ionization (API):** The Micromass Quattro II triple quadrupole systems allow for various methods of ionization by utilizing various sources, probes, and interfaces. These include but are not limited to: Electrospray Ionization (ESI), Atmospheric Pressure chemical Ionization (APcI), Thermospray, etc. The ionization process in these techniques 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 charged droplets are produced by the application of a strong electrical field.

3.3 **Mass Spectrometry, Mass Spectrometer (MS), Tandem Mass Spectrometer (MS/MS):** The API Quattro II triple quadrupole systems are equipped with quadrupole mass selective detectors. Ions are selectively discriminated by mass to charge ratio (m/z) and subsequently detected. A single MS may be employed for ion detection or a series (MS/MS) for more specific fragmentation information.

3.4 **Conventional vs. Z-spray probe interface:** The latest models of Micromass Quattro II triple quadrupole systems (post 1998) utilize a "Z-spray" conformation. The spray emitted from a 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.)

3.5 **Mass Lynx Software:** System software designed for the specific operation of these Quattro II triple quadrupole systems. Currently MassLynx has Windows 95 and WindowsNT 4.0 versions. All versions are similar. For more details see the manual specific to the instrument (Micromass Quattro II triple quadrupole MassLynx or MassLynx NT 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 gloves, eyewear, and clothing.

4.2 Cautions:

4.2.1 Do not operate solvent pumps above capacity of 400 bar (5800 psi) back pressure. If the back pressure 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 Quattro II triple quadrupole 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 (House air system)

7.1.2 HPLC analytical column, specifics to be determined by the analyst and documented in the raw data.

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, 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

8.2 Standards

8.2.1 Typically two method blanks, two matrix blanks, and eighteen matrix standards are prepared during the extraction procedure. See **ETS-8-4.1**.

9.0 SAMPLE HANDLING

9.1 Fresh matrix standards are prepared with each analysis. Extracted standards and samples are stored in capped autovials or capped 15 mL centrifuge tubes until analysis.

9.2 If analysis will be delayed, extracted standards and samples can be refrigerated at approximately 4° C, or at room temperature, until analysis 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 batch 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 and analyzed to determine the matrix effect on the recovery efficiency.

10.2.2 Matrix spike duplicates are prepared and analyzed to measure the precision and the recovery for each analyte.

10.2.3 Analyze a matrix spike and matrix spike duplicate per forty samples, with a minimum of 2 spikes per batch.

10.2.4 Matrix spike and matrix spike duplicate concentrations will fall in the mid-range of the initial calibration curve. Additional spike concentrations may fall in the low-range of the initial calibration curve.

10.3 Continuing Calibration Verifications

10.3.1 Continuing calibration verifications 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 batch.

11.0 CALIBRATION AND STANDARDIZATION

11.1 Analyze the extracted matrix standards prior to and following each set of extracts. The average of two standard curves will be plotted by linear regression ($y = my + b$), weighted $1/x$, not forced through zero, using MassLynx or other suitable software.

11.2 If the curve does not meet requirements, perform routine maintenance or reextract the 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 of the standard curve. 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 Click on start button in the Acquisition Control Panel. Set up a sample list. Assign a filename using MO-DAY-last digit of year-sample number, assign a method (MS) for acquiring, and type in sample descriptions.

12.1.2 To create a method click on scan button in the Acquisition control panel and select SIR (Single Ion Recording) or MRM. Set Ionization Mode as appropriate and mass to 499 or other appropriate masses. A full scan is usually collected along with the SIRs. Save acquisition method. If MS/MS instruments are employed, additional product ion fragmentation information may be collected. See Micromass MassLynx GUIDE TO DATA ACQUISITION for additional information and MRM (Multiple Reaction Monitoring).

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

12.1.4 Samples are analyzed with a continuing calibration check injected after every tenth sample. Solvent blanks should be analyzed periodically to monitor possible analyte carryover and are not considered samples but may be included as such.

12.2 Using the Autosampler

12.2.1 Set up sample tray according to the sample list prepared in Section **12.1.1**.

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

12.2.2.1 Sample size = 10 µL injection

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 13.5 minutes

12.2.2.4 Solvent ramp =

Time	MeOH	2.0 mM Ammonium acetate
0.00 min.	40%	60%
8.50 min.	90%	10%
11.0 min.	90%	10%
12.0 min.	40%	60%

12.2.2.5 Press the "Start" button.

12.3 Instrument Set-up

12.3.1 Refer to **ETS-9-24.0** for more details.

12.3.2 Check the solvent level in reservoirs and refill if necessary.

12.3.3 Check the stainless steel capillary at the end of the probe. Use an eyepiece to check the tip. 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.

12.3.4 Set HPLC pump to "On". Set the flow to 10 - 500 uL/min or as appropriate. Observe droplets coming out of the tip of the probe. Allow to equilibrate for approximately 10 minutes.

12.3.5 Turn on the nitrogen. A fine mist should be expelled with no nitrogen leaking around the tip of the probe. Readjust the tip of the probe if no mist is observed.

12.3.6 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:

12.3.6.1 Drying gas 250-400 liters/hour

12.3.6.2 ESI nebulizing gas 10-15 liters/hour

12.3.6.3 HPLC constant flow mode, flow rate 10 – 500 µL/min

12.3.6.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the HPLC is operating correctly.)

12.3.7 Carefully guide the probe into the opening. Insert probe until it will not go any further. Connect the voltage cables to the probe.

12.3.8 Print the tune page, with its parameters, and store it in the study binder with a copy taped into the instrument log.

12.3.9 Using the cross-flow counter electrode in the ES/MS source is recommended for the analysis of biological matrices.

12.3.10 Click on start button in the Acquisition Control Panel (this may vary among MassLynx versions, see appropriate MassLynx USER'S GUIDE). Press the start button. Ensure start and end sample number includes all samples to be analyzed.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.4 Calculate matrix spike percent recoveries using the following equation:

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

13.1.5 Calculate percent difference using the following equation:

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

13.1.6 Calculate actual concentration of PFOS, or other fluorochemical, in matrix (µg/mL):

$$\frac{\text{(ng of PFOS calc. from std. Curve x Dilution Factor)}}{\text{(Initial Volume of matrix (mL) + mL of Surrogate Standard)}} \times \frac{1\ \mu g}{1000\ ng}$$

Final Volume (mL)

14.0 METHOD PERFORMANCE

14.1 Method Detection Limit (MDL) and Limit of Quantitation (LOQ) are method, analyte, and matrix specific. Please see **ETS-8-4.1, Attachment B,** for a listing of current validated MDL and LOQ values.

14.2 Solvent Blanks, Method Blanks, and Matrix Blanks

14.2.1 Solvent blanks, method blanks, and matrix blanks values are must be below the lowest standard in the calibration curve

14.3 Calibration Curves

14.3.1 The r^2 value for the calibration curve must be 0.980 or better.

14.4 Matrix Spikes

14.4.1 Matrix spike percent recoveries are must be within ± 30% of the spiked concentration.

14.5 Continuing Calibration Verifications

14.5.1 Continuing calibration verification percent recoveries must be ± 30% of the spiked concentration.

14.6 If criteria listed in this method performance section isn't met, maintenance may be performed on the system and samples reanalyzed or other actions as determined by the analyst. Document all actions in the appropriate logbook.

14.7 If data are 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 to include in the appropriate study folder. Copy these pages and tape into the instrument runlog.

16.3 Plot the calibration curve by linear regression, weighted 1/x, 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 (Excel 5.0) and store in the study folder, see **Attachment A** for an example of a summary spreadsheet.

16.6 Back up electronic data to appropriate medium. Record in study notebook the file name and location of backup electronic data.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 Attachment A: ETS-8-5.1 Data summary spreadsheet.

18.0 References

18.1 FACT-M-4.1, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry

18.2 ETS-9-24.0, "Operation and Maintenance of the Micromass Atmospheric Pressure Ionization/Mass Spectrometer Quattro II triple quadrupole Systems"

18.3 The validation report associated with this method is **ETS-8-4.0 & 5.0-V-1**.

19.0 Affected Documents

19.1 ETS-8-4.1, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Serum for Analysis Using HPLC-Electrospray/Mass Spectrometry"

20.0 Revisions

Revision Number.	Reason For Revision	Revision Date
1	Section 6.1.2 Clarification of HP1100 system components. Section 11.1 Average of two curves, not standard values, are used for plotting linear regression and added the 1/x weighting of the curve. Section 12.2.2.4 Clarification of solvent ramp. Section 17.1 Changed from attachment B to A.	04/02/99

Laboratory Study

Study:
Test Material:
Matrix/Final Solvent:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y Intercept:
Date of Extraction/Analyst:
Date of Analysis/Analyst:

Group Dose	Sample#	Concentration ug/mL	Initial Vol. mL	Dilution Factor	Final Conc. ug/mL

Slope: Taken from linear regression equation.
Group/Dose: Taken from the study folder.
Sample#: Taken from the study folder.
Concentration (ug/mL): Taken from the MassLynx integration summary.
Initial Volume (mL): Taken from the study folder.
Dilution Factor: Taken from the study folder.
Final Conc. (ug/mL): Calculated by dividing the initial volume from the concentration

3M ENVIRONMENTAL LABORATORY

METHOD

ANALYSIS OF POTASSIUM PERFLUOROOCTANESULFONATE OR OTHER FLUOROCHEMICALS IN LIVER EXTRACTS USING HPLC-ELECTROSPRAY/MASS SPECTROMETRY

Method Number: ETS-8-7.0 **Adoption Date:** 07/22/99

Revision Date: NA

Author: Lisa Clemen, Glenn Langenburg

Approved By:

Laboratory Manager 7/22/99 Date

Group Leader 7/14/99 Date

Technical Reviewer 07/14/99 Date

Initial RSD Date 6/5/01

Original

1.0 SCOPE AND APPLICATION

1.1 Scope: This method is for the analysis of liver extracts for fluorochemical surfactants using HPLC-electrospray/mass spectrometry.

1.2 Applicable Compounds: Fluorochemical surfactants or other fluorinated compounds, or other ionizable compounds.

1.3 Matrices: Rabbit, rat, bovine, monkey liver, or other tissues as designated in the validation report.

2.0 Summary of Method

2.1 This method describes the analysis of fluorochemical surfactants extracted from liver using HPLC-electrospray/mass spectrometry, or similar system as appropriate. The analysis is performed by monitoring a single ion characteristic of a particular fluorochemical, such as the perfluorooctanesulfonate (PFOS) anion, m/z = 499. Additionally, samples may be analyzed using a tandem mass spectrometer to further verify the identity of a compound by detecting daughter ions of the selected parent ion.

3.0 Definitions

3.1 **Atmospheric Pressure Ionization (API):** The Micromass Quattro II triple quadrupole systems allow for various methods of ionization by utilizing various sources, probes, and interfaces. These include but are not limited to: Electrospray Ionization (ESI), Atmospheric Pressure chemical Ionization (APcI), Thermospray, etc. The ionization process in these techniques 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 charged droplets are produced by the application of a strong electrical field.

3.3 **Mass Spectrometry, Mass Spectrometer (MS), Tandem Mass Spectrometer (MS/MS):** The API Quattro II triple quadrupole mass spectrometer is equipped with two quadrupole mass selective detectors and a collision cell. Ions are selectively discriminated by mass to charge ratio (m/z) and subsequently detected. A single MS may be employed for ion detection or an ion may be selected in the first quadrupole, fragmented in the collision cell, and these fragments may be analyzed in the second quadrupole.

3.4 **Conventional vs. Z-spray probe interface:** The latest models of Micromass Quattro II triple quadrupole (post 1998) utilize a "Z-spray" conformation. The spray emitted from a 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 other Z-spray systems, etc.)

3.5 **Mass Lynx Software:** System software designed for the specific operation of these Quattro II triple quadrupole systems. Currently MassLynx has Windows 95 and WindowsNT 4.0 versions. All versions are similar. For more details refer to the manual specific to the instrument (Micromass Quattro II triple quadrupole MassLynx or MassLynx NT 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 gloves, eyewear, and clothing.

4.2 Cautions:

4.2.1 Operate the solvent pumps below a back pressure of 400 bar (5800 psi). If the back pressure 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 shall 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 Quattro II triple quadrupole 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 air regulated to approximately 100 psi (house air system)

7.1.2 HPLC analytical column, specifics to be determined by the analyst and documented in the raw data

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 ATSM type I, or equivalent, and 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 mL volumetric container containing 2000 mL Milli-Q™ water, mix until all solids are dissolved. Store at room temperature.

8.2 Standards

8.2.1 Typically two method blanks, two matrix blanks, and eighteen matrix standards are prepared during the extraction procedure. Refer to **ETS-8-6.0**.

9.0 SAMPLE HANDLING

9.1 Fresh matrix standards are prepared with each analysis. Extracted standards and samples are stored in capped autovials or capped 15 ml centrifuge tubes until analysis.

9.2 If analysis will be delayed, extracted standards and samples may be stored at room temperature, or refrigerated at approximately 4° C, until analysis can be performed.

10.0 QUALITY CONTROL

10.1 Method Blanks and Matrix Blanks

10.1.1 Solvent blanks, method blanks, and matrix blanks are prepared and analyzed with each batch 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 and analyzed to determine the matrix effect on the recovery efficiency.

10.2.2 Matrix spike duplicates are prepared and analyzed to measure the precision and the recovery for each analyte.

10.2.3 Analyze a matrix spike and matrix spike duplicate per forty samples. With a minimum of 2 spikes per batch.

10.2.4 Matrix spike and matrix spike duplicate concentrations will fall in the mid-range of the initial calibration curve. Additional spike concentrations may fall in the low-range of the initial calibration curve.

10.3 Continuing Calibration Checks

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

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

11.0 CALIBRATION AND STANDARDIZATION

11.1 Analyze the extracted matrix standards prior to and following each set of sample extracts. The average of two standard curves will be plotted by linear regression ($y = mx + b$), weighted 1/x, not forced through the origin, using MassLynx or other suitable software.

11.2 If the curve does not meet requirements perform routine maintenance or reextract the 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 of the standard curve. 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 MO-DAY-last digit of year-increasing letter of the alphabet starting with a

12.1.1.2 Assign a method (MS file) for acquiring

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 (Single Ion Recording) or MRM (Multiple Reaction Monitoring). Set Ionization Mode as appropriate and mass to 499 or other appropriate masses. A full scan is usually collected along with the SIRs. Save acquisition method. If MS/MS instruments are employed, additional product ion fragmentation information may be collected. Refer to Micromass MassLynx GUIDE TO DATA ACQUISITION for additional information and MRM.

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

12.1.4 Samples are analyzed with a continuing calibration verification injected standard after every tenth sample. Solvent blanks should be analyzed periodically to monitor possible analyte carryover and are not considered samples but may be included as such.

12.2 Using the Autosampler

12.2.1 Set up sample tray according to the sample list prepared in Section **12.1.1**.

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

12.2.2.1 Sample size = 10 µL injection

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 9 minutes

12.2.2.4 Solvent ramp conditions

Time	MeOH	2.0 mM Ammonium acetate
0.00 min.	40%	60%
1.0 min.	40%	60%
4.5 min.	95%	5%
6.5 min.	95%	5%
7.0 min.	40%	60%
9.0 mi.	40%	60%

12.2.2.5 Press the "Start" button.

12.3 Instrument Set-up

12.3.1 Refer to **ETS-9-24.0**, "Operation and Maintenance of the Micromass Quattro II Triple Quadrupole Mass Spectrometer Fitted with an Atmospheric Pressure Ionization Source," for more details.

12.3.2 Check the solvent level in reservoirs and refill if necessary.

12.3.3 Check the stainless steel capillary at the end of the probe. Use an eyepiece to check the tip. 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.

12.3.4 Turn on the nitrogen.

12.3.5 Open the tune page. Clicks on operate to initiate source block and desolvation heaters.

12.3.6 Open the Inlet Editor.

12.3.6.1 Set HPLC pump to "On"

12.3.6.2 Set the flow to 10 - 500 uL/min or as appropriate

12.3.6.3 Observe droplets coming out of the tip of the probe. A fine mist should be expelled with no nitrogen leaking around the tip of the probe. Readjust the tip of the probe if no mist is observed

12.3.6.4 Allow to equilibrate for approximately 10 minutes.

12.3.7 The instrument uses these parameters at the following settings. These settings may change in order to optimize the response:

12.3.7.1 Drying gas 250-400 liters/hour

12.3.7.2 ESI nebulizing gas 10-15 liters/hour

12.3.7.3 HPLC constant flow mode flow rate 10 – 500 µL/min

12.3.7.4 Pressure <400 bar (This parameter is not set, it is a guide to ensure the HPLC is operating correctly.)

12.3.7.5 Source block temperature 150°

12.3.7.6 Desolvation temperature 250°

12.3.8 Print the tune page, with its parameters, and store it in the study binder with a copy taped into the instrument log.

12.3.9 Click on start button in the Acquisition Control Panel (this may vary among MassLynx versions, refer to appropriate MassLynx User's Guide). Ensure start and end sample number includes all samples to be analyzed.

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

13.1.4 Calculate matrix spike percent recoveries using the following equation:

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

13.1.5 Calculate percent difference using the following equation:

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

13.1.6 Calculate actual concentrations in matrix (µg/g):

$$\frac{\frac{\text{(ng of PFOS calc. from std. Curve x Dilution Factor)}}{\text{(Initial Weight of Liver (g)}}}{\text{Final Volume (mL)}} \times \frac{1\ \mu g}{1000\ ng}$$

14.0 METHOD PERFORMANCE

14.1 Method Detection Limit (MDL) and Limit of Quantitation (LOQ) are method, analyte, and matrix specific. Refer to **ETS-8-6.0, Attachment B** for a listing of current validated MDL and LOQ values.

14.2 Solvent Blanks, Method Blanks and Matrix Blanks

14.2.1 Solvent blanks, method blanks, and matrix blanks must be below the lowest standard in the calibration curve.

14.3 Calibration Curves

14.3.1 The r^2 value for the calibration must be 0.980 or better.

14.4 Matrix Spikes

14.4.1 Matrix spike percent recoveries must be within ± 30% of the spiked concentration.

14.5 Continuing Calibration Verification

14.5.1 Continuing calibration verification percent recoveries must be within ± 30% of the spiked concentration.

14.6 If criteria listed in the 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 appropriate logbook.

14.7 If data are 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 to include in the appropriate study folder. Copy these pages and tape into the instrument runlog.

16.3 Plot the calibration curve by linear regression, weighted 1/x, 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 (Excel 5.0+) and store in the study folder, refer to **Attachment A** for an example of a summary spreadsheet.

16.6 Back up electronic data to appropriate medium. Record in study notebook the file name and location of backup electronic data.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

17.1 Attachment A: ETS-8-7.0 Data summary spreadsheet

18.0 References

18.1 FACT-M-2.1, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Liver for Analysis Using HPLC-Electrospray/Mass Spectrometry"

18.2 ETS-9-24.0, "Operation and Maintenance of the Micromass Atmospheric Pressure Ionization/Mass Spectrometer Quattro II triple quadrupole Systems"

18.3 The validation report associated with this method is **ETS-8-6.0 & 7.0-V-1**

19.0 Affected Documents

19.1 ETS-8-6.0, "Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Liver or Fluid for Analysis Using HPLC-Electrospray/Mass Spectrometry"

20.0 REVISIONS

Revision Number	Reason For Revision	Revision Date

Laboratory Study

Study:
Test Material:
Matrix/Final Solvent:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y Intercept:
Date of Extraction/Analyst:
Date of Analysis/Analyst:

Group Dose	Sample#	Concentration ng/g	Initial Wt. g	Dilution Factor	Final Conc. ug/g

Slope: Taken from linear regression equation.
Group/Dose: Taken from the study folder.
Sample#: Taken from the study folder.
Concentration (ng/g): Taken from the MassLynx integration summary.
Initial Wt. (g): Taken from the study folder.
Dilution Factor: Taken from the study folder.
Final Conc. (ug/g): Calculated by dividing the initial volume from the concentration

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Appendix D: Data Summary Tables

Table 9. FACT TOX-003 Data Summary for PFOS, PFOSA, PFOSAA, EtFOSE, M556 and PFOSEA—Rat Serum (µg/mL)

Timept	Group		PFOS Avg ± SD	PFOSA Avg ± SD	PFOSAA Avg ± SD	EtFOSE** Avg ± SD	M556 Avg ± SD	PFOSEA** Avg ± SD
Week 4	Group 8 0.0 ppm	M	<LOQ[a] (n=5)	<LOQ[b] (n=5)	0.00460 ± 0.00158* (n=5)	NA	NA	NA
		F	<LOQ[a] (n=5)	<LOQ[b] (n=5)	0.0103 ± 0.00375* (n=5)	NA	NA	NA
	Group 9 1.0 ppm	M	0.174 ± 0.0510 (n=5)	0.00500 ± 0.000125 (n=5)	0.247 ± 0.195* (n=5)	NA	NA	NA
		F	0.270 ± 0.0687 (n=5)	0.00778 ± 0.00254 (n=5)	0.294 ± 0.111* (n=5)	NA	NA	NA
Week 14	Group 8 0.0 ppm	M	0.0393 ± 0.0109 (n=5)	<LOQ[c] (n=5)	<LOQ[d] (n=5)	NA	NA	NA
		F	0.126 ± 0.0159 (n=5)	<LOQ[c] (n=5)	<LOQ[d] (n=5)	NA	NA	NA
	Group 9 1.0 ppm	M	2.19 ± 0.733 (n=5)	0.0110 ± 0.000716 (n=5)	0.497 ± 0.618 (n=5)	NA	NA	NA
		F	3.26 ± 0.629 (n=6)	0.0164 ± 0.00354 (n=6)	0.578 ± 0.425 (n=6)	NA	NA	NA
Week 53	Group 8 0.0 ppm	M	0.0490 ± 0.0352 (n=5)	<LOQ[e] (n=5)	<LOQ[f] (n=5)	<LOQ[g] (n=5)	<LOQ[h] (n=5)	<LOQ[i] (n=5)
		F	0.0727 ± 0.0565 (n=5)	<LOQ[e] (n=5)	<LOQ[f] (n=5)	<LOQ[g] (n=5)	<LOQ[h] (n=5)	<LOQ[i] (n=5)
	Group 9 1.0 ppm	M	1.35 ± 0.452 (n=5)	0.00672 ± 0.00251 (n=5)	0.297 ± 0.194 (n=5)	<LOQ[g] (n=5)	0.120 ± 0.0289 (n=5)	<LOQ[i] (n=5)
		F	3.02 ± 1.54 (n=5)	0.0102 ± 0.00252 (n=5)	0.273 ± 0.110 (n=5)	<LOQ[g] (n=5)	0.202 ± 0.0723 (n=5)	<LOQ[i] (n=5)

NA—Not applicable
*Qualitative data on 4 points in calibration curve
**EtFOSE and PFOSEA results are qualitative only.
[a]LOQ—Limit of Quantitation = 0.0230 µg/mL
[b]LOQ—Limit of Quantitation = 0.0494 µg/mL
[c]LOQ—Limit of Quantitation = 0.00989 µg/mL
[d]LOQ—Limit of Quantitation = 0.0282 µg/mL
[e]LOQ—Limit of Quantitation = 0.00516 µg/mL
[f]LOQ—Limit of Quantitation = 0.0253 µg/mL
[g]LOQ—Limit of Quantitation = 0.0225 µg/mL
[h]LOQ—Limit of Quantitation = 0.00494 µg/mL
[i]LOQ—Limit of Quantitation = 0.00978 µg/mL
NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.
NOTE: All PFOSA, PFOSAA, PFOSEA, M556, and PFOS Week 4 results are not corrected for purity of the reference standard material.
It is not possible to verify true recovery of endogenous analyte from tissues without radio-labeled reference material. The only measurement of accuracy available at this time, matrix spike studies, indicate that, with the exception of PFOSEA and EtFOSE-OH, the sera and liver data can be considered to be accurate to within one standard deviation of the average fortified samples recovery.

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Table 9. (Continued) FACT TOX-003 Data Summary for PFOS, PFOSA, PFOSAA, EtFOSE, M556 and PFOSEA—Rat Serum (µg/mL)

Timept	Group		PFOS Avg ± SD	PFOSA Avg ± SD	PFOSAA Avg ± SD	EtFOSE** Avg ± SD	M556 Avg ± SD	PFOSEA** Avg ± SD
Week 105	Group 8 0.0 ppm	M	0.0137 ± 0.0181 (n=10)	<LOQ[e] (n=10)	<LOQ[j] (n=10)	<LOQ[k] (n=10)	<LOQ[l] (n=10)	<LOQ[m] (n=10)
		F	0.0256 ± 0.0145 (n=10)	<LOQ[e] (n=10)	<LOQ[j] (n=10)	<LOQ[k] (n=10)	<LOQ[l] (n=10)	<LOQ[m] (n=10)
	Group 9 1.0 ppm	M	1.12 ± 1.10 (n=10)	0.00720 ± 0.00330 (n=10)	0.252 ± 0.160 (n=10)	<LOQ[k] (n=10)	0.0645 ± 0.0445 (n=10)	<LOQ[m] (n=10)
		F	1.61 ± 0.631 (n=10)	0.00797 ± 0.00198 (n=10)	0.259 ± 0.156 (n=10)	<LOQ[k] (n=10)	0.0868 ± 0.0294 (n=10)	<LOQ[m] (n=10)

**EtFOSE and PFOSEA results are qualitative only.
[a]LOQ—Limit of Quantitation = 0.0230 µg/mL
[b]LOQ—Limit of Quantitation = 0.0494 µg/mL
[c]LOQ—Limit of Quantitation = 0.00989 µg/mL
[d]LOQ—Limit of Quantitation = 0.0282 µg/mL
[e]LOQ—Limit of Quantitation = 0.00516 µg/mL
[f]LOQ—Limit of Quantitation = 0.0253 µg/mL
[g]LOQ—Limit of Quantitation = 0.0225 µg/mL
[h]LOQ—Limit of Quantitation = 0.00494 µg/mL
[i]LOQ—Limit of Quantitation = 0.00978 µg/mL
[j]LOQ—Limit of Quantitation = 0.00503 µg/mL
[k]LOQ—Limit of Quantitation = 0.00448 µg/mL
[l]LOQ—Limit of Quantitation = 0.0248 µg/mL
[m]LOQ—Limit of Quantitation = 0.00493 µg/mL

NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.

NOTE: All PFOSA, PFOSAA, PFOSEA, M556, and PFOS Week 4 results are not corrected for purity of the reference standard material.

It is not possible to verify true recovery of endogenous analyte from tissues without radio-labeled reference material. The only measurement of accuracy available at this time, matrix spike studies, indicate that, with the exception of PFOSEA and EtFOSE-OH, the sera and liver data can be considered to be accurate to within one standard deviation of the average fortified samples recovery.

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Table 10. FACT TOX-003 Data Summary for PFOS, PFOSA, PFOSAA, EtFOSE, M556 and PFOSEA—Rat Liver (µg/g)

Timept	Group		PFOS Avg ± SD	PFOSA Avg ± SD	PFOSAA Avg ± SD	EtFOSE* Avg ± SD	M556 Avg ± SD	PFOSEA* Avg ± SD
Week 4	Group 8 0.0 ppm	M	0.198 ± 0.0822 (n=5)	<LOQ[a] (n=5)	<LOQ[b] (n=5)	NR	NA	NA
		F	0.0891 ± 0.0315 (n=5)	<LOQ[a] (n=5)	<LOQ[b] (n=5)	NR	NA	NA
	Group 9 1.0 ppm	M	6.53 ± 0.628 (n=5)	0.187 ± 0.0401 (n=5)	0.683 ± 0.613 (n=5)	NR	NA	NA
		F	2.97 ± 0.937 (n=5)	0.222 ± 0.104 (n=5)	0.582 ± 0.193 (n=5)	NR	NA	NA
Week 14	Group 8 0.0 ppm	M	2.96 ± 4.83 (n=5)	0.0822 ± 0.156 (n=5)	0.330 ± 0.656 (n=5)	NR	NA	NA
		F	0.411 ± 0.0625 (n=5)	0.0130 ± 0.000872 (n=5)	0.0411 ± 0.0121 (n=5)	NR	NA	NA
	Group 9 1.0 ppm	M	12.4 ± 6.99 (n=5)	0.366 ± 0.221 (n=5)	2.89 ± 1.88 (n=5)	NR	NA	NA
		F	9.19 ± 1.53 (n=5)	0.580 ± 0.239 (n=5)	2.61 ± 2.49 (n=5)	NR	NA	NA
Week 53	Group 8 0.0 ppm	M	1.44 ± 1.18 (n=5)	<LOQ[c] (n=5)	<LOQ[d] (n=5)	<LOQ[e] (n=5)	<LOQ[f] (n=5)	<LOQ[g] (n=5)
		F	0.178 ± 0.115 (n=5)	0.00647 ± 0.0000786 (n=5)	<LOQ[d] (n=5)	<LOQ[e] (n=5)	<LOQ[f] (n=5)	<LOQ[g] (n=5)
	Group 9 1.0 ppm	M	37.1 ± 10.9 (n=5)	0.204 ± 0.0769 (n=5)	0.906 ± 0.605 (n=5)	<LOQ[e] (n=5)	0.336 ± 0.0682 (n=5)	<LOQ[g] (n=5)
		F	25.2 ± 13.9 (n=5)	0.242 ± 0.0999 (n=5)	0.527 ± 0.131 (n=5)	<LOQ[e] (n=5)	0.267 ± 0.0660 (n=5)	<LOQ[g] (n=5)

*EtFOSE and PFOSEA results are qualitative only.
NR—No result
NA—Not applicable

[a]LOQ—Limit of Quantitation = 0.0121 µg/g
[b]LOQ—Limit of Quantitation = 0.0341 µg/g
[c]LOQ—Limit of Quantitation = 0.00644 µg/g
[d]LOQ—Limit of Quantitation = 0.0125 µg/g
[e]LOQ—Limit of Quantitation = 0.0559 µg/g
[f]LOQ—Limit of Quantitation = 0.0307 µg/g
[g]LOQ—Limit of Quantitation = 0.0308 µg/g
[h]LOQ—Limit of Quantitation = 0.0322 µg/g
[i]LOQ—Limit of Quantitation = 0.0314 µg/g

NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.

NOTE: All PFOSA, PFOSAA, PFOSEA, M556 and EtFOSE-OH Week 4 and Week 14 results are not corrected for purity of the reference standard material.

It is not possible to verify true recovery of endogenous analyte from tissues without radio-labeled reference material. The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered to be accurate to within one standard deviation of the average fortified samples recovery.

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Table 10. (Continued) FACT TOX-003 Data Summary for PFOS, PFOSA, PFOSAA, EtFOSE, M556 and PFOSEA—Rat Liver (µg/g)

Timept	Group		PFOS Avg ± SD	PFOSA Avg ± SD	PFOSAA Avg ± SD	EtFOSE* Avg ± SD	M556 Avg ± SD	PFOSEA* Avg ± SD
Week 105	Group 8 0.0 ppm	M	0.0653 ± 0.0923 (n=10)	<LOQ[h] (n=10)	<LOQ[i] (n=10)	<LOQ[e] (n=10)	<LOQ[f] (n=10)	<LOQ[g] (n=10)
		F	0.0872 ± 0.0522 (n=10)	<LOQ[h] (n=10)	<LOQ[i] (n=10)	<LOQ[e] (n=10)	<LOQ[f] (n=10)	<LOQ[g] (n=10)
	Group 9 1.0 ppm	M	6.21 ± 3.80 (n=10)	0.371 ± 0.187 (n=10)	0.736 ± 0.551 (n=10)	<LOQ[e] (n=10)	0.250 ± 0.0865 (n=10)	<LOQ[g] (n=10)
		F	9.63 ± 4.35 (n=10)	0.550 ± 0.112 (n=10)	0.771 ± 0.411 (n=10)	<LOQ[e] (n=10)	0.336 ± 0.0748 (n=10)	<LOQ[g] (n=10)

*EtFOSE and PFOSEA results are qualitative only.
NR—No result
NA—Not applicable

[a]LOQ—Limit of Quantitation = 0.0121 µg/g
[b]LOQ—Limit of Quantitation = 0.0341 µg/g
[c]LOQ—Limit of Quantitation = 0.00644 µg/g
[d]LOQ—Limit of Quantitation = 0.0125 µg/g
[e]LOQ—Limit of Quantitation = 0.0559 µg/g
[f]LOQ—Limit of Quantitation = 0.0307 µg/g
[g]LOQ—Limit of Quantitation = 0.0308 µg/g
[h]LOQ—Limit of Quantitation = 0.0322 µg/g
[i]LOQ—Limit of Quantitation = 0.0314 µg/g

NOTE: Results are expressed as group/gender averages ± the standard deviation associated with that group/gender.

NOTE: All PFOSA, PFOSAA, PFOSEA, M556 and EtFOSE-OH Week 4 and Week 14 results are not corrected for purity of the reference standard material.

It is not possible to verify true recovery of endogenous analyte from tissues without radio-labeled reference material. The only measurement of accuracy available at this time, matrix spike studies, indicate that the sera and liver data can be considered to be accurate to within one standard deviation of the average fortified samples recovery.

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Appendix E: Data Spreadsheets

Covance# 6329-228

Study:	TOX003 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M 3.0 & FACT-M 4.0, ETS-8-51
Analytical Equipment System Number:	Machine 041098
Instrument Software/Version:	MassLynx 3.1
Filename:	See List to Right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Date of Extraction/Analyst:	5/27/98 OK
Date of Analysis/Analyst:	07/13/98, 08/30/00 MEE/MMH
Date of Data Reduction/Analyst:	2/14/00, 8/31/00 mmh

Sample Data

WEEK 4 RAT SERA

Group Dose	Sample #	Extraction Vol. Ratio	PFOS Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ug/mL	Filename	Concentration of PFOS ug/mL or % Rec	Average PFOS ug/mL	RSD Std. Dev.	PFOSA Purity Correction Factor	PFOSA Dilution Factor	PFOSA Conc. ug/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Average PFOSA ug/mL	RSD Std. Dev.	PFOSAA Old Purity Correction Factor	PFOSAA Purity Correction Factor	PFOSAA Dilution Factor	PFOSAA Conc. ug/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Average PFOSAA ug/mL	RSD Std. Dev.
				lot 215							lot L-2353							lot 617							
Method Blk	H2O Blk-1	1	0.9275	Unknown	1	0.00	M71398011	<LOQ (0.0230 ug/mL)			Unknown	1	1.99	M71398011	<LOQ (0.0494 ug/mL)			0.5382	Unknown	1	NR	NR	NR		
Method Blk	H2O Blk-2	1	0.9275	Unknown	NR	NR	NR	NR	<LOQ	NA	Unknown	1	NR	NR	NR	<LOQ	NA	0.5382	Unknown	1	NR	NR	NR	NR	NR
Matrix Blk	Rat Serum Blk-1	1	0.9275	Unknown	NR	NR	NR	NR			Unknown	1	NR	NR	NR			0.5382	Unknown	1	NR	NR	NR		
Matrix Blk	Rat Serum Blk-2	1	0.9275	Unknown	1	0.00	M71398002	<LOQ (0.0230 ug/mL)	<LOQ	NA	Unknown	1	0.00	M71398002	<LOQ (0.0494 ug/mL)	<LOQ	NA	0.5382	Unknown	1	NR	NR	NR	NR	NR
QC-75 ppb	RTS-052698-M5	1	NA	NA	1	108	M71398041	144%			NA	1	82.6	A083000016	112%			NA	NA	1	86.9	A083000016	115% A		
	RTS-052698-MSD	1	NA	NA	1	70.8	M71398042	96%	120%	40%	NA	1	81.7	A083000017	111%	111%	1%	NA	NA	1	90.9	A083000017	120% A	117%	5%
	RTS-052698-M5	1	NA	NA	1	166	A083000016	226% 2nd			NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA		
	RTS-052698-MSD	1	NA	NA	1	142	A083000017	193% 2nd	210%	16%	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 8 Control 0.0 ppm	C92332F	1	0.9275	Unknown	1	21.5	M71398013	<LOQ (0.0230 ug/mL) 1			Unknown	1	0.84	A083000021	<LOQ (0.0494 ug/mL)			0.5382	Unknown	1	18.7	A083000021	0.0101 A		
	C92352F	1	0.9275	Unknown	1	11.0	M71398014	<LOQ (0.0230 ug/mL) 1			Unknown	1	0.84	A083000022	<LOQ (0.0494 ug/mL)			0.5382	Unknown	1	19.1	A083000022	0.0103 A		
	C92355F	1	0.9275	Unknown	1	5.22	M71398015	<LOQ (0.0230 ug/mL) 1			Unknown	1	0.70	A083000023	<LOQ (0.0494 ug/mL)			0.5382	Unknown	1	11.7	A083000023	0.00629 A		
	C92366F	1	0.9275	Unknown	1	5.01	M71398016	<LOQ (0.0230 ug/mL) 1		NA	Unknown	1	0.78	A083000024	<LOQ (0.0494 ug/mL)		NA	0.5382	Unknown	1	16.1	A083000024	0.00864 A		36.3
	C92371F	1	0.9275	Unknown	1	3.08	M71398017	<LOQ (0.0230 ug/mL) 1	<LOQ	NA	Unknown	1	0.83	A083000025	<LOQ (0.0494 ug/mL)	<LOQ	NA	0.5382	Unknown	1	30.5	A083000025	0.0164 A	0.0103	0.00375
	C92192M	1	0.9275	Unknown	1	0.00	M71398020	<LOQ (0.0230 ug/mL) 1			Unknown	1	0.63	A083000030	<LOQ (0.0494 ug/mL)			0.5382	Unknown	1	5.68	A083000030	0.00306 A		
	C92196M	1	0.9275	Unknown	1	0.00	M71398021	<LOQ (0.0230 ug/mL) 1			Unknown	1	0.71	A083000031	<LOQ (0.0494 ug/mL)			0.5382	Unknown	1	11.9	A083000031	0.00640 A		
	C92207M	1	0.9275	Unknown	1	0.00	M71398022	<LOQ (0.0230 ug/mL) 1			Unknown	1	0.62	A083000032	<LOQ (0.0494 ug/mL)			0.5382	Unknown	1	5.45	A083000032	0.00293 A		
	C92210dM	1	0.9275	Unknown	1	0.00	M71398023	<LOQ (0.0230 ug/mL) 1		NA	Unknown	1	0.57	A083000033	<LOQ (0.0494 ug/mL)		NA	0.5382	Unknown	1	8.82	A083000033	0.00475 A		34.3
	C92259M	1	0.9275	Unknown	1	0.00	M71398024	<LOQ (0.0230 ug/mL) 1	<LOQ	NA	Unknown	1	0.66	A083000034	<LOQ (0.0494 ug/mL)	<LOQ	NA	0.5382	Unknown	1	10.9	A083000034	0.00584 A	0.00460	0.00158
Group 9 Low Dose 1.0 ppm	C92435F	1	0.9275	Unknown	10	28.3	M71398027	0.262 3			Unknown	1	7.25	A083000038	0.00725			0.5382	Unknown	1	383	A083000038	0.206 A		
	C92402F	1	0.9275	Unknown	10	36.0	M71398028	0.334 3			Unknown	1	8.85	A083000039	0.00885			0.5382	Unknown	1	509	A083000039	0.274 A		
	C92463F	1	0.9275	Unknown	10	19.8	M71398029	0.183 3			Unknown	1	5.24	A083000040	0.00524			0.5382	Unknown	1	336	A083000040	0.181 A		
	C92467F	1	0.9275	Unknown	10	37.1	M71398030	0.344 3		25.4	Unknown	1	11.6	A083000041	0.0116		32.7	0.5382	Unknown	1	664	A083000041	0.358 A		37.9
	C92470F	1	0.9275	Unknown	10	24.6	M71398031	0.229 3	0.270	0.0687	Unknown	1	5.95	A083000042	0.00595	0.00778	0.00254	0.5382	Unknown	1	837	A083000042	0.451 4A	0.294	0.111
	C92264M	1	0.9275	Unknown	5	34.7	M71398034	0.161 2			Unknown	1	4.37	A083000047	< LOQ (0.0494 ug/mL)			0.5382	Unknown	1	330	A083000047	0.177 A		
	C92280M	1	0.9275	Unknown	5	41.7	M71398035	0.193 2			Unknown	1	3.49	A083000048	< LOQ (0.0494 ug/mL)			0.5382	Unknown	1	249	A083000048	0.134 A		
	C92284M	1	0.9275	Unknown	5	29.4	M71398036	0.136 2			Unknown	1	5.22	A083000049	0.00522			0.5382	Unknown	1	369	A083000049	0.198 A		
	C92300M	1	0.9275	Unknown	5	27.2	M71398037	0.126 2		29.4	Unknown	1	4.18	A083000050	< LOQ (0.0494 ug/mL)		2.51	0.5382	Unknown	1	248	A083000050	0.134 A		78.9
	C92329M	1	0.9275	Unknown	5	54.5	M71398038	0.253 2	0.174	0.0510	Unknown	1	4.89	A083000051	< LOQ (0.0494 ug/mL)	0.00500	0.000125	0.5382	Unknown	1	1100	A083000051	0.592 4A	0.247	0.195

Date Entered/By: 12/19/00, 1/5/01 mmh
Date Verified/ By: 03/20/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate

1 = CCVs failed. Data is estimated and is biased high
2 = CCVs failed. Data is estimated and is biased low.
3 = CCVs failed. Data is estimated.
4 = Extract above calibration range. Data is estimated.
A = Qualitative only, 4 calibration points were used to plot the calibration curve. LAC 05/03/01

		PFOS	PFOSA	PFOSAA
07/13/98	Initial LOQ ug/mL	24.8	4.94	10.5
	Corrected LOQ ug/mL	0.0230	0.00494	0.0108
08/30/00	Initial LOQ ug/mL	9.76	4.94	5.20
	Corrected LOQ ug/mL	0.00905	0.00494	0.00533

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance)	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	FACT-M-3.0 & FACT-M-4.0; ETS-8-5.1
Analytical Equipment System Number	Machine 041008
Instrument Software/Version:	MassLynx 3.1
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Date of Extraction/Analyst:	7/13/98 LAS/SAH
Date of Analysis/Analyst:	7/17/98, 8/7/98 MEE/HOJ
Date of Data Reduction/Analyst	2/10/00 mmh

Sample Data

WEEK 14 RAT SERA

Group Dose	Sample #	Extraction Vol. Ratio	PFOS Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ug/mL	Filename	Concentration of PFOS ug/mL or % Rec	Average PFOS ug/mL	RSD Std. Dev.	PFOSA Purity Correction Factor	PFOSA Dilution Factor	PFOSA Conc. ug/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Average PFOSA ug/mL	RSD Std. Dev.	PFOSAA Old Purity Correction Factor	PFOSAA Purity Correction Factor	PFOSAA Dilution Factor	PFOSAA Conc. ug/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Average PFOSAA ug/mL	RSD Std. Dev.
				lot 193							Lot L-2353							lot 617							
Method Blk	H2O Blk-1	1	0.9275	Unknown	1	1.21	M71798002	<LOQ (0.0230 ug/mL)			Unknown	1	1.50	M71798002	<LOQ (0.00989 ug/mL)			0.5382	Unknown	1	0.00	M71798002	<LOQ (0.0282 ug/mL)		
	H2O Blk-2	1	0.9275	Unknown	1	0.00	M71798043	<LOQ (0.0230 ug/mL)	<LOQ	NA	Unknown	1	0.00	M71798043	<LOQ (0.00989 ug/mL)	<LOQ	NA	0.5382	Unknown	1	0.00	M71798043	<LOQ (0.0282 ug/mL)	<LOQ	NA
Matrix Blk	Rat Serum Blk-1	1	0.9275	Unknown	1	4.68	M71798003	<LOQ (0.0230 ug/mL)			Unknown	1	1.59	M71798003	<LOQ (0.00989 ug/mL)			0.5382	Unknown	1	0.00	M71798003	<LOQ (0.0282 ug/mL)		
	Rat Serum Blk-2	1	0.9275	Unknown	1	2.04	M71798003	<LOQ (0.0230 ug/mL)	<LOQ	NA	Unknown	1	0.00	M71798044	<LOQ (0.00989 ug/mL)	<LOQ	NA	0.5382	Unknown	1	0.00	M71798003	<LOQ (0.0282 ug/mL)	<LOQ	NA
QC-100 ppb	RTS07158-MS	1	NA	NA	1	107	M71798040	109%			NA	1	102	M71798040	103%			NA	NA	1	76.6	M71798040	74%		
	RTS07158-MSD	1	NA	NA	1	114	M71798041	117%	113%	7%	NA	1	109	M71798041	110%	107%	7%	NA	NA	1	85.1	M71798041	82%	78%	11%
Group 8 Control 0.0 ppm	C92255M	1	0.9275	Unknown	1	58.9	M71798012	0.0546			Unknown	1	1.95	M71798012	<LOQ (0.00989 ug/mL)			0.5382	Unknown	1	0.10	M71798012	<LOQ (0.0282 ug/mL)		
	C92256M	1	0.9275	Unknown	1	31.0	M71798013	0.0288			Unknown	1	1.69	M71798013	<LOQ (0.00989 ug/mL)			0.5382	Unknown	1	0.00	M71798013	<LOQ (0.0282 ug/mL)		
	C92257M	1	0.9275	Unknown	1	40.5	M71798014	0.0376			Unknown	1	1.59	M71798014	<LOQ (0.00989 ug/mL)			0.5382	Unknown	1	0.00	M71798014	<LOQ (0.0282 ug/mL)		
	C92258M	1	0.9275	Unknown	1	39.0	M71798015	0.0362		27.8	Unknown	1	1.63	M71798015	<LOQ (0.00989 ug/mL)		NA	0.5382	Unknown	1	0.00	M71798015	<LOQ (0.0282 ug/mL)		NA
	C92260M	1	0.9275	Unknown	1	**	NA	**	0.0393	0.0109	Unknown	1	**	NA	**	<LOQ	NA	0.5382	Unknown	1	**	NA	**	<LOQ	NA
	C92396F	1	0.9275	Unknown	1	152	M71798018	0.141			Unknown	1	1.69	M71798018	<LOQ (0.00989 ug/mL)			0.5382	Unknown	1	3.35	M71798018	<LOQ (0.0282 ug/mL)		
	C92397F	1	0.9275	Unknown	1	130	M71798019	0.120			Unknown	1	1.64	M71798019	<LOQ (0.00989 ug/mL)			0.5382	Unknown	1	0.00	M71798019	<LOQ (0.0282 ug/mL)		
	C92398F	1	0.9275	Unknown	1	120	M71798020	0.111			Unknown	1	1.35	M71798020	<LOQ (0.00989 ug/mL)			0.5382	Unknown	1	0.00	M71798020	<LOQ (0.0282 ug/mL)		
	C92399F	1	0.9275	Unknown	1	156	M71798021	0.144		12.6	Unknown	1	1.54	M71798021	<LOQ (0.00989 ug/mL)		NA	0.5382	Unknown	1	0.00	M71798021	<LOQ (0.0282 ug/mL)		NA
	C92400F	1	0.9275	Unknown	1	121	M71798022	0.112	0.126	0.0159	Unknown	1	1.61	M71798022	<LOQ (0.00989 ug/mL)	<LOQ	NA	0.5382	Unknown	1	0.00	M71798022	<LOQ (0.0282 ug/mL)	<LOQ	NA
Group 9 Low Dose 1.0 ppm	C92325M	1	0.9275	Unknown	1	1534	M71798025	1.42 1			Unknown	1	11.2	M71798025	0.0112			0.5382	Unknown	1	437	M71798025	0.235		
	C92326M	1	0.9275	Unknown	25	142	A80798014	3.30			Unknown	1	11.6	M71798026	0.0116			0.5382	Unknown	25	118	A80798015	1.59		
	C92327M	1	0.9275	Unknown	25	77.4	A80798015	1.80			Unknown	1	11.6	M71798027	0.0116			0.5382	Unknown	1	553	M71798027	0.297		
	C92328M	1	0.9275	Unknown	25	83.0	A80798016	1.92		33.5	Unknown	1	7.98	M71798028	<LOQ (0.00989 ug/mL)		6.48	0.5382	Unknown	1	194	M71798028	0.104		124
	C92330M	1	0.9275	Unknown	25	108	A80798017	2.51	2.19	0.733	Unknown	1	10.8	M71798029	0.0108	0.0110	0.000716	0.5382	Unknown	1	470	M71798029	0.253	0.497	0.618
	C92464F 1/2	1	0.9275	Unknown	50	79.3	A80798020	3.68			Unknown	1	17.5	M71798032	0.0175			0.5382	Unknown	50	37.8	A80798020	1.02		
	C92464F 2/2	1	0.9275	Unknown	1	3384	M71798033	3.14 1			Unknown	1	17.9	M71798033	0.0179			0.5382	Unknown	1	1275	M71798033	0.686 1*		
	C92465F	1	0.9275	Unknown	50	59.9	A80798021	2.78			Unknown	1	14.7	M71798034	0.0147			0.5382	Unknown	1	587	M71798034	0.316		
	C92466F	1	0.9275	Unknown	50	79.6	A80798022	3.69			Unknown	1	12.9	M71798035	0.0129			0.5382	Unknown	1	300	M71798035	0.162		
	C92468F	1	0.9275	Unknown	50	50.1	A80798023	2.32		19.3	Unknown	1	13.1	M71798036	0.0131		21.6	0.5382	Unknown	1	317	M71798036	0.171		73.5
	C92469F	1	0.9275	Unknown	50	85.4	A80798024	3.96	3.26	0.629	Unknown	1	22.2	M71798037	0.0222	0.0164	0.00354	0.5382	Unknown	50	41.5	A80798024	1.12	0.578	0.425

* Sample went to dryness after initial injection and prior to dilutions.
1 Extract above calibration range. Data is estimated.
** Sample spilled during extraction, no sample remaining.

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate

Date Entered/By: 12/22/00 & 1/8/01 mmh
Date Verified/ By: 03/29/01 tjh, 5/22/01 mmh
Party Entered/Verifie 03/29/01 LAC/

		PFOS	PFOSA	PFOSAA
07/13/98	Initial LOQ ug/mL	24.8	4.94	10.5
	Corrected LOQ ug/mL	0.0230	0.00494	0.00565
07/17/98	Initial LOQ ug/mL	24.8	9.89	52.4
	Corrected LOQ ug/mL	0.0230	0.00989	0.0282
08/07/98	Initial LOQ ug/mL	24.8	9.89	52.4
	Corrected LOQ ug/mL	0.0230	0.00989	0.0282

Covance# 6329-228

Study	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number/Test Substance	T-6316 (EtFOSE-OH)
Matrix	Rat Serum
Method/Revision	ETS-8-4.1 & ETS-8-5.1
Analysis Equipment System Number	Ruby 100699
Instrument Software/Version	Masslynx 3.4
Filename	See listing to the right
R-Squared Value	See Attachments
Slope	See Attachments
Y-Intercept	See Attachments
Dates of Extraction/Analyst	04/20/00 SAL/KJK
Dates of Analysis/Analyst	04/21/00, 04/24/00, 08/15/00, 08/28/00 LAS/MMH/HOI/SAH
Date of Data Reduction/Analyst	05/05/00, 04/25/00, 08/16/00, 08/30/00 MMH/LAS

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Old Purity Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	PFOSA Old Purity Correction Factor	PFOSA Purity Correction Factor	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD
					lot 171									lot L-15709						
Method Blk	04200-H2O Blk-3	1	NA	0.9949	0.8640	1	0.00	R042100016	< LOQ (0.00428 ug/mL)			NA	0.9510	Unknown	1	0.00	R042100016	< LOQ (0.00516 ug/mL)		
	04200-H2O Blk-4	1	NA	0.9949	0.8640	1	0.00	R042100017	< LOQ (0.00428 ug/mL)	<LOQ	NA	NA	0.9510	Unknown	1	0.20	R042100017	< LOQ (0.00516 ug/mL)	<LOQ	NA
Matrix Blk	RBS04200-Sera Blk-3	1	NA	0.9949	0.8640	1	0.00	R042100018	< LOQ (0.00428 ug/mL)			NA	0.9510	Unknown	1	0.00	R042100018	< LOQ (0.00516 ug/mL)		
	RBS04200-Sera Blk-4	1	NA	0.9949	0.8640	1	0.00	R042100019	< LOQ (0.00428 ug/mL)	<LOQ	NA	NA	0.9510	Unknown	1	0.00	R042100019	< LOQ (0.00516 ug/mL)	<LOQ	NA
	RTS04200-Sera Blk-3	1	NA	0.9949	0.8640	1	0.00	R042100020	< LOQ (0.00428 ug/mL)			NA	0.9510	Unknown	1	0.00	R042100020	< LOQ (0.00516 ug/mL)		
	RTS04200-Sera Blk-4	1	NA	0.9949	0.8640	1	0.00	R042100024	< LOQ (0.00428 ug/mL)	<LOQ	NA	NA	0.9510	Unknown	1	0.00	R042100024	< LOQ (0.00516 ug/mL)	<LOQ	NA
QC	RTS04200-MS-250-3	1	NA	NA	NA	1	182	R042100025	73%			NA	NA	NA	1	196	R042100025	79%		
	RTS04200-MSD-250-3	1	NA	NA	NA	1	197	R042100026	79%	76%	8%	NA	NA	NA	1	222	R042100026	90%	84%	12%
	RTS04200-MS-250-3	1	NA	NA	NA	1	NA	NA	NA			NA	NA	NA	1	NA	NA	NA		
	RTS04200-MSD-250-3	1	NA	NA	NA	1	NA	NA	NA	NA	NA	NA	NA	NA	1	NA	NA	NA	NA	NA
Group 8 Control 0.0 ppm	C92195M	1	NA	0.9949	0.8640	1	117	R042100027	0.101			NA	0.9510	Unknown	1	0.00	R042100027	< LOQ (0.00516 ug/mL)		
	C92199M	1	NA	0.9949	0.8640	1	35.9	R042100028	0.0312			NA	0.9510	Unknown	1	0.00	R042100028	< LOQ (0.00516 ug/mL)		
	C92205M	1	NA	0.9949	0.8640	1	32.0	R042100032	0.0278			NA	0.9510	Unknown	1	0.00	R042100032	< LOQ (0.00516 ug/mL)		
	C92236M	1	NA	0.9949	0.8640	1	18.9	R042100033	0.0164		71.7	NA	0.9510	Unknown	1	0.00	R042100033	< LOQ (0.00516 ug/mL)		NA
	C92246M	1	NA	0.9949	0.8640	1	79.1	R042100034	0.0687	0.0490	0.0352	NA	0.9510	Unknown	1	0.00	R042100034	< LOQ (0.00516 ug/mL)	<LOQ	NA
	C92341F	1	NA	0.9949	0.8640	1	2.47	R042100035	< LOQ (0.00428 ug/mL)			NA	0.9510	Unknown	1	0.00	R042100035	< LOQ (0.00516 ug/mL)		
	C92356F	1	NA	0.9949	0.8640	1	25.3	R042100036	0.0219			NA	0.9510	Unknown	1	0.00	R042100036	< LOQ (0.00516 ug/mL)		
	C92372F	1	NA	0.9949	0.8640	1	104	R042100040	0.0905			NA	0.9510	Unknown	1	0.00	R042100040	< LOQ (0.00516 ug/mL)		
	C92376F	1	NA	0.9949	0.8640	1	148	R042100041	0.129		77.7	NA	0.9510	Unknown	1	0.00	R042100041	< LOQ (0.00516 ug/mL)		NA
	C92396F	1	NA	0.9949	0.8640	1	136	R042100042	0.118	0.0777	0.0565	NA	0.9510	Unknown	1	0.00	R042100042	< LOQ (0.00516 ug/mL)	<LOQ	NA
Group 9 Low Dose 1.0 ppm	C92267M	1	NA	0.9949	0.8640	10	210	D081500025	1.82			NA	0.9510	Unknown	1	3.55	R042100043	< LOQ (0.00516 ug/mL)		
	C92274M	1	NA	0.9949	0.8640	10	140	D081500026	1.21			NA	0.9510	Unknown	1	4.54	R042100044	< LOQ (0.00516 ug/mL)		
	C92287M	1	NA	0.9949	0.8640	10	191	D081500027	1.65			NA	0.9510	Unknown	1	6.85	R042100048	0.00720		
	C92284M	1	NA	0.9949	0.8640	10	164	D081500028	1.43		33.4	NA	0.9510	Unknown	1	10.4	R042100049	0.0109		37.3
	C92320M	1	NA	0.9949	0.8640	1	759	D081500029 A	0.659	1.35	0.452	NA	0.9510	Unknown	1	4.09	R042100050	< LOQ (0.00516 ug/mL)	0.00672	0.00251
	C92406F	1	NA	0.9949	0.8640	10	417	D081500032	3.62			NA	0.9510	Unknown	1	8.52	R042100051	0.00896		
	C92411F	1	NA	0.9949	0.8640	10	450	D081500033	3.90			NA	0.9510	Unknown	1	8.41	R042100052	0.00884		
	C92418F	1	NA	0.9949	0.8640	10	452	D081500034	3.92			NA	0.9510	Unknown	1	9.49	R042100056	0.00998		
	C92422F	1	NA	0.9949	0.8640	10	386	D081500035	3.35		51.1	NA	0.9510	Unknown	1	13.9	R042100057	0.0146		24.7
	C92438F	1	NA	0.9949	0.8640	1	338	R042100058	0.294	3.02	1.54	NA	0.9510	Unknown	1	8.21	R042100058	0.00863	0.0102	0.00252

* Recoveries confirmed LAC 09/03/00

A = Estimated concentration, above the calibration range LAC 05/10/01

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N((H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

Date Entered/By: 05/12/00, 06/01/00, 08/22/00, 08/23/00, 09/03/00 LAC/MMH 05/09/01 mmh
Date Verified/ By: 03/28/01 tjh, 05/10/01 LAC, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/

		PFOS	PFOSA	PFOSAA	EtFOSE	M556	PFOSEA
04/21/00	Initial LOQ ng/mL	4.93	4.91	49.1	24.7	49.3	9.71
	Corrected LOQ ng/mL	0.00428	0.00516	0.0503	0.0225	0.0494	0.00978
04/24/00	Initial LOQ ng/mL	NA	NA	24.7	NA	97.6	NA
	Corrected LOQ ng/mL	NA	NA	0.0253	NA	0.0977	NA
08/15/00	Initial LOQ ng/mL	4.93	NA	NA	4.92	24.8	24.6
	Corrected LOQ ng/mL	0.00428	NA	NA	0.00448	0.0248	0.0248
08/28/00	Initial LOQ ng/mL	4.93	4.91	NA	NA	4.93	NA
	Corrected LOQ ng/mL	0.00428	0.00516	NA	NA	0.00494	NA

See Week 53

Covance# 6329-228

Study	TOX003: 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Protocol Number(Test Substance)	T-6316 (EtFOSE-OH)
Matrix	Rat Serum
Method/Revision	ETS-8-4-1 & ETS-8-5-1
Analytical Equipment System Number	Ruby 100699
Instrument Software/Version	Masslynx 3.4
Filename	See listing to the right
R Squared Value	See Attachments
Slope	See Attachments
Y-Intercept	See Attachments
Dates of Extraction/Analyst	04/20/00 SAL/KJK
Dates of Analysis/Analyst	04/21/00, 04/24/00, 08/15/00, 08/28/00 IAS/MMH/HOI/SAH
Date of Data Reduction/Analyst	05/05/00, 04/25/00, 08/16/00, 08/30/00 MMH/IAS

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	Surrogate Verified	PFOSAA Old Purity Correction Factor (lot 98-0207-030-3)	PFOSAA Purity Correction Factor	PFOSAA Dilution Factor	PFOSAA Conc. ug/mL	Filename	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	EtFOSE Old Purity Correction Factor (lot SD013)	EtFOSE Purity Correction Factor	EtFOSE Dilution Factor	EtFOSE Conc. ug/mL	Filename	Concentration of EtFOSE ug/mL or % Rec	Mean EtFOSE ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	04200-H2O Blk-3	NA	0.9760	Unknown	1	0.00	R042400025	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100016	< LOQ (0.0225 ug/mL)		
	04200-H2O Blk-4	NA	0.9760	Unknown	1	0.00	R042400026	< LOQ (0.0253 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	0.00	R042100017	< LOQ (0.0225 ug/mL)	<LOQ	NA
Matrix Blk	RBS04200-Sera Blk-3	NA	0.9760	Unknown	1	0.00	R042400027	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100018	< LOQ (0.0225 ug/mL)		
	RBS04200-Sera Blk-4	NA	0.9760	Unknown	1	0.00	R042400028	< LOQ (0.0253 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	0.00	R042100019	< LOQ (0.0225 ug/mL)	<LOQ	NA
	RTS04200-Sera Blk-3	NA	0.9760	Unknown	1	0.00	R042400029	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100020	< LOQ (0.0225 ug/mL)		
	RTS04200-Sera Blk-4	NA	0.9760	Unknown	1	0.00	R042400033	< LOQ (0.0253 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	0.00	R042100024	< LOQ (0.0225 ug/mL)	<LOQ	NA
QC	RTS04200-MS-250-3	NA	NA	NA	1	207	R042100025	83%			NA	NA	NA	1	40.1	R042100025	16% *		
	RTS04200-MSD-250-3	NA	NA	NA	1	244	R042100026	99%	91%	17%	NA	NA	NA	1	45.9	R042100026	18% *	17%	13%
	RTS04200-MS-250-3	NA	NA	NA	1	NA	NA	NA			NA	NA	NA	1	78.8	D081500021	32% 2nd*		
	RTS04200-MSD-250-3	NA	NA	NA	1	NA	NA	NA	NA	NA	NA	NA	NA	1	90.2	D081500022	36% 2nd*	34%	13%
Group 8 Control 0.0 ppm	C9195/M	NA	0.9760	Unknown	1	0.00	R042400036	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100027	< LOQ (0.0225 ug/mL)		
	C9199/M	NA	0.9760	Unknown	1	0.00	R042400037	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100028	< LOQ (0.0225 ug/mL)		
	C9205/M	NA	0.9760	Unknown	1	0.00	R042400041	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100032	< LOQ (0.0225 ug/mL)		
	C9238/M	NA	0.9760	Unknown	1	0.00	R042400042	< LOQ (0.0253 ug/mL)		NA	NA	0.9770	0.8890	1	0.00	R042100033	< LOQ (0.0225 ug/mL)		NA
	C9246/M	NA	0.9760	Unknown	1	0.00	R042400043	< LOQ (0.0253 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	0.00	R042100034	< LOQ (0.0225 ug/mL)	<LOQ	NA
	C9234/F	NA	0.9760	Unknown	1	0.00	R042400044	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100035	< LOQ (0.0225 ug/mL)		
	C9236/F	NA	0.9760	Unknown	1	0.00	R042400045	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100036	< LOQ (0.0225 ug/mL)		
	C9237/F	NA	0.9760	Unknown	1	0.00	R042400049	< LOQ (0.0253 ug/mL)			NA	0.9770	0.8890	1	0.00	R042100040	< LOQ (0.0225 ug/mL)		
	C9240/F	NA	0.9760	Unknown	1	0.00	R042400050	< LOQ (0.0253 ug/mL)		NA	NA	0.9770	0.8890	1	0.00	R042100041	< LOQ (0.0225 ug/mL)		NA
	C9260/F	NA	0.9760	Unknown	1	0.00	R042400051	< LOQ (0.0253 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	0.00	R042100042	< LOQ (0.0225 ug/mL)	<LOQ	NA
Group 9 Low Dose 1.0 ppm	C9267/M	NA	0.9760	Unknown	1	92.3	R042100043	0.0946			NA	0.9770	0.8890	1	0.00	R042100043	< LOQ (0.0225 ug/mL)		
	C9274/M	NA	0.9760	Unknown	1	273	R042100044	0.279			NA	0.9770	0.8890	1	0.00	R042100044	< LOQ (0.0225 ug/mL)		
	C9287/M	NA	0.9760	Unknown	1	596	R042100048	0.610			NA	0.9770	0.8890	1	0.00	R042100048	< LOQ (0.0225 ug/mL)		
	C9288/M	NA	0.9760	Unknown	1	304	R042100049	0.311		65.3	NA	0.9770	0.8890	1	0.00	R042100049	< LOQ (0.0225 ug/mL)		NA
	C9320/M	NA	0.9760	Unknown	1	186	R042100050	0.191	0.297	0.194	NA	0.9770	0.8890	1	0.00	R042100050	< LOQ (0.0225 ug/mL)	<LOQ	NA
	C9240/F	NA	0.9760	Unknown	1	189	R042100051	0.194			NA	0.9770	0.8890	1	0.00	R042100051	< LOQ (0.0225 ug/mL)		
	C9241/F	NA	0.9760	Unknown	1	201	R042100052	0.206			NA	0.9770	0.8890	1	0.00	R042100052	< LOQ (0.0225 ug/mL)		
	C9418/F	NA	0.9760	Unknown	1	332	R042100056	0.340			NA	0.9770	0.8890	1	0.00	R042100056	< LOQ (0.0225 ug/mL)		
	C9423/F	NA	0.9760	Unknown	1	426	R042100057	0.436		40.3	NA	0.9770	0.8890	1	0.00	R042100057	< LOQ (0.0225 ug/mL)		NA
	C9438/F	NA	0.9760	Unknown	1	186	R042100058	0.191	0.273	0.110	NA	0.9770	0.8890	1	0.00	R042100058	< LOQ (0.0225 ug/mL)	<LOQ	NA

* Recoveries confirmed: LAC 09/03/00

A = Estimated concentration, above the calibration range 1

Date Entered/By:	05/12/00, 06/01/00, 08/22/00, 08/
Data Verified/ By:	03/20/01 hjh, 05/10/01 LAC, SJ
Purity Entered/Verified:	03/29/01 LAC/

04/21/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
04/24/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
08/15/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
08/28/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number/Test Substance:	T-6316 (EtFOSE OH)
Matrix:	Rat Serum
Method/Revision:	ETS 8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Ruby 100699
Instrument Software/Version:	Matalyzer 3.4
Filename:	See listing to the right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Date of Extraction/Analyst:	04/20/00 SAL/KJK
Date of Analysis/Analyst:	04/21/00, 04/24/00, 08/15/00, 08/28/00 IAS/MMB/HOJ/SAH
Date of Data Reduction/Analyst:	05/05/00, 04/25/00, 08/16/00, 08/30/00 MMB/IAS

Sample Data

WEEK 53 RAT SERA

Group Dose	Sample #	Surrogate Verified	M556 Old Purity Correction Factor (lot NB113047-80)	M556 Purity Correction Factor*	M556 Dilution Factor	M556 Conc. ug/mL	Filename	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	04200-H2O Blk-3	NA	0.9989	Unknown	1	0.67	R082800015	<LOQ (0.00494 ug/mL)		
	04200-H2O Blk-4	NA	0.9989	Unknown	1	0.34	R082800020	<LOQ (0.00494 ug/mL)	<LOQ	NA
Matrix Blk	RBS04200-Sera Blk-3	NA	0.9989	Unknown	1	0.88	R082800016	<LOQ (0.00494 ug/mL)		
	RBS04200-Sera Blk-4	NA	0.9989	Unknown	1	0.00	R082800021	<LOQ (0.00494 ug/mL)	<LOQ	NA
	RTS04200-Sera Blk-3	NA	0.9989	Unknown	1	0.00	R082800017	<LOQ (0.00494 ug/mL)		
	RTS04200-Sera Blk-4	NA	0.9989	Unknown	1	0.00	R082800022	<LOQ (0.00494 ug/mL)	<LOQ	NA
QC	RTS04200-MS-250-3	NA	NA	NA	1	268	R042400034	108%		
	RTS04200-MSD-250-3	NA	NA	NA	1	275	R042400035	110%	109%	2%
	RTS04200-MS-250-3	NA	NA	NA	1	NA	NA	NA		
	RTS04200-MSD-250-3	NA	NA	NA	1	NA	NA	NA	NA	NA
Group 8 Control 0.0 ppm	C92195M	NA	0.9989	Unknown	1	3.43	R082800025	<LOQ (0.00494 ug/mL)		
	C92199M	NA	0.9989	Unknown	1	0.00	R082800026	<LOQ (0.00494 ug/mL)		
	C92205M	NA	0.9989	Unknown	1	0.00	R082800027	<LOQ (0.00494 ug/mL)		
	C92216M	NA	0.9989	Unknown	1	0.00	R082800028	<LOQ (0.00494 ug/mL)		NA
	C92246M	NA	0.9989	Unknown	1	0.00	R082800029	<LOQ (0.00494 ug/mL)	<LOQ	NA
	C92341F	NA	0.9989	Unknown	1	1.63	R082800032	<LOQ (0.00494 ug/mL)		
	C92356F	NA	0.9989	Unknown	1	0.00	R082800033	<LOQ (0.00494 ug/mL)		
	C92372F	NA	0.9989	Unknown	1	0.00	R082800034	<LOQ (0.00494 ug/mL)		
	C92376F	NA	0.9989	Unknown	1	0.00	R082800035	<LOQ (0.00494 ug/mL)		NA
	C92396F	NA	0.9989	Unknown	1	0.00	R082800036	<LOQ (0.00494 ug/mL)	<LOQ	NA
Group 9 Low Dose 1.0 ppm	C92267M	NA	0.9989	Unknown	1	94.6	R042400052	0.0947		
	C92274M	NA	0.9989	Unknown	1	136	R042400053	0.136		
	C92287M	NA	0.9989	Unknown	1	162	R042400057	0.162		
	C92288M	NA	0.9989	Unknown	1	106	R042400058	0.106	24.2	NA
	C92320M	NA	0.9989	Unknown	1	98.9	R042400059	0.0990	0.120	0.0289
	C92406F	NA	0.9989	Unknown	1	207	R042400060	0.207		
	C92411F	NA	0.9989	Unknown	1	202	R042400061	0.202		1
	C92418F	NA	0.9989	Unknown	1	183	R042400065	0.183		
	C92422F	NA	0.9989	Unknown	1	311	R042400066	0.311	35.8	NA
	C92438F	NA	0.9989	Unknown	1	109	R042400067	0.109	0.202	0.0723

Group Dose	Sample #	Surrogate Verified	PFOSEA Old Purity Correction Factor (lot 529)	PFOSEA Purity Correction Factor	PFOSEA Dilution Factor	PFOSEA Conc. ng/mL	Filename	Concentration of PFOSEA ng/mL or % Rec	Mean PFOSEA ng/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	04200-H2O Blk-3	NA	0.9930	Unknown	1	0.00	R042100016	< LOQ (0.00978 ng/mL)		
	04200-H2O Blk-4	NA	0.9930	Unknown	1	0.00	R042100017	< LOQ (0.00978 ng/mL)	<LOQ	NA
Matrix Blk	RBS04200-Sera Blk-3	NA	0.9930	Unknown	1	0.00	R042100018	< LOQ (0.00978 ng/mL)		
	RBS04200-Sera Blk-4	NA	0.9930	Unknown	1	0.00	R042100019	< LOQ (0.00978 ng/mL)	<LOQ	NA
	RTS04200-Sera Blk-3	NA	0.9930	Unknown	1	0.00	R042100020	< LOQ (0.00978 ng/mL)		
	RTS04200-Sera Blk-4	NA	0.9930	Unknown	1	0.00	R042100024	< LOQ (0.00978 ng/mL)	<LOQ	NA
QC	RTS04200-MS-250-3	NA	NA	NA	1	89.2	R042100025	36% *		
	RTS04200-MSD-250-3	NA	NA	NA	1	119	R042100026	48% *	42%	28%
	RTS04200-MS-250-3	NA	NA	NA	1	106	D081500021	43% 2nd*		
	RTS04200-MSD-250-3	NA	NA	NA	1	133	D081500022	54% 2nd*	48%	22%
Group 8 Control 0.0 ppm	C92195M	NA	0.9930	Unknown	1	0.00	R042100027	< LOQ (0.00978 ng/mL)		
	C92199M	NA	0.9930	Unknown	1	0.00	R042100028	< LOQ (0.00978 ng/mL)		
	C92205M	NA	0.9930	Unknown	1	0.00	R042100032	< LOQ (0.00978 ng/mL)		
	C92216M	NA	0.9930	Unknown	1	0.00	R042100033	< LOQ (0.00978 ng/mL)		NA
	C92246M	NA	0.9930	Unknown	1	0.00	R042100034	< LOQ (0.00978 ng/mL)	<LOQ	NA
	C92341F	NA	0.9930	Unknown	1	0.00	R042100035	< LOQ (0.00978 ng/mL)		
	C92356F	NA	0.9930	Unknown	1	0.00	R042100036	< LOQ (0.00978 ng/mL)		
	C92372F	NA	0.9930	Unknown	1	0.00	R042100040	< LOQ (0.00978 ng/mL)		
	C92376F	NA	0.9930	Unknown	1	0.00	R042100041	< LOQ (0.00978 ng/mL)		NA
	C92396F	NA	0.9930	Unknown	1	0.00	R042100042	< LOQ (0.00978 ng/mL)	<LOQ	NA
Group 9 Low Dose 1.0 ppm	C92267M	NA	0.9930	Unknown	1	0.00	R042100043	< LOQ (0.00978 ng/mL)		
	C92274M	NA	0.9930	Unknown	1	0.00	R042100044	< LOQ (0.00978 ng/mL)		
	C92287M	NA	0.9930	Unknown	1	0.00	R042100048	< LOQ (0.00978 ng/mL)		
	C92288M	NA	0.9930	Unknown	1	0.00	R042100049	< LOQ (0.00978 ng/mL)		NA
	C92320M	NA	0.9930	Unknown	1	0.00	R042100050	< LOQ (0.00978 ng/mL)	<LOQ	NA
	C92406F	NA	0.9930	Unknown	1	0.00	R042100051	< LOQ (0.00978 ng/mL)		
	C92411F	NA	0.9930	Unknown	1	0.00	R042100052	< LOQ (0.00978 ng/mL)		
	C92418F	NA	0.9930	Unknown	1	0.00	R042100056	< LOQ (0.00978 ng/mL)		
	C92422F	NA	0.9930	Unknown	1	0.00	R042100057	< LOQ (0.00978 ng/mL)		NA
	C92438F	NA	0.9930	Unknown	1	0.00	R042100058	< LOQ (0.00978 ng/mL)	<LOQ	NA

* Recoveries confirmed. LAC 09/03/00

A = Estimated concentration, above the calibration range. 1

Date Entered/By: 05/12/00, 06/01/00, 08/22/00, 08/
Date Verified/By: 03/20/01 JJB, 05/10/01 LAC, 5/
Purity Entered/Verified: 03/29/01 LAC/

04/21/00	Initial LOQ ng/mL
	Corrected LOQ ng/mL
04/24/00	Initial LOQ ng/mL
	Corrected LOQ ng/mL
08/15/00	Initial LOQ ng/mL
	Corrected LOQ ng/mL
08/28/00	Initial LOQ ng/mL
	Corrected LOQ ng/mL

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = $C_8F_{17}SO_2N(H)CH_2COO^-$
PFOSEA = Perfluorooctane sulfonyl ethylamide

FACT-M-4.0

Sera Week 53

05/29/2001

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98 1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98 1%) N-Ethyl Perfluorooctanesulfonamido
Product Number(Test Substance):	T-6316 (EtFOSE-OH)	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum	Rat Serum
Method/Revision:	ETS-8-4 1 & ETS-8 5.1	ETS-8-4 1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799	Davey 070799
Instrument Software/Version:	MassLynx 3 4	MassLynx 3 4
Filename:	See Attachments	See Attachments
R-Squared Value:	See Attachments	See Attachments
Slope:	See Attachments	See Attachments
Y-Intercept:	See Attachments	See Attachments
Dates of Extraction/Analyst:	06/29/00 SAL/KJK	06/29/00 SAL/KJK
Dates of Analysis/Analyst:	07/06/00, 07/19/00, 07/24/00, 08/24/00 MMH/SAH	07/06/00, 07/19/00, 07/24/00, 08/24/00 MMH/SAH
Date of Data Reduction/Analyst:	07/07/00, 07/25/00 07/27/00, 08/28/00 MMH/HGJ	07/07/00, 07/25/00, 07/27/00, 08/28/00 MMH/HGJ

Sample Data

WEEK 105 RAT SERA

lot 171 | lot L-15709 | lot 98-0207-036-3

Group Dose	Sample #	Extraction Vol. mL	Surrogate Verified	PFOS Old Purity Correction Factor	PFOS Purity Correction Factor	PFOS Dilution Factor	PFOS Conc. ng/mL	Filename	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	PFOSA Old Purity Correction Factor	PFOSA Purity Correction Factor	PFOSA Dilution Factor	PFOSA Conc. ng/mL	Filename	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	PFOSA Old Purity Correction Factor	PFOSA Purity Correction Factor	PFOSAA Dilution Factor	PFOSAA Conc. ng/mL	Filename
Method Blk	06290-H2O Blk-5	1	NA	0 9949	0 8640	1	0 00	R072400007	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0.00	D070600007	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 16	R072400007
	06290-H2O Blk-6	1	NA	0 9949	0 8640	1	0 33	R072400021	<LOQ (0 00428 ug/mL)	<LOQ	NA	NA	0 9510	Unknown	1	0 00	D070600021	<LOQ (0 00516 ug/mL)	<LOQ	NA	NA	0 9760	Unknown	1	1 95	R072400021
Matrix Blk	RBS06290 Sera Blk 5	1	NA	0 9949	0 8640	1	0 39	R072400008	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0.00	D070600008	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 04	R072400008
	RBS06290 Sera Blk 6	1	NA	0 9949	0 8640	1	0 47	R072400022	<LOQ (0 00428 ug/mL)	<LOQ	NA	NA	0 9510	Unknown	1	0 00	D070600022	<LOQ (0 00516 ug/mL)	<LOQ	NA	NA	0 9760	Unknown	1	1 05	R072400022
Matrix Blk	RTS06290 Sera Blk 5	1	NA	0 9949	0 8640	1	3.23	R072400009	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0 00	D070600009	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 07	R072400009
	RTS06290 Sera Blk 6	1	NA	0 9949	0 8640	1	3 95	R072400023	<LOQ (0 00428 ug/mL)	<LOQ	NA	NA	0 9510	Unknown	1	0 00	D070600023	<LOQ (0 00516 ug/mL)	<LOQ	NA	NA	0 9760	Unknown	1	1 40	R072400023
QC	RTS06290-MS 250 ppb-5	1	NA	NA	NA	1	340	D070600024	135% *			NA	NA	NA	1	307	D070600024	124%			NA	NA	NA	1	342	D070600024
	RTS06290-MSD-250 ppb-5	1	NA	NA	NA	1	345	D070600025	137% *	136%	1%	NA	NA	NA	1	310	D070600025	126%	125%	1%	NA	NA	NA	1	347	D070600025
	RTS06290-MS-250 ppb-5	1	NA	NA	NA	1	343	R072400034	137% 2nd*			NA	NA	NA	NA	NA	NA	NA			NA	NA	NA	NA	NA	NA
	RTS06290-MSD-250 ppb-5	1	NA	NA	NA	1	365	R072400025	146% 2nd*	141%	6%	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 8 Control 0 0 ppm	C92197/M	1	NA	0 9949	0.8640	1	4.58	R072400029	<LOQ (0 00428 ug/mL)			NA	0.9510	Unknown	1	0.00	D070600028	<LOQ (0 00516 ug/mL)			NA	0.9760	Unknown	1	1 93	R072400029
	C9220/M	1	NA	0 9949	0 8640	1	3 01	R072400030	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0.00	D070600029	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 60	R072400030
	C92206/M	1	NA	0 9949	0 8640	1	3.39	R072400031	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0 00	D070600030	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 51	R072400031
	C9220/M	1	NA	0 9949	0 8640	1	3.37	R072400032	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0 00	D070600031	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 35	R072400032
	C92216/M	1	NA	0 9949	0 8640	1	27.8	R072400033	0 0242			NA	0 9510	Unknown	1	0 00	D070600032	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 75	R072400033
	C92222/M	1	NA	0 9949	0 8640	1	7.75	R072400037	0 00673			NA	0 9510	Unknown	1	0.00	D070600035	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	2 31	R072400037
	C92231/M	1	NA	0 9949	0 8640	1	6 06	R072400038	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0 00	D070600036	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 47	R072400038
	C92233/M	1	NA	0 9949	0 8640	1	2 41	R072400039	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0 00	D070600037	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 95	R072400039
	C92239/M	1	NA	0 9949	0 8640	1	70.1	R072400040	0 0608		132	NA	0 9510	Unknown	1	0 00	D070600038	<LOQ (0 00516 ug/mL)		NA	NA	0 9760	Unknown	1	1 40	R072400040
	C92252/M	1	NA	0 9949	0 8640	1	23.0	R072400041	0 0200	0 0137	0 0181	NA	0 9510	Unknown	1	0 00	D070600039	<LOQ (0 00516 ug/mL)	<LOQ	NA	NA	0 9760	Unknown	1	1 23	R072400041
	C92331/F	1	NA	0 9949	0 8640	1	41 2	R072400045	0 0358			NA	0 9510	Unknown	1	0 00	D070600042	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 62	R072400045
	C92334/F	1	NA	0 9949	0 8640	1	50.6	R072400046	0 0439			NA	0 9510	Unknown	1	0 00	D070600043	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 35	R072400046
	C92344/F	1	NA	0 9949	0 8640	1	53 9	R072400047	0 0468			NA	0 9510	Unknown	1	0 00	D070600044	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 39	R072400047
	C92354/F	1	NA	0 9949	0 8640	1	15 9	R072400048	0 0138			NA	0 9510	Unknown	1	0 00	D070600045	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 55	R072400048
	C92358/F	1	NA	0 9949	0 8640	1	4.43	R072400049	<LOQ (0 00428 ug/mL)			NA	0 9510	Unknown	1	0 00	D070600046	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 27	R072400049
	C92360/F	1	NA	0 9949	0 8640	1	23 6	R072400053	0 0205			NA	0 9510	Unknown	1	0 00	D070600049	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 64	R072400053
	C92362/F	1	NA	0 9949	0 8640	1	8 99	R072400054	0 00781			NA	0 9510	Unknown	1	0 00	D070600050	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 64	R072400054
	C92369/F	1	NA	0 9949	0 8640	1	58.5	R072400055	0 0503			NA	0 9510	Unknown	1	0 00	D070600051	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	1 75	R072400055
	C92374/F	1	NA	0 9949	0 8640	1	30.1	R072400056	0 0262		56.5	NA	0 9510	Unknown	1	0 00	D070600052	<LOQ (0 00516 ug/mL)		NA	NA	0 9760	Unknown	1	1 17	R072400056
	C92381/F	1	NA	0 9949	0 8640	1	27.8	R072400057	0 0241	0 0256	0 0145	NA	0 9510	Unknown	1	0 00	D070600053	<LOQ (0 00516 ug/mL)	<LOQ	NA	NA	0 9760	Unknown	1	1 29	R072400057
Group 9 Low Dose 1 0 ppm	C92261/M	1	NA	0 9949	0 8640	1	684	D070600056	0 594			NA	0 9510	Unknown	1	5 58	D070600056	0 00587			NA	0 9760	Unknown	1	247	D070600056
	C92266/M	1	NA	0 9949	0 8640	10	110	R072400061	0 953			NA	0 9510	Unknown	1	3 82	D070600057	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	121	D070600057
	C92270/M	1	NA	0 9949	0 8640	20	183	R072400062	3 17			NA	0 9510	Unknown	1	14 4	D070600058	0 0152			NA	0 9760	Unknown	1	640	D070600058
	C92278/M	1	NA	0 9949	0 8640	10	100	R072400063	0 868			NA	0 9510	Unknown	1	9 79	D070600059	0 0103			NA	0 9760	Unknown	1	362	D070600059
	C92281/M	1	NA	0 9949	0 8640	1	495	D070600060	0 430			NA	0 9510	Unknown	1	3 59	D070600060	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	196	D070600060
	C92284/M	1	NA	0 9949	0 8640	20	174	R072400064	3 01			NA	0 9510	Unknown	1	6 06	D070600063	0 00637			NA	0 9760	Unknown	1	243	D070600063
	C92309/M	1	NA	0 9949	0 8640	1	99 0	D070600064	0 0860			NA	0 9510	Unknown	1	2 05	D070600064	<LOQ (0 00516 ug/mL)			NA	0 9760	Unknown	1	134	D070600064
	C92311/M	1	NA	0 9949	0 8640	1	503	D070600065	0 437			NA	0 9510	Unknown	1	8 05	D070600065	0 00846			NA	0 9760	Unknown	1	225	D070600065
	C92318/M	1	NA	0 9949	0 8640	10	154	R072400065	1 33		97.8	NA	0 9510	Unknown	1	3 52	D070600066	<LOQ (0 00516 ug/mL)		45 8	NA	0 9760	Unknown	1	164	D070600066
	C92321/M	1	NA	0 9949	0 8640	1	387	D070600067	0 336	1.12	1.10	NA	0 9510	Unknown	1	1 41	D070600067	<LOQ (0 00516 ug/mL)	0.00720	0.00330	NA	0 9760	Unknown	1	131	D070600067
	C92410/F	1	NA	0 9949	0 8640	20	131	R072400069	2.27			NA	0 9510	Unknown	1	8.73	D070600070	0.00918			NA	0 9760	Unknown	1	161	D070600070
	C92416/F	1	NA	0 9949	0 8640	20	137	R072400070	2 38			NA	0 9510	Unknown	1	11 3	D070600071	0 0119			NA	0 9760	Unknown	1	505	D070600071
	C92419/F	1	NA	0 9949	0 8640	1	623	D070600072	0 541			NA	0 9510	Unknown	1	8 39	D070600072	0 00882			NA	0 9760	Unknown	1	138	D070600072
	C92421/F	1	NA	0 9949	0 8640	10	101	R072400071	0 879			NA	0 9510	Unknown	1	5 59	D070600073	0 00588			NA	0 9760	Unknown	1	255	D070600073
	C92430/F	1	NA	0 9949	0 8640	10	199	R072400072	1 73			NA	0 9510	Unknown	1	7 13	D070600074	0 00750			NA	0 9760	Unknown	1	183	D070600074
	C92431/F	1	NA	0 9949	0 8640	10	124	R072400073	1 08			NA	0 9510	Unknown	1	5 49	D070600077	0 00577			NA	0 9760	Unknown	1	175	D070600077
	C92434/F	1	NA	0 9949	0 8640	20	108	R072400077	1 88			NA	0 9510	Unknown	1	7 26	D070600078	0 00763			NA	0 9760	Unknown	1	121	D070600078
	C92434/F	1	NA	0 9949	0 8640	10	148	R072400078	1 28			NA	0 9510	Unknown	1	7.29	D070600079	0 00767			NA	0 9760	Unknown	1	203	D070600079
	C92451/F	1	NA	0 9949	0 8640	20	113	R072400079	1 96		39.1	NA	0 9510	Unknown	1	9 23	D070600080	0 00971		24.9	NA	0 9760	Unknown	1	555	D070600080
	C92452/F	1	NA	0 9949	0 8640	20	124	R072400080	2 15	1 61	0.631	NA	0 9510	Unknown	1	5.37	D070600081	0.00565	0.00797	0.00198	NA	0 9760	Unknown	1	231	D070600081

* High recoveries confirmed with 07/24/00 and 08/24/00 analysis LAC 08/21/00 and 09/03/00

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = $C_8F_{17}SO_2N(H)CH_2COO^-$
PFOSEA = Perfluorooctane sulfooyl ethylamide

Date Entered/By: 07/13/00, 08/15/00, 08/21/00, 09/03/00 LAC
Date Verified/ By: 03/20/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC

		PFOS	PFOSA	PFOSAA	EtFOSE	M556	PFOSEA
07/06/00	Initial LOQ ng/mL	49 3	4.91	49 1	4.92	4.93	4.90
	Corrected LOQ ng/mL	0.0428	0 00516	0 0503	0 00448	0 00494	0.00493
07/19/00	Initial LOQ ng/mL	4 93	NA	4.91	NA	24 8	NA
	Corrected LOQ ng/mL	0 00428	NA	0 00503	NA	0 0248	NA
07/24/00	Initial LOQ ng/mL	4.93	NA	4 91	NA	24 8	NA
	Corrected LOQ ng/mL	0.00428	NA	0 00503	NA	0.0248	NA
08/24/00	Initial LOQ ng/mL	9.76	NA	NA	NA	74 8	NA
	Corrected LOQ ng/mL	0.00848	NA	NA	NA	0.0248	NA

FACT-M 4 0

Covance# 6329-228

Study: nido Ethanol in Rats
Product Number(Test Substance):
Matrix:
Method/Revision:
Analytical Equipment System Number:
Instrument Software/Version:
Filename:
R-Squared Value:
Slope:
Y-Intercept:
Dates of Extraction/Analyst:
Dates of Analysis/Analyst:
Date of Data Reduction/Analyst:

Sample Data

WEEK 105 RAT SERA lot SD013

Group Dose	Sample #	Concentration of PFOSAA ug/mL or % Rec	Mean PFOSAA ug/mL	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	EtFOSE Old Purity Correction Factor	EtFOSE Purity Correction Factor	EtFOSE Dilution Factor	EtFOSE Conc. ug/mL	Filename	Concentration of EtFOSE ug/mL or % Rec	Mean EtFOSE ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	06290-H2O Blk-5	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.40	D070600007	<LOQ (0.00448 ug/mL)		
	06290-H2O Blk-6	<LOQ (0.00503 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	1.42	D070600021	<LOQ (0.00448 ug/mL)	<LOQ	NA
Matrix Blk	RBS06290-Sera Blk-5	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.00	D070600008	<LOQ (0.00448 ug/mL)		
	RBS06290-Sera Blk-6	<LOQ (0.00503 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	1.54	D070600022	<LOQ (0.00448 ug/mL)	<LOQ	NA
Matrix Blk	RTS06290-Sera Blk-5	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.37	D070600009	<LOQ (0.00448 ug/mL)		
	RTS06290-Sera Blk-6	<LOQ (0.00503 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	0.00	D070600023	<LOQ (0.00448 ug/mL)	<LOQ	NA
QC	RTS06290-MS-250 ppb-5	138%			NA	NA	NA	1	148	D070600024	60%		
	RTS06290-MSD-250 ppb-5	140%	139%	2%	NA	NA	NA	1	151	D070600025	61%	60%	2%
	RTS06290-MS-250 ppb-5	NA			NA	NA	NA	NA	NA	NA	NA		
	RTS06290-MSD-250 ppb-5	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 8 Control 0.0 ppm	C92197/M	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	1.28	D070600028	<LOQ (0.00448 ug/mL)		
	C92203/M	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.00	D070600029	<LOQ (0.00448 ug/mL)		
	C92208/M	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.95	D070600030	<LOQ (0.00448 ug/mL)		
	C92209/M	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.56	D070600031	<LOQ (0.00448 ug/mL)		
	C92218/M	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.15	D070600032	<LOQ (0.00448 ug/mL)		
	C92222/M	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.17	D070600035	<LOQ (0.00448 ug/mL)		
	C92231/M	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.41	D070600036	<LOQ (0.00448 ug/mL)		
	C92233/M	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.38	D070600037	<LOQ (0.00448 ug/mL)		
	C92239/M	<LOQ (0.00503 ug/mL)		NA	NA	0.9770	0.8890	1	0.12	D070600038	<LOQ (0.00448 ug/mL)		NA
	C92252/M	<LOQ (0.00503 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	0.97	D070600039	<LOQ (0.00448 ug/mL)	<LOQ	NA
	C92335/F	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.87	D070600042	<LOQ (0.00448 ug/mL)		
	C92334/F	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.77	D070600043	<LOQ (0.00448 ug/mL)		
	C92348/F	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.34	D070600044	<LOQ (0.00448 ug/mL)		
	C92354/F	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.36	D070600045	<LOQ (0.00448 ug/mL)		
	C92358/F	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.85	D070600046	<LOQ (0.00448 ug/mL)		
	C92360/F	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.57	D070600049	<LOQ (0.00448 ug/mL)		
	C92362/F	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.06	D070600050	<LOQ (0.00448 ug/mL)		
	C92369/F	<LOQ (0.00503 ug/mL)			NA	0.9770	0.8890	1	0.27	D070600051	<LOQ (0.00448 ug/mL)		
	C92374/F	<LOQ (0.00503 ug/mL)		NA	NA	0.9770	0.8890	1	0.25	D070600052	<LOQ (0.00448 ug/mL)		NA
	C92381/F	<LOQ (0.00503 ug/mL)	<LOQ	NA	NA	0.9770	0.8890	1	0.24	D070600053	<LOQ (0.00448 ug/mL)	<LOQ	NA
Group 9 Low Dose 1.0 ppm	C92261/M	0.253			NA	0.9770	0.8890	1	0.92	D070600056	<LOQ (0.00448 ug/mL)		
	C92266/M	0.124			NA	0.9770	0.8890	1	0.54	D070600057	<LOQ (0.00448 ug/mL)		
	C92270/M	0.656			NA	0.9770	0.8890	1	0.79	D070600058	<LOQ (0.00448 ug/mL)		
	C92278/M	0.371			NA	0.9770	0.8890	1	2.57	D070600059	<LOQ (0.00448 ug/mL)		
	C92281/M	0.201			NA	0.9770	0.8890	1	0.15	D070600060	<LOQ (0.00448 ug/mL)		
	C92285/M	0.249			NA	0.9770	0.8890	1	1.32	D070600063	<LOQ (0.00448 ug/mL)		
	C92309/M	0.138			NA	0.9770	0.8890	1	2.19	D070600064	<LOQ (0.00448 ug/mL)		
	C92311/M	0.231			NA	0.9770	0.8890	1	1.37	D070600065	<LOQ (0.00448 ug/mL)		
	C92318/M	0.168		63.4	NA	0.9770	0.8890	1	0.08	D070600066	<LOQ (0.00448 ug/mL)		NA
	C92321/M	0.135	0.232	0.160	NA	0.9770	0.8890	1	0.06	D070600067	<LOQ (0.00448 ug/mL)	<LOQ	NA
	C92410/F	0.165			NA	0.9770	0.8890	1	0.72	D070600070	<LOQ (0.00448 ug/mL)		
	C92416/F	0.517			NA	0.9770	0.8890	1	0.63	D070600071	<LOQ (0.00448 ug/mL)		
	C92419/F	0.142			NA	0.9770	0.8890	1	0.61	D070600072	<LOQ (0.00448 ug/mL)		
	C92423/F	0.261			NA	0.9770	0.8890	1	0.56	D070600073	<LOQ (0.00448 ug/mL)		
	C92430/F	0.187			NA	0.9770	0.8890	1	0.96	D070600074	<LOQ (0.00448 ug/mL)		
	C92431/F	0.179			NA	0.9770	0.8890	1	1.03	D070600077	<LOQ (0.00448 ug/mL)		
	C92439/F	0.123			NA	0.9770	0.8890	1	1.00	D070600078	<LOQ (0.00448 ug/mL)		
	C92443/F	0.208			NA	0.9770	0.8890	1	0.26	D070600079	<LOQ (0.00448 ug/mL)		
	C92451/F	0.569		60.2	NA	0.9770	0.8890	1	0.00	D070600080	<LOQ (0.00448 ug/mL)		NA
	C92452/F	0.236	0.259	0.156	NA	0.9770	0.8890	1	0.20	D070600081	<LOQ (0.00448 ug/mL)	<LOQ	NA

* High recoveries confirmed with 07/24/00 and 08/24/00 ana

Date Entered/By: 07/13/00, 08/15/00, 08/21/00, 09/0
Date Verified/ By: 03/20/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/

07/06/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
07/19/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
07/24/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
08/24/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98/1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799
Instrument Software/Version:	MassLynx 3.4
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	06/29/00 SAL/KJK
Dates of Analysis/Analyst:	07/06/00, 07/19/00, 07/24/00, 08/24/00 MMH/SAH
Date of Data Reduction/Analyst:	07/07/00, 07/25/00, 07/27/00, 08/28/00 MMH/HOJ

Sample Data

WEEK 105 RAT SERA

ka NB110347-80 ka 529

Group Dose	Sample #	Surrogate Verified	M556 Old Purity Correction Factor	M556 Purity Correction Factor	M556 Dilution Factor	M556 Conc. ug/mL	Filename	Concentration of M556 ug/mL or % Rec	Mean M556 ug/mL	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	PFOSEA Old Purity Correction Factor	PFOSEA Purity Correction Factor	PFOSEA Dilution Factor	PFOSEA Conc. ug/mL	Filename	Concentration of PFOSEA ug/mL or % Rec	Mean PFOSEA ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	06290-H2O Blk-5	NA	0.9989	Unknown	1	0.00	A082400005	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.00	D070600007	<LOQ (0.00493 ug/mL)		
	06290-H2O Blk-6	NA	0.9989	Unknown	1	0.00	A082400019	<LOQ (0.0248 ug/mL)	<LOQ	NA	NA	0.9930	Unknown	1	2.19	D070600021	<LOQ (0.00493 ug/mL)	<LOQ	NA
Matrix Blk	RBS06290-Sera Blk-5	NA	0.9989	Unknown	1	0.00	A082400006	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	1.09	D070600008	<LOQ (0.00493 ug/mL)		
	RBS06290-Sera Blk-6	NA	0.9989	Unknown	1	0.00	A082400020	<LOQ (0.0248 ug/mL)	<LOQ	NA	NA	0.9930	Unknown	1	1.89	D070600022	<LOQ (0.00493 ug/mL)	<LOQ	NA
Matrix Blk	RTS06290-Sera Blk-5	NA	0.9989	Unknown	1	0.00	A082400007	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.85	D070600009	<LOQ (0.00493 ug/mL)		
	RTS06290-Sera Blk-6	NA	0.9989	Unknown	1	0.00	A082400021	<LOQ (0.0248 ug/mL)	<LOQ	NA	NA	0.9930	Unknown	1	1.03	D070600023	<LOQ (0.00493 ug/mL)	<LOQ	NA
QC	RTS06290-MS-250 ppb-5	NA	NA	NA	1	431	A071900024	134% *			NA	NA	NA	1	222	D070600024	90%		
	RTS06290-MSD-250 ppb-5	NA	NA	NA	1	412	A071900025	166% *	170%	5%	NA	NA	NA	1	223	D070600025	90%	90%	1%
	RTS06290-MS-250 ppb-5	NA	NA	NA	1	381	A082400022	154% 2nd*			NA	NA	NA	NA	NA	NA	NA		
	RTS06290-MSD-250 ppb-5	NA	NA	NA	1	378	A082400023	153% 2nd*	153%	1%	NA	NA	NA	NA	NA	NA	NA	NA	NA
Group 8 Control 0.0 ppm	C92197/M	NA	0.9989	Unknown	1	0.00	A082400026	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.00	D070600028	<LOQ (0.00493 ug/mL)		
	C92203/M	NA	0.9989	Unknown	1	0.00	A082400027	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.00	D070600029	<LOQ (0.00493 ug/mL)		
	C92208/M	NA	0.9989	Unknown	1	0.00	A082400028	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.82	D070600030	<LOQ (0.00493 ug/mL)		
	C92209/M	NA	0.9989	Unknown	1	0.00	A082400029	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	1.29	D070600031	<LOQ (0.00493 ug/mL)		
	C92218/M	NA	0.9989	Unknown	1	0.00	A082400030	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.98	D070600032	<LOQ (0.00493 ug/mL)		
	C92222/M	NA	0.9989	Unknown	1	0.00	A082400033	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	1.20	D070600035	<LOQ (0.00493 ug/mL)		
	C92231/M	NA	0.9989	Unknown	1	0.00	A082400034	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	1.07	D070600036	<LOQ (0.00493 ug/mL)		
	C92233/M	NA	0.9989	Unknown	1	0.00	A082400035	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.00	D070600037	<LOQ (0.00493 ug/mL)		
	C92239/M	NA	0.9989	Unknown	1	0.00	A082400036	<LOQ (0.0248 ug/mL)		NA	NA	0.9930	Unknown	1	0.00	D070600038	<LOQ (0.00493 ug/mL)		NA
	C92252/M	NA	0.9989	Unknown	1	0.00	A082400037	<LOQ (0.0248 ug/mL)	<LOQ	NA	NA	0.9930	Unknown	1	1.42	D070600039	<LOQ (0.00493 ug/mL)	<LOQ	NA
	C92333/F	NA	0.9989	Unknown	1	0.00	A082400040	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.97	D070600042	<LOQ (0.00493 ug/mL)		
	C92334/F	NA	0.9989	Unknown	1	0.00	A082400041	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.00	D070600043	<LOQ (0.00493 ug/mL)		
	C92348/F	NA	0.9989	Unknown	1	0.00	A082400042	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.84	D070600044	<LOQ (0.00493 ug/mL)		
	C92354/F	NA	0.9989	Unknown	1	0.00	A082400043	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	1.09	D070600045	<LOQ (0.00493 ug/mL)		
	C92358/F	NA	0.9989	Unknown	1	0.00	A082400044	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.00	D070600046	<LOQ (0.00493 ug/mL)		
	C92360/F	NA	0.9989	Unknown	1	0.00	A082400047	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.98	D070600049	<LOQ (0.00493 ug/mL)		
	C92362/F	NA	0.9989	Unknown	1	0.00	A082400048	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.82	D070600050	<LOQ (0.00493 ug/mL)		
	C92366/F	NA	0.9989	Unknown	1	0.00	A082400049	<LOQ (0.0248 ug/mL)			NA	0.9930	Unknown	1	0.00	D070600051	<LOQ (0.00493 ug/mL)		
	C92374/F	NA	0.9989	Unknown	1	0.00	A082400050	<LOQ (0.0248 ug/mL)		NA	NA	0.9930	Unknown	1	1.08	D070600052	<LOQ (0.00493 ug/mL)		NA
	C92381/F	NA	0.9989	Unknown	1	0.00	A082400051	<LOQ (0.0248 ug/mL)	<LOQ	NA	NA	0.9930	Unknown	1	1.14	D070600053	<LOQ (0.00493 ug/mL)	<LOQ	NA
Group 9 Low Dose 1.0 ppm	C92261/M	NA	0.9989	Unknown	1	44.2	A071900076	0.0443			NA	0.9930	Unknown	1	1.74	D070600056	<LOQ (0.00493 ug/mL)		
	C92266/M	NA	0.9989	Unknown	1	53.1	A071900077	0.0532			NA	0.9930	Unknown	1	0.83	D070600057	<LOQ (0.00493 ug/mL)		
	C92270/M	NA	0.9989	Unknown	1	140	A071900078	0.140			NA	0.9930	Unknown	1	1.22	D070600058	<LOQ (0.00493 ug/mL)		
	C92278/M	NA	0.9989	Unknown	1	96.4	A071900079	0.0965			NA	0.9930	Unknown	1	1.37	D070600059	<LOQ (0.00493 ug/mL)		
	C92281/M	NA	0.9989	Unknown	1	21.1	A082400054	0.0211			NA	0.9930	Unknown	1	1.06	D070600060	<LOQ (0.00493 ug/mL)		
	C92283/M	NA	0.9989	Unknown	1	129	A071900083	0.129			NA	0.9930	Unknown	1	1.39	D070600063	<LOQ (0.00493 ug/mL)		
	C92309/M	NA	0.9989	Unknown	1	15.6	A082400055	0.0156			NA	0.9930	Unknown	1	2.43	D070600064	<LOQ (0.00493 ug/mL)		
	C92311/M	NA	0.9989	Unknown	1	42.4	A071900085	0.0425			NA	0.9930	Unknown	1	3.13	D070600065	<LOQ (0.00493 ug/mL)		
	C92318/M	NA	0.9989	Unknown	1	77.6	A071900086	0.0777		68.9	NA	0.9930	Unknown	1	1.04	D070600066	<LOQ (0.00493 ug/mL)		NA
	C92321/M	NA	0.9989	Unknown	1	25.7	A071900087	0.0257	0.0645	0.0445	NA	0.9930	Unknown	1	1.17	D070600067	<LOQ (0.00493 ug/mL)	<LOQ	NA
	C92410/F	NA	0.9989	Unknown	1	124	A071900090	0.125			NA	0.9930	Unknown	1	0.00	D070600070	<LOQ (0.00493 ug/mL)		
	C92416/F	NA	0.9989	Unknown	1	97.4	A071900091	0.0975			NA	0.9930	Unknown	1	1.45	D070600071	<LOQ (0.00493 ug/mL)		
	C92419/F	NA	0.9989	Unknown	1	93.4	A071900092	0.0935			NA	0.9930	Unknown	1	0.96	D070600072	<LOQ (0.00493 ug/mL)		
	C92423/F	NA	0.9989	Unknown	1	106	A071900093	0.106			NA	0.9930	Unknown	1	0.96	D070600073	<LOQ (0.00493 ug/mL)		
	C92430/F	NA	0.9989	Unknown	1	88.5	A071900094	0.0886			NA	0.9930	Unknown	1	0.00	D070600074	<LOQ (0.00493 ug/mL)		
	C92431/F	NA	0.9989	Unknown	1	43.1	A071900097	0.0431			NA	0.9930	Unknown	1	1.20	D070600077	<LOQ (0.00493 ug/mL)		
	C92439/F	NA	0.9989	Unknown	1	59.2	A071900098	0.0592			NA	0.9930	Unknown	1	1.23	D070600078	<LOQ (0.00493 ug/mL)		
	C92443/F	NA	0.9989	Unknown	1	56.2	A071900099	0.0563			NA	0.9930	Unknown	1	1.17	D070600079	<LOQ (0.00493 ug/mL)		
	C92451/F	NA	0.9989	Unknown	1	130	A071900100	0.130		33.8	NA	0.9930	Unknown	1	1.15	D070600080	<LOQ (0.00493 ug/mL)		NA
	C92452/F	NA	0.9989	Unknown	1	69.1	A071900101	0.0692	0.0868	0.0294	NA	0.9930	Unknown	1	1.00	D070600081	<LOQ (0.00493 ug/mL)	<LOQ	NA

* High recoveries confirmed with 07/24/00 and 08/24/00 analysis.

Date Entered/By: 07/13/00, 08/15/00, 08/21/00, 09/0
Date Verified/ By: 03/20/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
M556 = C8F17SO2N(H)CH2COO)
PFOSEA = Perfluorooctane sulfonyl ethylamide

07/06/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
07/19/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
07/24/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL
08/24/00	Initial LOQ ug/mL
	Corrected LOQ ug/mL

Study:	TOX003, 104 Week Dietary Carcinogenicity
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Serum
Method/Revision:	ETS-8-4.1 & ETS-8-5.1
Analytical Equipment System Number:	Davey 070799
Instrument Software/Version:	MassLynx 3.4
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	06/29/00 SAL/KJK
Dates of Analysis/Analyst:	07/06/00, 07/19/00, 07/24/00, 08/24/00 MM
Date of Data Reduction/Analyst:	07/07/00, 07/25/00, 07/27/00, 08/28/00 MM

Sample Data

WEEK 105 RAT SERA

Group Dose	Sample #	PFOS Conc. ug/mL	Concentration of PFOS ug/mL or % Rec
Method Blk	06290-H2O Blk-5	0.00	<LOQ (0.00428 ug/mL)
	06290-H2O Blk-6	0.33	<LOQ (0.00428 ug/mL)
Matrix Blk	RBS06290-Sera Blk-5	0.39	<LOQ (0.00428 ug/mL)
	RBS06290-Sera Blk-6	0.47	<LOQ (0.00428 ug/mL)
Matrix Blk	RTS06290-Sera Blk-5	3.23	<LOQ (0.00428 ug/mL)
	RTS06290-Sera Blk-6	3.95	<LOQ (0.00428 ug/mL)
QC	RTS06290-MS-250 ppb-5	340	135% *
	RTS06290-MSD-250 ppb-5	345	137% *
	RTS06290-MS-250 ppb-5	343	137% 2nd*
	RTS06290-MSD-250 ppb-5	365	146% 2nd*
Group 8 Control 0.0 ppm	C92197/M	4.58	<LOQ (0.00428 ug/mL)
	C92203/M	3.01	<LOQ (0.00428 ug/mL)
	C92208/M	3.39	<LOQ (0.00428 ug/mL)
	C92209/M	3.37	<LOQ (0.00428 ug/mL)
	C92218/M	27.8	0.0242
	C92222/M	7.75	0.00673
	C92231/M	4.06	<LOQ (0.00428 ug/mL)
	C92233/M	2.41	<LOQ (0.00428 ug/mL)
	C92239/M	70.1	0.0608
	C92252/M	23.0	0.0260
	C92333/F	41.2	0.0358
	C92334/F	50.6	0.0439
	C92348/F	53.9	0.0468
	C92354/F	15.9	0.0138
	C92358/F	4.43	<LOQ (0.00428 ug/mL)
	C92360/F	23.6	0.0205
	C92362/F	8.99	0.00781
	C92366/F	38.3	0.0333
	C92374/F	30.1	0.0262
	C92381/F	27.8	0.0241
Group 9 Low Dose 1.0 ppm	C92261/M	684	0.596
	C92266/M	110	0.953
	C92270/M	183	1.17
	C92278/M	100	0.87
	C92281/M	495	0.430
	C92283/M	174	3.01
	C92309/M	99.0	0.0860
	C92311/M	503	0.437
	C92318/M	154	1.33
	C92321/M	387	0.336
	C92410/F	131	2.27
	C92416/F	137	2.38
	C92419/F	623	0.541
	C92423/F	101	0.88
	C92430/F	199	1.73
	C92431/F	124	1.08
	C92439/F	108	1.88
	C92443/F	148	1.28
	C92451/F	113	1.96
	C92452/F	124	2.15

* High recoveries confirmed with 07/24/00 and 08/24/00 analysis. LAC 08/21/00 and 09/03/00

Date Entered/By: 07/13/00, 08/15/00, 08/21/00, 09/03/00 LAC
Date Verified/ By: 03/20/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver	Filename:	See Below
Method/Revision:	FACT-M-1.0 & FACT-M-2.0, ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Madeline 041098, Amelia 062498, Soup020199	Slope:	See Attachments
Instrument Software/Version:	MassLynx 3.1	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	6/09/98 RWW		
Date of Analysis/Analyst:	6/26/98, 6/30/98, 10/13/99 MEE/JJ/MMH		
Date of Data Reduction/Analyst:	02/08/00, 02/09/00, 06/08/00, 05/04/01 MMH/IAS		

Sample Data

WEEK 4 RAT LIVER REWORK lot 193

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor	PFOS Conc. ug/g	PFOS Dilution Factor	PFOS Calc. Conc. ug/g	Filename	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	RBL06098-H2O Blk-1	1.0000	NA	0.9275	Unknown	17.2	1	16.0	M062698002	< LOQ (0.0552 ug/g)		
	RBL06098-H2O Blk-2	1.0000	NA	0.9275	Unknown	0.73	1	0.68	A06309802	< LOQ (0.0552 ug/g)	<LOQ	NA
Matrix Blk	RBL06098-Lvr Blk-1	1.0080	NA	0.9275	Unknown	14.5	1	13.3	M062698003	< LOQ (0.0552 ug/g)		
	RBL06098-Lvr Blk-2	1.0080	NA	0.9275	Unknown	5.95	1	5.47	A06309803	< LOQ (0.0552 ug/g)	<LOQ	NA
QC - 100 ppb	C92196M-MS	1.0106	NA	NA	NA	334	1	79.9	M062698040	67%		
	C92196M-MSD	1.0106	NA	NA	NA	368	1	113	M062698041	95%	81%	35%
QC - 100 ppb	C92196M-MS	1.0106	NA	NA	NA	413	1	158	A06309840	132% 2nd		
	C92196M-MSD	1.0106	NA	NA	NA	456	1	200	A06309841	168% 2nd	150%	24%
QC - 100 ppb	C92196M-MS	1.0106	NA	NA	NA	NA	NA	NA	NA	NA 3rd		
	C92196M-MSD	1.0106	NA	NA	NA	NA	NA	NA	NA	NA 3rd	NA	NA
QC - 500 ppb	C92192M-MS	1.0052	NA	NA	NA	965	1	608	S101399046	102%		
	C92192M-MSD	1.0052	NA	NA	NA	1067	1	710	S101399047	119%	110%	15%
QC - 500 ppb	C92192M-MS	1.0052	NA	NA	NA	NA	NA	NA	NA	NA 2nd		
	C92192M-MSD	1.0052	NA	NA	NA	NA	NA	NA	NA	NA 2nd	NA	NA
Group 8	C92192M	1.0052	NA	0.9275	Unknown	353	1	326	M062698012	0.326		
Control	C92196M	1.0106	NA	0.9275	Unknown	254	1	233	M062698013	0.233		
0.0 ppm	C92207M	1.0133	NA	0.9275	Unknown	173	1	158	M062698014	0.158		
	C92216M	1.0029	NA	0.9275	Unknown	155	1	144	M062698015	0.144		41.6
	C92259M	1.0334	NA	0.9275	Unknown	142	1	127	M062698016	0.127	0.198	0.0822
	C92332F	1.0267	NA	0.9275	Unknown	85.8	1	77.5	M062698019	0.0775		
	C92352F	1.0512	NA	0.9275	Unknown	85.9	1	75.8	M062698020	0.0758		
	C92355F	1.0140	NA	0.9275	Unknown	151	1	138	M062698021	0.138		
	C92366F	1.0217	NA	0.9275	Unknown	53.0	1	48.1	M062698022	< LOQ (0.0552 ug/g)		35.4
	C92373F	1.0285	NA	0.9275	Unknown	109	1	98.4	M062698023	0.0984	0.0891	0.0315
Group 9	C92264M	1.0069	NA	0.9275	Unknown	147	50	6788	A06309812	6.79		
Low Dose	C92280M	1.0390	NA	0.9275	Unknown	160	50	7152	A06309813	7.15		
1.0 ppm	C92284M	1.0242	NA	0.9275	Unknown	154	50	6978	A06309814	6.98		
	C92300M	1.0230	NA	0.9275	Unknown	126	50	5706	A06309815	5.71		9.62
	C92329M	1.0315	NA	0.9275	Unknown	134	50	6034	A06309816	6.03	6.53	0.628
	C92402F	1.0435	NA	0.9275	Unknown	797	5	3541	A06309833	3.54		
	C92435F	1.0030	NA	0.9275	Unknown	760	5	3513	A06309834	3.51		
	C92463F	1.0376	NA	0.9275	Unknown	858	5	3835	A06309835	3.84		
	C92467F	1.0071	NA	0.9275	Unknown	360	5	1658	A06309836	1.66		31.5
	C92470F	1.0170	NA	0.9275	Unknown	509	5	2322	A06309837	2.32	2.97	0.937

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

Date Entered/By: 04/17/00, 06/20/00 MMH/LAC, 05/08/01 mmh
Date Verified/ By: 03/22/01 boj, 04/03/01 kjh, 05/10/01 LAC 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC / 04/03/01 KJH

AMDT# 050598.2
Covance# 6329-228

Study: TOX003. 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0, ETS-8-7.0
Analytical Equipment System Number: Madeline 041098, Amelia 062498, Soup020199
Instrument Software/Version: MassLynx 3.1
Date of Extraction/Analyst: 6/09/98 RWW
Date of Analysis/Analyst: 6/26/98, 6/30/98, 10/13/99 MEE/JJ/MMH
Date of Data Reduction/Analyst: 02/08/00, 02/09/00, 06/08/00, 05/04/01 MMH/IAS

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 4 RAT LIVER REWORK

Group Dose	Sample #	PFOSA Purity Correction Factor	PFOSA Conc. ng/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ng/g	Filename	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	PFOSAA Old Purity Correction Factor	PFOSAA Purity Correction Factor	PFOSAA Conc. ng/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ng/g	Filename	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD
	lot L-2353									lot 617								
Method Blk	RBL06098-H2O Blk-1	Unknown	0.00	1	0.00	M62698002	< LOQ (0.0121 ug/g)			0.5382	Unknown	0.00	1	0.00	M062698002	< LOQ (0.0341 ug/g)		
	RBL06098-H2O Blk-2	Unknown	0.00	1	0.00	A06309802	< LOQ (0.0121 ug/g)	<LOQ	NA	0.5382	Unknown	0.00	1	0.00	A06309802	< LOQ (0.0341 ug/g)	<LOQ	NA
Matrix Blk	RBL06098-Lvr Blk-1	Unknown	0.00	1	0.00	M62698003	< LOQ (0.0121 ug/g)			0.5382	Unknown	0.00	1	0.00	M062698003	< LOQ (0.0341 ug/g)		
	RBL06098-Lvr Blk-2	Unknown	1.27	1	1.26	A06309803	< LOQ (0.0121 ug/g)	<LOQ	NA	0.5382	Unknown	0.00	1	0.00	A06309803	< LOQ (0.0341 ug/g)	<LOQ	NA
QC - 100 ppb	C92196M-MS	NA	104	1	103	M62698040	85%			NA	NA	68.1	1	67.4	A06309840	53%		
	C92196M-MSD	NA	101	1	100	M62698041	83%	84%	3%	NA	NA	158	1	156	A06309841	123%	88%	79%
QC - 100 ppb	C92196M-MS	NA	107	1	106	A06309840	88% 2nd			NA	NA	227	1	225	S101399054	177% 2nd		
	C92196M-MSD	NA	124	1	122	A06309841	101% 2nd	94%	14%	NA	NA	254	1	251	S101399055	198% 2nd	188%	11%
QC - 100 ppb	C92196M-MS	NA	84.4	1	84.4	S101399054	70% 3rd			NA	NA	NA	NA	NA	NA	NA 3rd		
	C92196M-MSD	NA	68.7	1	68.0	S101399055	56% 3rd	63%	21%	NA	NA	NA	NA	NA	NA	NA 3rd	NA	NA
QC - 500 ppb	C92192M-MS	NA	382	1	380	S101399046	63%			NA	NA	647	1	644	S101399046	101%		
	C92192M-MSD	NA	352	1	350	S101399047	58%	60%	8%	NA	NA	763	1	759	S101399047	119%	110%	16%
QC - 500 ppb	C92192M-MS	NA	NA	NA	NA	NA	NA 2nd			NA	NA	NA	NA	NA	NA	NA 2nd		
	C92192M-MSD	NA	NA	NA	NA	NA	NA 2nd	NA	NA	NA	NA	NA	NA	NA	NA	NA 2nd	NA	NA
Group 8 Control 0.0 ppm	C92192M	Unknown	0.00	1	0.00	M62698012	< LOQ (0.0121 ug/g)			0.5382	Unknown	0.00	1	0.00	M62698012	< LOQ (0.0341 ug/g)		
	C92196M	Unknown	0.00	1	0.00	M62698013	< LOQ (0.0121 ug/g)			0.5382	Unknown	0.00	1	0.00	M62698013	< LOQ (0.0341 ug/g)		
	C92207M	Unknown	0.00	1	0.00	M62698014	< LOQ (0.0121 ug/g)			0.5382	Unknown	0.00	1	0.00	M62698014	< LOQ (0.0341 ug/g)		
	C92216M	Unknown	0.00	1	0.00	M62698015	< LOQ (0.0121 ug/g)		NA	0.5382	Unknown	0.00	1	0.00	M62698015	< LOQ (0.0341 ug/g)		NA
	C92259M	Unknown	0.00	1	0.00	M62698016	< LOQ (0.0121 ug/g)	<LOQ	NA	0.5382	Unknown	0.00	1	0.00	M62698016	< LOQ (0.0341 ug/g)	<LOQ	NA
	C92332F	Unknown	0.00	1	0.00	M62698019	< LOQ (0.0121 ug/g)			0.5382	Unknown	0.00	1	0.00	M62698019	< LOQ (0.0341 ug/g)		
	C92352F	Unknown	5.06	1	4.81	M62698020	< LOQ (0.0121 ug/g)			0.5382	Unknown	0.00	1	0.00	M62698020	< LOQ (0.0341 ug/g)		
	C92355F	Unknown	5.24	1	5.17	M62698021	< LOQ (0.0121 ug/g)			0.5382	Unknown	0.00	1	0.00	M62698021	< LOQ (0.0341 ug/g)		
	C92366F	Unknown	0.00	1	0.00	M62698022	< LOQ (0.0121 ug/g)		NA	0.5382	Unknown	0.00	1	0.00	M62698022	< LOQ (0.0341 ug/g)		NA
	C92373F	Unknown	0.00	1	0.00	M62698023	< LOQ (0.0121 ug/g)	<LOQ	NA	0.5382	Unknown	0.00	1	0.00	M62698023	< LOQ (0.0341 ug/g)	<LOQ	NA
Group 9 Low Dose 1.0 ppm	C92264M	Unknown	194	1	192	M62698026	0.192			0.5382	Unknown	160	5	428	M06309826	0.428		
	C92280M	Unknown	244	1	234	M62698027	0.234			0.5382	Unknown	132	5	342	M06309827	0.342		
	C92284M	Unknown	161	1	158	M62698028	0.158			0.5382	Unknown	225	5	590	M06309828	0.590		
	C92300M	Unknown	139	1	136	M62698029	0.136		21.5	0.5382	Unknown	111	5	293	M06309829	0.293		89.8
	C92329M	Unknown	220	1	214	M62698030	0.214	0.187	0.0401	0.5382	Unknown	675	5	1760	M06309830	1.76	0.683	0.613
	C92402F	Unknown	227	1	217	M62698033	0.217			0.5382	Unknown	248	5	641	M06309833	0.641		
	C92435F	Unknown	392	1	391	M62698034	0.391			0.5382	Unknown	229	5	613	M06309834	0.613		
	C92463F	Unknown	200	1	193	M62698035	0.193			0.5382	Unknown	172	5	446	M06309835	0.446		
	C92467F	Unknown	203	1	201	M62698036	0.201		47.2	0.5382	Unknown	133	5	355	M06309836	0.355		33.2
	C92470F	Unknown	107	1	105	M62698037	0.105	0.222	0.104	0.5382	Unknown	324	5	856	M06309837	0.856	0.582	0.193

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

Date Entered/By: 04/17/00, 06/20/00 MMH/LAC, 05/08/01 mmh
Date Verified/ By: 03/22/01 hoj, 04/03/01 kjh, 05/10/01 LAC 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC / 04/03/01 KJH

Date Entered/By: 04/17/00, 06/20/00 MMH/LAC, 05/08/01 mmh
Date Verified/ By: 03/22/01 hoj, 04/03/01 kjh, 05/10/01 LAC 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC / 04/03/01 KJH

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Liver
Method/Revision:	FACT-M-1.0 & FACT-M-2.0, ETS-8-7.0
Analytical Equipment System Number:	Madeline 041098, Amelia 062498, Soup020199
Instrument Software/Version:	MassLynx 3.1
Date of Extraction/Analyst:	6/09/98 RWW
Date of Analysis/Analyst:	6/26/98, 6/30/98, 10/13/99 MEE/JJ/MMH
Date of Data Reduction/Analyst:	02/08/00, 02/09/00, 06/08/00, 05/04/01 MMH/IAS

Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments

Sample Data

WEEK 4 RAT LIVER REWORK lot 936

Group Dose	Sample #	EtFOSE Purity Correction Factor	EtFOSE Conc. ug/g	EtFOSE Dilution Factor	EtFOSE Calc. Conc. ug/g	Filename	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	RBL06098-H2O Blk-1	Unknown	NR	1	NR	NA	NR		
	RBL06098-H2O Blk-2	Unknown	NR	1	NR	NA	NR	NR	NR
Matrix Blk	RBL06098-Lvr Blk-1	Unknown	NR	1	NR	NA	NR		
	RBL06098-Lvr Blk-2	Unknown	NR	1	NR	NA	NR	NR	NR
QC - 100 ppb	C92196M-MS	NA	NR	1	NR	NA	NR		
	C92196M-MSD	NA	NR	1	NR	NA	NR	NR	NR
QC - 100 ppb	C92196M-MS	NA	NR	1	NR	NA	NR		
	C92196M-MSD	NA	NR	1	NR	NA	NR	NR	NR
QC - 100 ppb	C92196M-MS	NA	NR	1	NR	NA	NR		
	C92196M-MSD	NA	NR	1	NR	NA	NR	NR	NR
QC - 500 ppb	C92192M-MS	NA	NR	1	NR	NA	NR		
	C92192M-MSD	NA	NR	1	NR	NA	NR	NR	NR
QC - 500 ppb	C92192M-MS	NA	NR	1	NR	NA	NR		
	C92192M-MSD	NA	NR	1	NR	NA	NR	NR	NR
Group 8	C92192M	Unknown	NR	1	NR	NA	NR		
Control	C92196M	Unknown	NR	1	NR	NA	NR		
0.0 ppm	C92207M	Unknown	NR	1	NR	NA	NR		
	C92216M	Unknown	NR	1	NR	NA	NR		NR
	C92259M	Unknown	NR	1	NR	NA	NR	NR	NR
	C92332F	Unknown	NR	1	NR	NA	NR		
	C92352F	Unknown	NR	1	NR	NA	NR		
	C92355F	Unknown	NR	1	NR	NA	NR		
	C92366F	Unknown	NR	1	NR	NA	NR		NR
	C92373F	Unknown	NR	1	NR	NA	NR	NR	NR
Group 9	C92264M	Unknown	NR	1	NR	NA	NR		
Low Dose	C92280M	Unknown	NR	1	NR	NA	NR		
1.0 ppm	C92284M	Unknown	NR	1	NR	NA	NR		
	C92300M	Unknown	NR	1	NR	NA	NR		NR
	C92329M	Unknown	NR	1	NR	NA	NR	NR	NR
	C92402F	Unknown	NR	1	NR	NA	NR		
	C92435F	Unknown	NR	1	NR	NA	NR		
	C92463F	Unknown	NR	1	NR	NA	NR		
	C92467F	Unknown	NR	1	NR	NA	NR		NR
	C92470F	Unknown	NR	1	NR	NA	NR	NR	NR

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

Date Entered/By:	04/17/00, 06/20/00 MMH/LAC, 05/08/01 mmh
Date Verified/ By:	03/22/01 hoj, 04/03/01 kjh, 05/10/01 LAC 5/22/01 mmh
Purity Entered/Verified:	03/29/01 LAC / 04/03/01 KJH

AMDT# 050598.2
Covance# 6329-228

Study: TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0, ETS-8-7.0
Analytical Equipment System Number: Madeline 041098, Amelia 062498, Soup 020199
Instrument Software/Version: MassLynx 3.1, 3.2, 3.3
Date of Extraction/Analyst: 07/17/98 IAS/SAH
Date of Analysis/Analyst: 07/17/98, 7/29/99, 08/07/98, 10/12/99 HOJ/SAH/MEE/MMH
Date of Data Reduction/Analyst: 10/13/99, 10/26/99, 02/09/00 MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 14 RAT LIVER REWORK

Group Dose	Sample #	Initial Wt. g	Total Mass of Liver g	PFOS Std Correction Factor	PFOS Purity Correction Factor (lot 193)	PFOS Conc. ug/g	PFOS Dilution Factor	PFOS Calc. Conc. ug/g	Filename	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	PFOSA Purity Correction Factor (lot L-2353)	PFOSA Conc. ug/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ug/g	Filename	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	1.0000	NA	0.9275	Unknown	0.00	1	0.00	A71798002	< LOQ (0.0114 ug/g)			Unknown	4.20	1	4.20	A71798002	< LOQ (0.0124 ug/g)		
	H2O Blk-2	1.0000	NA	0.9275	Unknown	0.00	1	0.00	A71798044	< LOQ (0.0114 ug/g)	<LOQ	NA	Unknown	3.82	1	3.82	A71798044	< LOQ (0.0124 ug/g)	<LOQ	NA
Matrix Blk	Rabbit Liver Blk-1	40.13	NA	0.9275	Unknown	0.00	1	0.00	A71798003	< LOQ (0.0114 ug/g)			Unknown	4.99	1	0.124	A71798003	< LOQ (0.0124 ug/g)		
	Rabbit Liver Blk-2	40.13	NA	0.9275	Unknown	0.00	1	0.00	A71798045	< LOQ (0.0114 ug/g)	<LOQ	NA	Unknown	3.90	1	0.0972	A71798045	< LOQ (0.0124 ug/g)	<LOQ	NA
QC - 100 ppb	C92255M-MS	1.0093	NA	NA	NA	1041	1	76.9	A71798040	65%			NA	167	1	156	A71798040	129%		
	C92255M-MSD	1.0093	NA	NA	NA	1100	1	136	A71798041	114%	89%	55%	NA	171	1	160	A71798041	132%	131%	2%
	C92255M-MS	1.0093	NA	NA	NA	NA	NA	NA	NA	NA 2nd			NA	159	1	148	S101299046	122% 2nd		
	C92255M-MSD	1.0093	NA	NA	NA	NA	NA	NA	NA	NA 2nd	NA	NA	NA	96.0	1	86	S101299047	71% 2nd	97%	53%
Group 8 Control 0.0 ppm	C92255M	1.0093	NA	0.9275	Unknown	963	1	885	A71798012	0.885			Unknown	9.20	1	9.12	A71798012	< LOQ (0.0124 ug/g)		
	C92256M	1.0115	NA	0.9275	Unknown	253	50	11589	A80798080	11.6			Unknown	366	1	362	A71798013	0.362		
	C92257M	0.9920	NA	0.9275	Unknown	1055	1	987	A71798014	0.987			Unknown	8.14	1	8.21	A71798014	< LOQ (0.0124 ug/g)		
	C92258M	1.0150	NA	0.9275	Unknown	674	1	616	A71798015	0.616		163	Unknown	7.23	1	7.12	A71798015	< LOQ (0.0124 ug/g)		190
	C92260M	0.9984	NA	0.9275	Unknown	772	1	717	A71798016	0.717	2.96	4.83	Unknown	8.64	1	8.65	A71798016	< LOQ (0.0124 ug/g)	0.0822	0.156
	C92396F	1.0116	NA	0.9275	Unknown	490	1	449	A71798019	0.449			Unknown	12.6	1	12.4	A71798019	0.0124		
	C92397F	0.9949	NA	0.9275	Unknown	498	1	464	A71798020	0.464			Unknown	12.3	1	12.3	A71798020	< LOQ (0.0124 ug/g)		
	C92398F	1.0015	NA	0.9275	Unknown	491	1	455	A71798021	0.455			Unknown	13.4	1	13.4	A71798021	0.0134		
	C92399F	1.0009	NA	0.9275	Unknown	362	1	335	A71798022	0.335		15.2	Unknown	9.45	1	9.44	A71798022	< LOQ (0.0124 ug/g)		6.71
	C92400F	0.9943	NA	0.9275	Unknown	375	1	350	A71798023	0.350	0.411	0.0625	Unknown	14.3	1	14.4	A71798023	0.0144	0.0130	0.000872
Group 9 Low Dose 1.0 ppm	C92325M	0.9911	NA	0.9275	Unknown	531	1	497	A71798026	0.497			Unknown	7.10	1	7.16	A71798026	< LOQ (0.0124 ug/g)		
	C92326M	0.9983	NA	0.9275	Unknown	254	80	18845	A80798081	18.8			Unknown	591	1	592	A71798027	0.592		
	C92327M	0.9977	NA	0.9275	Unknown	177	80	13190	A80798082	13.2			Unknown	454	1	455	A71798028	0.455		
	C92328M	1.0095	NA	0.9275	Unknown	204	80	15021	A80798083	15.0		56.2	Unknown	461	1	456	A71798029	0.456		60.3
	C92330M	1.0098	NA	0.9275	Unknown	199	80	14637	A80798084	14.6	12.4	6.99	Unknown	318	1	314	A71798030	0.314	0.366	0.221
	C92464F	1.0167	NA	0.9275	Unknown	485	25	11053	A80798087	11.1			Unknown	528	1	520	A71798033	0.520		
	C92465F	0.9997	NA	0.9275	Unknown	407	25	9443	A80798088	9.44			Unknown	497	1	497	A71798034	0.497		
	C92466F	1.0043	NA	0.9275	Unknown	308	25	7110	A80798089	7.11			Unknown	525	1	522	A71798035	0.522		
	C92468F	1.0114	NA	0.9275	Unknown	362	25	8299	A80798090	8.30		16.7	Unknown	374	1	370	A71798036	0.370		41.1
	C92469F	1.0089	NA	0.9275	Unknown	437	25	10052	A80798091	10.1	9.19	1.53	Unknown	1001	1	992	A71798037	0.992	0.580	0.239

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

Date Entered/By: 04/17/00 mmh
Date Verified/ By: 12/19/00 hoj, 03/22/01 hoj, 04/03/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/ 04/03/01 KJH

AMDA# 050598.2
Covance# 6329-228

Study: TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: FACT-M-1.0 & FACT-M-2.0, ETS-8-7.0
Analytical Equipment System Number: Madeline 041098, Amelia 062498, Soup 020199
Instrument Software/Version: MassLynx 3.1, 3.2, 3.3
Date of Extraction/Analyst: 07/17/98 IAS/SAH
Date of Analysis/Analyst: 07/17/98, 7/29/99, 08/07/98, 10/12/99 HOJ/SAH/MEE/MMH
Date of Data Reduction/Analyst: 10/13/99, 10/26/99, 02/09/00 MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 14 RAT LIVER REWORK

Group Dose	Sample #	PFOSAA Old Purity Correction Factor (lot 617)	PFOSAA Purity Correction Factor	PFOSAA Conc. ug/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ug/g	Filename	Concentration of PFOSAA ug/g or % Rec.	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD	EtFOSE Purity Correction Factor (lot 936)	EtFOSE Conc. ug/g	EtFOSE Dilution Factor	EtFOSE Calc. Conc. ug/g	Filename	Concentration of EtFOSE ug/g or % Rec.	Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	H2O Blk-1	0.5382	Unknown	NR	NR	NR	NR	NR			Unknown	NR	1	NR	NA	NR		
	H2O Blk-2	0.5382	Unknown	0.00	1	0.00	S101299004	<LOQ (0.0351 ug/g)	<LOQ	NA	Unknown	NR	1	NR	NA	NR	NR	NR
Matrix Blk	Rabbit Liver Blk-1	0.5382	Unknown	NR	NR	NR	NR	NR			Unknown	NR	1	NR	NA	NR		
	Rabbit Liver Blk-2	0.5382	Unknown	0.00	1	0.00	S101299005	<LOQ (0.0351 ug/g)	<LOQ	NA	Unknown	NR	1	NR	NA	NR	NR	NR
QC - 100 ppb	C92255M-MS	NA	NA	136	1	83.7	S101299046	66%			NA	NR	1	NR	NA	NR		
	C92255M-MSD	NA	NA	307	1	254	S101299047	200%	133%	101%	NA	NR	1	NR	NA	NR	NR	NR
	C92255M-MS	NA	NA	NA	NA	NA	NA	NA			NA	NR	1	NR	NA	NR		
	C92255M-MSD	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NR	1	NR	NA	NR	NR	NR
Group 8 Control 0.0 ppm	C92255M	0.5382	Unknown	51.1	1	27.3	S101299014	<LOQ (0.0351 ug/g)			Unknown	NR	1	NR	NA	NR		
	C92256M	0.5382	Unknown	283	10	1504	S101299030	1.50			Unknown	NR	1	NR	NA	NR		
	C92257M	0.5382	Unknown	21.7	1	11.8	S101299015	<LOQ (0.0351 ug/g)			Unknown	NR	1	NR	NA	NR		
	C92258M	0.5382	Unknown	31.0	1	16.4	S101299016	<LOQ (0.0351 ug/g)		199	Unknown	NR	1	NR	NA	NR		NR
	C92260M	0.5382	Unknown	72.5	1	39.1	S101299017	0.0391	0.330	0.656	Unknown	NR	1	NR	NA	NR	NR	NR
	C92396F	0.5382	Unknown	118	1	62.6	S101299018	0.0626			Unknown	NR	1	NR	NA	NR		
	C92397F	0.5382	Unknown	10.1	1	5.45	S101299022	<LOQ (0.0351 ug/g)			Unknown	NR	1	NR	NA	NR		
	C92398F	0.5382	Unknown	40.6	1	21.8	S101299023	<LOQ (0.0351 ug/g)			Unknown	NR	1	NR	NA	NR		
	C92399F	0.5382	Unknown	53.8	1	28.9	S101299024	<LOQ (0.0351 ug/g)		29.4	Unknown	NR	1	NR	NA	NR		NR
	C92400F	0.5382	Unknown	69.0	1	37.4	S101299025	0.0374	0.0411	0.0121	Unknown	NR	1	NR	NA	NR	NR	NR
Group 9 Low Dose 1.0 ppm	C92325M	0.5382	Unknown	79.3	1	43.1	S101299026	0.0431			Unknown	NR	1	NR	NA	NR		
	C92326M	0.5382	Unknown	469	20	5052	A102599062	5.05			Unknown	NR	1	NR	NA	NR		
	C92327M	0.5382	Unknown	537	10	2899	S101299042	2.90			Unknown	NR	1	NR	NA	NR		
	C92328M	0.5382	Unknown	461	10	2458	S101299032	2.46		65.2	Unknown	NR	1	NR	NA	NR		NR
	C92330M	0.5382	Unknown	747	10	3979	S101299033	3.98	2.89	1.88	Unknown	NR	1	NR	NA	NR	NR	NR
	C92464F	0.5382	Unknown	1149	10	6083	S101299034	6.08			Unknown	NR	1	NR	NA	NR		
	C92465F	0.5382	Unknown	421	10	2267	S101299038	2.27			Unknown	NR	1	NR	NA	NR		
	C92466F	0.5382	Unknown	142	5	380	S101299040	0.380			Unknown	NR	1	NR	NA	NR		
	C92468F	0.5382	Unknown	103	5	275	S101299041	0.275		95.1	Unknown	NR	1	NR	NA	NR		NR
	C92469F	0.5382	Unknown	381	20	4063	A102599063	4.06	2.61	2.49	Unknown	NR	1	NR	NA	NR	NR	NR

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

Date Entered/By: 04/17/00 mmh
Date Verified/ By: 12/19/00 hoj, 03/22/01 hoj, 04/03/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/ 04/03/01 KJH

Study: TOX003. 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS: 8.6.0 & ETS: 8.7.0
Analytical Equipment System Number: Amelia 062498, Davey 070799
Instrument Software/Version: MassLynx 3.3 & 3.4
Date of Extraction/Analyst: 4/14/00 SAL/RWW
Date of Analysis/Analyst: 04/17/00, 05/03/00, 05/15/00 IAS/MMH/IAS
Date of Data Reduction/Analyst: 04/18/00, 05/04/00, 05/16/00, 05/08/01 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 53 RAT LIVER

Group Dose	Sample #	Surrogate Verified	Initial Wt. g	Total Mass of Liver g	PFOS Old Purity Correction Factor	PFOS Purity Correction Factor	PFOS Conc. ug/g	PFOS Dilution Factor	PFOS Calc. Conc. ug/g	Filename	Concentration of PFOS ug/g or % Rec	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	PFOSA Old Purity Correction Factor	PFOSA Purity Correction Factor	PFOSA Conc. ug/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ug/g	Filename
			lot 171											lot 1.-15709						
Method Blk	RBL041400-H2OBlk-1	NA	1.0000	NA	0.9949	0.8640	0.00	1	0.00	A041700004	< LOQ (0.0267 ug/g)		NA	NA	0.9510	Unknown	2.59	1	2.51	A041700004
	RBL041400-H2OBlk-2	NA	1.0000	NA	0.9949	0.8640	0.00	1	0.00	D041400004	< LOQ (0.0107 ug/g)	<LOQ	NA	NA	0.9510	Unknown	0.00	1	0.00	D041400004
Matrix Blk	RBL041400-LiverBlk-1	NA	1.0000	NA	0.9949	0.8640	0.00	1	0.00	A041700005	< LOQ (0.0267 ug/g)		NA	NA	0.9510	Unknown	2.09	1	2.20	A041700005
	RBL041400-LiverBlk-2	NA	1.0000	NA	0.9949	0.8640	0.00	1	4.77	D041400005	< LOQ (0.0107 ug/g)	<LOQ	NA	NA	0.9510	Unknown	0.00	1	0.00	D041400005
QC - 300 ppb	C92197/M/G8-MS-1	NA	1.0453	NA	0.9949	0.8640	289	1	226	D041400017	78%			NA	NA	NA	239	1	224	D041400017
	C92197/M/G8-MSD-1	NA	1.0453	NA	0.9949	0.8640	287	1	224	D041400018	78%	78%	1%	NA	NA	NA	262	1	246	D041400018
	C92195/M/G8-MS-1	High - Not Confirmed	1.0243	NA	0.9949	0.8640	1192	1	403	A041700017	137% A			NA	NA	NA	291	1	280	A041700017
	C92195/M/G8-MSD-1	High - Not Confirmed	1.0243	NA	0.9949	0.8640	1112	1	324	A041700018	110% A	124%	22%	NA	NA	NA	196	1	188	A041700018
	RBL041400-MS-1	NA	1.0000	NA	0.9949	0.8640	232	1	232	D041400076	76%			NA	NA	NA	283	1	282	D041400075
	RBL041400-MSD-1	NA	1.0000	NA	0.9949	0.8640	261	1	261	D041400077	85%	80%	12%	NA	NA	NA	260	1	259	D041400076
Group 8 0.0 ppm	C92195M		1.0243	NA	0.9949	0.8640	920	1	780	A041700019	0.780 A			NA	0.9510	Unknown	4.08	1	4.19	A041700019
	C92199M	High - Not Confirmed	1.0416	NA	0.9949	0.8640	171	20	2855	A050300019	2.86			High - Not Confirmed	0.9510	Unknown	2.72	1	2.75	A041700020
	C92205M	NA	1.0515	NA	0.9949	0.8640	741	1	612	A041700021	0.612			NA	0.9510	Unknown	2.67	1	2.67	A041700021
	C92238M	NA	0.9947	NA	0.9949	0.8640	433	1	378	A041700024	0.378		81.7	NA	0.9510	Unknown	4.06	1	4.29	A041700024
	C92246M	High - Not Confirmed	1.0013	NA	0.9949	0.8640	298	10	2581	A050300020	2.58	1.44	1.18	NA	0.9510	Unknown	3.08	1	3.23	A041700025
	C92341F	NA	1.0282	NA	0.9949	0.8640	0.00	1	0.00	A041700026	< LOQ (0.0267 ug/g)			NA	0.9510	Unknown	3.07	1	3.14	A041700026
	C92356F	NA	0.9926	NA	0.9949	0.8640	114	1	100	A041700027	0.100			NA	0.9510	Unknown	3.66	1	3.88	A041700027
	C92372F	NA	1.0245	NA	0.9949	0.8640	223	1	189	A041700028	0.189			NA	0.9510	Unknown	1.91	1	1.96	A041700028
	C92376F	NA	1.0418	NA	0.9949	0.8640	349	1	291	A041700031	0.291		64.6	NA	0.9510	Unknown	6.55	1	6.61	A041700031
	C92390F	NA	1.0329	NA	0.9949	0.8640	334	1	281	A041700032	0.281	0.178	0.115	NA	0.9510	Unknown	3.06	1	3.12	A041700032
Group 9 1.0 ppm	C92267M	High - Not Confirmed	1.0137	NA	0.9949	0.8640	497	100	42575	A050300028	42.6			NA	0.9510	Unknown	132	1	137	A041700033
	C92274M	High - Not Confirmed	1.0853	NA	0.9949	0.8640	478	100	38248	A050300031	38.2			NA	0.9510	Unknown	147	1	142	A041700034
	C92287M	High - Not Confirmed	0.9901	NA	0.9949	0.8640	572	100	50148	A050300032	50.1			NA	0.9510	Unknown	227	1	241	A041700035
	C92288M	High - Not Confirmed	1.0555	NA	0.9949	0.8640	410	100	33720	A050300033	33.7		29.4	NA	0.9510	Unknown	321	1	320	A041700038
	C92320M	High - Not Confirmed	0.9914	NA	0.9949	0.8640	239	100	20904	A050300034	20.9	37.1	10.9	NA	0.9510	Unknown	170	1	181	A041700039
	C92406F	High - Not Confirmed	1.0459	NA	0.9949	0.8640	362	100	30075	A050300021	30.1			NA	0.9510	Unknown	289	1	290	A041700040
	C92411F	High - Not Confirmed	1.0815	NA	0.9949	0.8640	460	100	36954	A050300024	37.0			NA	0.9510	Unknown	197	1	192	A041700041
	C92418F	High - Not Confirmed	1.0528	NA	0.9949	0.8640	372	100	30647	A050300025	30.6			NA	0.9510	Unknown	179	1	179	A041700042
	C92422F	High - Not Confirmed	1.0250	NA	0.9949	0.8640	323	100	27346	A050300026	27.3		55.0	NA	0.9510	Unknown	386	1	396	A041700045
	C92438F		1.0810	NA	0.9949	0.8640	1516	1	1218	A041700046	1.22 A	25.2	13.9	NA	0.9510	Unknown	160	1	156	A041700046

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PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
PFOSEA = Perfluorooctane sulfonyl ethylamide
M556 = C8F17SO2N((H)CH2COO)

Date Entered/Analyst: 04/26/00 mmh, 09/15/00 LAC, 05/09/01 mmh
Date Verified/Analyst: 03/22/01 hoj, 04/03/01 kjh, 05/10/01 LAC 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/ 04/03/01 KJH

Covance# 6329-228

Study: TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T 6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Amelia 062498, Davey 070799
Instrument Software/Version: MassLynx 3.3 & 3.4
Date of Extraction/Analyst: 4/14/00 SAL/RWW
Date of Analysis/Analyst: 04/17/00, 05/03/00, 05/15/00 IAS/MMH/IAS
Date of Data Reduction/Analyst: 04/18/00, 05/04/00, 05/16/00, 05/08/01 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 53 RAT LIVER

Concentration of PFOSA ug/g or % Rec	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	Group Dose	Sample #	Surrogate Verified	PFOSAA Old Purity Correction Factor (lot 98-0207-0303-3)	PFOSAA Purity Correction Factor	PFOSAA Conc. ng/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ng/g	Filename	Concentration of PFOSAA ug/g or % Rec	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	EtFOSE Old Purity Correction Factor (lot SD013)	EtFOSE Purity Correction Factor	EtFOSE Conc. ng/g
< LOQ (0.00644 ug/g)		NA	Method Blk	RBL041400-H2OBlk-1	NA	0.9760	Unknown	0.00	1	0.00	A041700004	< LOQ (0.0125 ug/g)		NA	NA	0.9770	0.8890	0.00
< LOQ (0.0322 ug/g)	<LOQ	NA		RBL041400-H2OBlk-2	NA	0.9760	Unknown	0.00	1	0.00	D041400004	< LOQ (0.0314 ug/g)	<LOQ	NA	NA	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)		NA	Matrix Blk	RBL041400-LiverBlk-1	NA	0.9760	Unknown	0.00	1	0.00	A041700005	< LOQ (0.0125 ug/g)		NA	NA	0.9770	0.8890	0.00
< LOQ (0.0322 ug/g)	<LOQ	NA		RBL041400-LiverBlk-2	NA	0.9760	Unknown	0.00	1	0.00	D041400005	< LOQ (0.0314 ug/g)	<LOQ	NA	NA	0.9770	0.8890	0.00
78%			QC - 300 ppb	C92197/M/GB-MS-1	NA	0.9760	Unknown	224	1	215	D041400017	74%			NA	NA	NA	277
86%	82%	9%		C92197/M/GB-MSD-1	NA	0.9760	Unknown	260	1	249	D041400018	86%	80%	15%	NA	NA	NA	231
94%				C92195/M/GB-MS-1	NA	0.9760	Unknown	251	1	240	A041700017	82%			NA	NA	NA	472
63%	79%	39%		C92195/M/GB-MSD-1	NA	0.9760	Unknown	254	1	243	A041700018	83%	82%	2%	NA	NA	NA	459
92%				RBL041400-MS-1	NA	0.9760	Unknown	232	1	222	D041400075	73%			NA	NA	NA	NR
85%	88%	9%		RBL041400-MSD-1	NA	0.9760	Unknown	266	1	266	D041400076	87%	80%	18%	NA	NA	NA	NR
< LOQ (0.00644 ug/g)			Group 8	C92195M	NA	0.9760	Unknown	0.00	1	0.00	A041700019	< LOQ (0.0125 ug/g)			NA	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)			0.0 ppm	C92199M	High - Not Confirmed	0.9760	Unknown	0.00	1	0.00	A041700020	< LOQ (0.0125 ug/g)			High - Not Confirmed	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)				C92205M	NA	0.9760	Unknown	0.00	1	0.00	A041700021	< LOQ (0.0125 ug/g)			NA	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)		NA		C92238M	NA	0.9760	Unknown	0.00	1	0.00	A041700024	< LOQ (0.0125 ug/g)		NA	NA	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)	<LOQ	NA		C92246M	NA	0.9760	Unknown	0.00	1	0.00	A041700025	< LOQ (0.0125 ug/g)	<LOQ	NA	NA	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)				C92341F	NA	0.9760	Unknown	0.00	1	0.00	A041700026	< LOQ (0.0125 ug/g)			NA	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)				C92356F	NA	0.9760	Unknown	0.00	1	0.00	A041700027	< LOQ (0.0125 ug/g)			NA	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)				C92372F	NA	0.9760	Unknown	4.40	1	4.40	A041700028	< LOQ (0.0125 ug/g)			NA	0.9770	0.8890	0.00
0.00661		1.22		C92376F	NA	0.9760	Unknown	10.1	1	9.96	A041700031	< LOQ (0.0125 ug/g)		NA	NA	0.9770	0.8890	0.00
< LOQ (0.00644 ug/g)	0.00647	0.0000786		C92390F	NA	0.9760	Unknown	0.00	1	0.00	A041700032	< LOQ (0.0125 ug/g)	<LOQ	NA	NA	0.9770	0.8890	0.00
0.137			Group 9	C92267M	NA	0.9760	Unknown	344	1	348	A041700033	0.348			NA	0.9770	0.8890	0.00
0.142			1.0 ppm	C92274M	NA	0.9760	Unknown	885	1	836	A041700034	0.836			NA	0.9770	0.8890	0.00
0.241				C92287M	NA	0.9760	Unknown	1872	1	1937	A041700035	1.94 A			NA	0.9770	0.8890	0.00
0.320		37.7		C92288M	NA	0.9760	Unknown	678	1	658	A041700038	0.658		66.8	NA	0.9770	0.8890	0.00
0.181	0.204	0.0769		C92320M	NA	0.9760	Unknown	729	1	753	A041700039	0.753	0.906	0.605	NA	0.9770	0.8890	0.00
0.290				C92406F	NA	0.9760	Unknown	464	1	455	A041700040	0.455			NA	0.9770	0.8890	0.00
0.192				C92411F	NA	0.9760	Unknown	406	1	384	A041700041	0.384			NA	0.9770	0.8890	0.00
0.179				C92418F	NA	0.9760	Unknown	583	1	567	A041700042	0.567			NA	0.9770	0.8890	0.00
0.396		41.2		C92422F	NA	0.9760	Unknown	729	1	728	A041700045	0.728		24.8	NA	0.9770	0.8890	0.00
0.156	0.242	0.0999		C92438F	NA	0.9760	Unknown	530	1	502	A041700046	0.502	0.527	0.131	NA	0.9770	0.8890	0.00

A = Above linear range of the initial curve, estimated values.
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PFOSAA = Perfluorooctanesulfonamidoacetate
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M556 = C8F17SO2N((H)CH2COO)

Date Entered/Analyst: 04/26/00 mmh, 09/15/00 LAC, 05/09/01 mmh
Date Verified/Analyst: 03/22/01 hoj, 04/03/01 kjh, 05/10/01 LAC 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/ 04/03/01 KJH

Covance# 6329-228

Study: TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Amelia 062498, Davey 070799
Instrument Software/Version: Massl.ynx 3.3 & 3.4
Date of Extraction/Analyst: 4/14/00 SAL/RWW
Date of Analysis/Analyst: 04/17/00, 05/03/00, 05/15/00 IAS/MMH/IAS
Date of Data Reduction/Analyst: 04/18/00, 05/04/00, 05/16/00, 05/08/01 IAS/MMH

Filename: See Below
R-Squared Val See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 53 RAT LIVER

lot NB113047-80

lot 529

EtFOSE Dilution Factor	EtFOSE Calc. Conc. ug/g	Filename	Concentration of EtFOSE ug/g or % Rec		Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD	Group Dose	Sample #	Surrogate Verified	M556 Old Purity Correction Factor	M556 Purity Correction Factor	M556 Conc. ug/g	M556 Dilution Factor	M556 Calc. Conc. ug/g	Filename	Concentration of M556 ug/g or % Rec	Mean M556 ug/g	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	PFOSEA Old Purity Correction Factor
1	0.00	A041700004	< LOQ (0.0559 ug/g)	B		NA	Method Blk	RBL041400-H2OBlk-1	NA	0.9989	Unknown	0.00	1	0.00	A041700004	< LOQ (0.0307 ug/g)		NA	NA	0.9930
1	0.00	D041400004	< LOQ (0.0559 ug/g)	B	<LOQ	NA		RBL041400-H2OBlk-2	NA	0.9989	Unknown	0.00	1	0.00	D041400004	< LOQ (0.0307 ug/g)	<LOQ	NA	NA	0.9930
1	0.00	A041700005	< LOQ (0.0559 ug/g)	B		NA	Matrix Blk	RBL041400-LiverBlk-1	NA	0.9989	Unknown	0.00	1	0.00	A041700005	< LOQ (0.0307 ug/g)		NA	NA	0.9930
1	0.00	D041400005	< LOQ (0.0559 ug/g)	B	<LOQ	NA		RBL041400-LiverBlk-2	NA	0.9989	Unknown	0.00	1	0.00	D041400005	< LOQ (0.0307 ug/g)	<LOQ	NA	NA	0.9930
1	265	D041400017	92%	B			QC - 300 ppb	C92197M/G8-MS-1	NA	NA	NA	361	1	345	D041400017	119%			NA	NA
1	221	D041400018	77%	B	84%	18%		C92197/M/G8-MSD-1	NA	NA	NA	370	1	354	D041400018	122%	121%	2%	NA	NA
1	452	A041700017	154%	B				C92195/M/G8-MS-1	NA	NA	NA	316	1	303	A041700017	103%			NA	NA
1	439	A041700018	149%	B	151%	3%		C92195/M/G8-MSD-1	NA	NA	NA	331	1	317	A041700018	108%	105%	5%	NA	NA
NR	NR	NR	NR	B				RBL041400-MS-1	NA	NA	NA	256	1	245	D041400075	80%			NA	NA
NR	NR	NR	NR	B	NR	NR		RBL041400-MSD-1	NA	NA	NA	257	1	257	D041400076	84%	82%	5%	NA	NA
1	0.00	A041700019	< LOQ (0.0559 ug/g)	B			Group 8	C92195M	NA	0.9989	Unknown	0.00	1	0.00	A041700019	< LOQ (0.0307 ug/g)			NA	0.9930
1	0.00	A041700020	< LOQ (0.0559 ug/g)	B			0.0 ppm	C92199M	High - Not Confirmed	0.9989	Unknown	0.00	1	0.00	A041700020	< LOQ (0.0307 ug/g)			High - Not Confirmed	0.9930
1	0.00	A041700021	< LOQ (0.0559 ug/g)	B				C92205M	NA	0.9989	Unknown	0.00	1	0.00	A041700021	< LOQ (0.0307 ug/g)			NA	0.9930
1	0.00	A041700024	< LOQ (0.0559 ug/g)	B		NA		C92238M	NA	0.9989	Unknown	0.00	1	0.00	A041700024	< LOQ (0.0307 ug/g)		NA	NA	0.9930
1	0.00	A041700025	< LOQ (0.0559 ug/g)	B	<LOQ	NA		C92246M	NA	0.9989	Unknown	0.00	1	0.00	A041700025	< LOQ (0.0307 ug/g)	<LOQ	NA	NA	0.9930
1	0.00	A041700026	< LOQ (0.0559 ug/g)	B				C92341F	NA	0.9989	Unknown	0.00	1	0.00	A041700026	< LOQ (0.0307 ug/g)			NA	0.9930
1	0.00	A041700027	< LOQ (0.0559 ug/g)	B				C92356F	NA	0.9989	Unknown	0.00	1	0.00	A041700027	< LOQ (0.0307 ug/g)			NA	0.9930
1	0.00	A041700028	< LOQ (0.0559 ug/g)	B				C92372F	NA	0.9989	Unknown	0.00	1	0.00	A041700028	< LOQ (0.0307 ug/g)			NA	0.9930
1	0.00	A041700031	< LOQ (0.0559 ug/g)	B		NA		C92376F	NA	0.9989	Unknown	0.00	1	0.00	A041700031	< LOQ (0.0307 ug/g)		NA	NA	0.9930
1	0.00	A041700032	< LOQ (0.0559 ug/g)	B	<LOQ	NA		C92390F	NA	0.9989	Unknown	0.00	1	0.00	A041700032	< LOQ (0.0307 ug/g)	<LOQ	NA	NA	0.9930
1	0.00	A041700033	< LOQ (0.0559 ug/g)	B			Group 9	C92367M	NA	0.9989	Unknown	269	1	266	A041700033	0.266			NA	0.9930
1	0.00	A041700034	< LOQ (0.0559 ug/g)	B			1.0 ppm	C92274M	NA	0.9989	Unknown	359	1	331	A041700034	0.331			NA	0.9930
1	0.00	A041700035	< LOQ (0.0559 ug/g)	B				C92287M	NA	0.9989	Unknown	376	1	380	A041700035	0.380			NA	0.9930
1	0.00	A041700038	< LOQ (0.0559 ug/g)	B		NA		C92288M	NA	0.9989	Unknown	450	1	426	A041700038	0.426		20.3	NA	0.9930
1	0.00	A041700039	< LOQ (0.0559 ug/g)	B	<LOQ	NA		C92320M	NA	0.9989	Unknown	274	1	277	A041700039	0.277	0.336	0.0682	NA	0.9930
1	0.00	A041700040	< LOQ (0.0559 ug/g)	B				C92406F	NA	0.9989	Unknown	236	1	226	A041700040	0.226			NA	0.9930
1	0.00	A041700041	< LOQ (0.0559 ug/g)	B				C92411F	NA	0.9989	Unknown	324	1	300	A041700041	0.300			NA	0.9930
1	0.00	A041700042	< LOQ (0.0559 ug/g)	B				C92418F	NA	0.9989	Unknown	288	1	274	A041700042	0.274			NA	0.9930
1	0.00	A041700045	< LOQ (0.0559 ug/g)	B		NA		C92422F	NA	0.9989	Unknown	361	1	353	A041700045	0.353		24.7	NA	0.9930
1	0.00	A041700046	< LOQ (0.0559 ug/g)	B	<LOQ	NA		C92438F	NA	0.9989	Unknown	196	1	182	A041700046	0.182	0.267	0.0660	NA	0.9930

A = Above linear range of the initial curve, estimated values.
B = Qualitative only

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
PFOSEA = Perfuorooctane sulfonyl ethylamide
M556 = C8F17SO2N((H)CH2COO)

Date Entered/Analyst: 04/26/00 mmh, 09/15/00 LAC, 05/09/01 mmh
Date Verified/Analyst: 03/22/01 hoj, 04/03/01 kjh, 05/10/01 LAC 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC/ 04/03/01 KJH

ETS-8-7.0

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Liver
Method/Revision:	ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number:	Amelia 062498, Davey 070799
Instrument Software/Version:	MassLynx 3.3 & 3.4
Date of Extraction/Analyst:	4/14/00 SAL/RWW
Date of Analysis/Analyst:	04/17/00, 05/03/00, 05/15/00 IAS/MMH/IAS
Date of Data Reduction/Analyst:	04/18/00, 05/04/00, 05/16/00, 05/08/01 IAS/MMH

Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments

Sample Data

PFOSEA Purity Correction Factor	PFOSEA Conc. ug/g	PFOSEA Dilution Factor	PFOSEA Calc. Conc. ug/g	Filename	Concentration of PFOSEA ug/g or % Rec	Mean PFOSEA ug/g	RSD Std. Dev. MS/MSD RPD
Unknown	0.00	1	0.00	A041700004	< LOQ (0.0308 ug/g)		NA
Unknown	0.00	1	0.00	D041400004	< LOQ (0.0308 ug/g)	<LOQ	NA
Unknown	0.00	1	0.00	A041700005	< LOQ (0.0308 ug/g)		NA
Unknown	0.00	1	0.00	D041400005	< LOQ (0.0308 ug/g)	<LOQ	NA
NA	208	1	199	D041400017	69%		
NA	254	1	243	D041400018	84%	77%	20%
NA	442	1	423	A041700017	144%		
NA	353	1	338	A041700018	115%	130%	22%
NA	358	1	343	D041400075	112%		
NA	393	1	376	D041400076	123%	117%	9%
Unknown	0.00	1	0.00	A041700019	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700020	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700021	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700024	< LOQ (0.0308 ug/g)		NA
Unknown	0.00	1	0.00	A041700025	< LOQ (0.0308 ug/g)	<LOQ	NA
Unknown	0.00	1	0.00	A041700026	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700027	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700028	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700031	< LOQ (0.0308 ug/g)		NA
Unknown	0.00	1	0.00	A041700032	< LOQ (0.0308 ug/g)	<LOQ	NA
Unknown	0.00	1	0.00	A041700033	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700034	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700035	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700038	< LOQ (0.0308 ug/g)		NA
Unknown	0.00	1	0.00	A041700039	< LOQ (0.0308 ug/g)	<LOQ	NA
Unknown	0.00	1	0.00	A041700040	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700041	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700042	< LOQ (0.0308 ug/g)		
Unknown	0.00	1	0.00	A041700045	< LOQ (0.0308 ug/g)		NA
Unknown	0.00	1	0.00	A041700046	< LOQ (0.0308 ug/g)	<LOQ	NA

WEEK 53 RAT LIVER

Group Dose	Sample #	PFOS Calc. Conc. ug/g	Concentration of PFOS ug/g or % Rec	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	PFOSA Calc. Conc. ug/g	Concentration of PFOSA ug/g or % Rec	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	PFOSAA Calc. Conc. ug/g	Concentration of PFOSAA ug/g or % Rec
Method Blk	RBL041400-H2OBlk-1	0.00	< LOQ (0.0267 ug/g)			2.51	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)
	RBL041400-H2OBlk-2	0.00	< LOQ (0.0107 ug/g)	<LOQ	NA	0.00	< LOQ (0.0322 ug/g)	<LOQ	NA	0.00	< LOQ (0.0314 ug/g)
Matrix Blk	RBL041400-LiverBlk-1	0.00	< LOQ (0.0267 ug/g)			2.20	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)
	RBL041400-LiverBlk-2	4.77	< LOQ (0.0107 ug/g)	<LOQ	NA	0.00	< LOQ (0.0322 ug/g)	<LOQ	NA	0.00	< LOQ (0.0314 ug/g)
QC - 300 ppb	C92197/M/G8-MS-1	226	78%			224	78%			215	74%
	C92197/M/G8-MSD-1	224	78%	78%	1%	246	86%	82%	9%	249	86%
	C92195/M/G8-MS-1	403	137%			280	94%			240	82%
	C92195/M/G8-MSD-1	324	110%	124%	22%	188	63%	79%	39%	243	83%
	RBL041400-MS-1	232	76%			282	92%			222	73%
	RBL041400-MSD-1	261	85%	80%	12%	259	85%	88%	9%	266	87%
Group 8 0.0 ppm	C92195M	780	0.780 A			4.19	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)
	C92199M	2855	2.86			2.75	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)
	C92205M	612	0.612			2.67	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)
	C92238M	378	0.378		81.7	4.29	< LOQ (0.00644 ug/g)		NA	0.00	< LOQ (0.0125 ug/g)
	C92246M	2581	2.58	1.44	1.18	3.23	< LOQ (0.00644 ug/g)	<LOQ	NA	0.00	< LOQ (0.0125 ug/g)
	C92341F	0.00	< LOQ (0.0267 ug/g)			3.14	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)
	C92356F	100	0.100			3.88	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)
	C92372F	189	0.189			1.96	< LOQ (0.00644 ug/g)			4.40	< LOQ (0.0125 ug/g)
	C92376F	291	0.291		64.6	6.61	0.00661		1.22	9.96	< LOQ (0.0125 ug/g)
	C92390F	281	0.281	0.178	0.115	3.12	< LOQ (0.00644 ug/g)	0.00647	0.00	0.00	< LOQ (0.0125 ug/g)
Group 9 1.0 ppm	C92267M	42575	42.6			137	0.137			348	0.348
	C92274M	38248	38.2			142	0.142			836	0.836
	C92287M	50148	50.1			241	0.241			1937	1.94
	C92288M	33720	33.7		29.4	320	0.320		37.7	658	0.658
	C92320M	20904	20.9	37.1	10.9	181	0.181	0.204	0.0769	753	0.753
	C92406F	30075	30.1			290	0.290			455	0.455
	C92411F	36954	37.0			192	0.192			384	0.384
	C92418F	30647	30.6			179	0.179			567	0.567
	C92422F	27346	27.3		55.0	396	0.396		41.2	728	0.728
	C92438F	1218	1.22 A	25.2	13.9	156	0.156	0.242	0.0999	502	0.502

A = Above linear range of the initial curve, estimated values.
B = Qualitative only

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
PFOSEA = Perfluorooctane sulfonyl ethylamide
M556 = C8F17SO2N(H)CH2COO)

Date Entered/Analyst:	04/26/00 mmh, 09/15/00 LAC, 05/09/01 mmh
Date Verified/Analyst:	03/22/01 hoj, 04/03/01 kjh, 05/10/01 LAC 5/22/01 mmh
Purity Entered/Verified:	03/29/01 LAC/ 04/03/01 KJH

Covance# 6329-228

Study: TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Ameka 062498, Davey 070799
Instrument Software/Version: MassLynx 3.3 & 3.4
Date of Extraction/Analyst: 4/14/00 SAL/RWW
Date of Analysis/Analyst: 4/17/00, 5/15/00 IAS/MMH/IAS
Date of Data Reduction/Analyst: 4/18/00, 5/16/00, 05/08/01 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 105 RAT LIVER

Group Dose	Sample #	Surrogate Verified	Initial Wt. g	Total Mass of Liver g	PFOS Old Purity Correction Factor (lot 171)	PFOS Purity Correction Factor	PFOS Conc. ng/g	PFOS Dilution Factor	PFOS Calc. Conc. ng/g	Filename	Concentration of PFOS ug/g or % Rec	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	PFOSA Old Purity Correction Factor (lot L-15709)	PFOSA Purity Correction Factor	PFOSA Conc. ng/g	PFOSA Dilution Factor	PFOSA Calc. Conc. ng/g	Filename
Method Blk	RBL041400-H2OBlk-1	NA	1.0000	NA	0.9949	0.8640	0.00	1	0.00	A041700004	< LOQ (0.0267 ug/g)		NA	NA	0.9510	Unknown	2.39	1	2.51	A041700004
	RBL041400-H2OBlk-2	NA	1.0000	NA	0.9949	0.8640	0.00	1	0.00	D041400004	< LOQ (0.0107 ug/g)	<LOQ	NA	NA	0.9510	Unknown	0.00	1	0.00	D041400004
Matrix Blk	RBL041400-LiverBlk-1	NA	1.0000	NA	0.9949	0.8640	0.00	1	0.00	A041700005	< LOQ (0.0267 ug/g)		NA	NA	0.9510	Unknown	2.09	1	2.20	A041700005
	RBL041400-LiverBlk-2	NA	1.0000	NA	0.9949	0.8640	0.00	1	4.77	D041400005	< LOQ (0.0107 ug/g)	<LOQ	NA	NA	0.9510	Unknown	0.00	1	0.00	D041400005
QC - 300 ppb	C92197/MGB-MS-1	NA	1.0453	NA	0.9949	0.8640	289	1	226	D041400017	78%			NA	0.9510	Unknown	239	1	224	D041400017
	C92197/MGB-MSD-1	NA	1.0453	NA	0.9949	0.8640	287	1	224	D041400018	78%	78%	1%	NA	0.9510	Unknown	262	1	246	D041400018
	C92195/MGB-MS-1	High - Not Confirmed	1.0243	NA	0.9949	0.8640	1192	1	403	A041700017	137%			NA	0.9510	Unknown	291	1	280	A041700017
	C92195/MGB-MSD-1	High - Not Confirmed	1.0243	NA	0.9949	0.8640	1112	1	324	A041700018	110%	124%	22%	NA	0.9510	Unknown	196	1	188	A041700018
	RBL041400-MS-1	NA	1.0000	NA	0.9949	0.8640	232	1	232	D041400076	76%			NA	0.9510	Unknown	283	1	282	D041400075
	RBL041400-MSD-1	NA	1.0000	NA	0.9949	0.8640	261	1	261	D041400077	85%	80%	12%	NA	0.9510	Unknown	260	1	259	D041400076
Group 8 0.0 ppm	C92197M	NA	1.0453	NA	0.9949	0.8640	53.1	1	44.1	D041400019	0.0441			NA	0.9510	Unknown	0.000	1	0.00	D041400019
	C92203M	NA	1.0172	NA	0.9949	0.8640	3.44	1	2.94	D041400020	< LOQ (0.0107 ug/g)			NA	0.9510	Unknown	0.000	1	0.00	D041400020
	C92208M	NA	1.0160	NA	0.9949	0.8640	9.43	1	8.06	D041400021	< LOQ (0.0107 ug/g)			NA	0.9510	Unknown	0.000	1	0.00	D041400021
	C92209M	NA	1.0160	NA	0.9949	0.8640	0.00	1	0.00	D041400024	< LOQ (0.0107 ug/g)			NA	0.9510	Unknown	0.23	1	0.24	D041400024
	C92218M	NA	0.9845	NA	0.9949	0.8640	83.0	1	73.2	D041400025	0.0732			NA	0.9510	Unknown	0.000	1	0.00	D041400025
	C92222M	NA	1.0021	NA	0.9949	0.8640	3.96	1	3.43	D041400026	< LOQ (0.0107 ug/g)			NA	0.9510	Unknown	0.000	1	0.00	D041400026
	C92231M	NA	1.0036	NA	0.9949	0.8640	2.78	1	2.41	D041400027	< LOQ (0.0107 ug/g)			NA	0.9510	Unknown	0.000	1	0.00	D041400027
	C92233M	NA	0.9986	NA	0.9949	0.8640	0.00	1	0.00	D041400028	< LOQ (0.0107 ug/g)			NA	0.9510	Unknown	0.000	1	0.00	D041400028
	C92239M	NA	1.0052	NA	0.9949	0.8640	261	1	225	D041400031	0.225		141	NA	0.9510	Unknown	0.000	1	0.00	D041400031
	C92252M	NA	0.9930	NA	0.9949	0.8640	281	1	246	D041400032	0.246	0.0653	0.0923	NA	0.9510	Unknown	0.000	1	0.00	D041400032
	C92333F	NA	0.9977	NA	0.9949	0.8640	125	1	109	D041400033	0.109			NA	0.9510	Unknown	0.000	1	0.00	D041400033
	C92334F	NA	1.0419	NA	0.9949	0.8640	112	1	93.0	D041400034	0.0930			NA	0.9510	Unknown	0.37	1	0.37	D041400034
	C92348F	NA	1.0545	NA	0.9949	0.8640	186	1	153	D041400035	0.153			NA	0.9510	Unknown	0.09	1	0.090	D041400035
	C92354F	NA	1.0607	NA	0.9949	0.8640	16.9	1	13.8	D041400038	0.0138			NA	0.9510	Unknown	0.000	1	0.00	D041400038
	C92358F	NA	0.9868	NA	0.9949	0.8640	7.29	1	6.42	D041400039	< LOQ (0.0107 ug/g)			NA	0.9510	Unknown	0.08	1	0.09	D041400039
	C92360F	NA	1.0691	NA	0.9949	0.8640	109	1	88.4	D041400040	0.0884			NA	0.9510	Unknown	0.26	1	0.26	D041400040
	C92362F	NA	1.0245	NA	0.9949	0.8640	35.3	1	29.9	D041400041	0.0299			NA	0.9510	Unknown	0.53	1	0.54	D041400041
	C92369F	NA	1.0813	NA	0.9949	0.8640	140	1	112	D041400042	0.112			NA	0.9510	Unknown	0.000	1	0.00	D041400042
	C92374F	NA	1.0801	NA	0.9949	0.8640	138	1	111	D041400045	0.111		59.9	NA	0.9510	Unknown	0.47	1	0.46	D041400045
	C92383F	NA	1.0817	NA	0.9949	0.8640	187	1	150	D041400046	0.150	0.0872	0.0522	NA	0.9510	Unknown	0.33	1	0.32	D041400046
Group 9 1.0 ppm	C92261M	NA	0.9963	NA	0.9949	0.8640	289	20	5037	D051500017	5.04			NA	0.9510	Unknown	400	1	422	D041400047
	C92266M	NA	0.9909	NA	0.9949	0.8640	429	20	7515	D051500018	7.51			NA	0.9510	Unknown	264	1	280	D041400048
	C92270M	NA	1.0076	NA	0.9949	0.8640	737	20	12702	D051500019	12.7			NA	0.9510	Unknown	795	1	829	D041400049
	C92278M	NA	1.0037	NA	0.9949	0.8640	218	20	3781	D051500020	3.78			NA	0.9510	Unknown	482	1	505	D041400052
	C92281M	NA	1.0052	NA	0.9949	0.8640	270	20	4660	D051500021	4.66			NA	0.9510	Unknown	235	1	246	D041400053
	C92283M	NA	1.0182	NA	0.9949	0.8640	636	20	10841	D051500025	10.8			NA	0.9510	Unknown	341	1	352	D041400054
	C92309M	NA	0.9823	NA	0.9949	0.8640	643	1	568	D041400055	0.57			NA	0.9510	Unknown	169	1	180	D041400055
	C92311M	NA	0.9877	NA	0.9949	0.8640	118	20	2084	D051500027	2.08			NA	0.9510	Unknown	355	1	378	D041400056
	C92318M	NA	0.9963	NA	0.9949	0.8640	498	20	8678	D051500028	8.68		61.2	NA	0.9510	Unknown	251	1	265	D041400059
	C92321M	NA	0.9981	NA	0.9949	0.8640	359	20	6249	D051500029	6.25	6.21	3.80	NA	0.9510	Unknown	245	1	258	D041400060
	C92410F	NA	1.0894	NA	0.9949	0.8640	672	20	10717	D051500033	10.7			NA	0.9510	Unknown	566	1	546	D041400061
	C92416F	NA	0.9916	NA	0.9949	0.8640	623	20	10911	D051500034	10.9			NA	0.9510	Unknown	729	1	773	D041400062
	C92419F	NA	1.0143	NA	0.9949	0.8640	127	20	2177	D051500035	2.18			NA	0.9510	Unknown	494	1	512	D041400063
	C92423F	NA	0.9980	NA	0.9949	0.8640	294	20	5114	D051500036	5.11			NA	0.9510	Unknown	592	1	624	D041400066
	C92430F	NA	0.9956	NA	0.9949	0.8640	479	20	8351	D051500045	8.35			NA	0.9510	Unknown	380	1	402	D041400074
	C92431F	NA	1.0113	NA	0.9949	0.8640	360	20	6178	D051500037	6.18			NA	0.9510	Unknown	465	1	484	D041400067
	C92439F	NA	1.0007	NA	0.9949	0.8640	570	20	9900	D051500041	9.90			NA	0.9510	Unknown	524	1	551	D041400068
	C92443F	High - Not Confirmed	0.9890	NA	0.9949	0.8640	934	20	16395	D051500042	16.4			NA	0.9510	Unknown	631	1	671	D041400069
	C92451F	NA	0.9974	NA	0.9949	0.8640	667	20	11614	D051500043	11.6		45.2	NA	0.9510	Unknown	464	1	490	D041400070
	C92452F	NA	0.9927	NA	0.9949	0.8640	856	20	14968	D051500044	15.0	9.63	4.35	NA	0.9510	Unknown	421	1	446	D041400073

A = Qualitative only

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
PFOSEA = Perfluorooctane sulfonyl ethylamide
M556 = C8F17SO2N((H)CH2COO)

Date Entered/Analyst: 05/01/00, 05/26/00 MMH, CSH
Date Verified/Analyst: 03/22/01 hoj, 04/03/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC / 04/03/01 KJH

AMD I# 050598.2
Covance# 6329-228

Study: TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance): T-6316 (EtFOSE-OH)
Matrix: Rat Liver
Method/Revision: ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number: Amelia 062498, Davey 070799
Instrument Software/Version: MassLynx 3.3 & 3.4
Date of Extraction/Analyst: 4/14/00 SAL/RWW
Date of Analysis/Analyst: 4/17/00, 5/15/00 IAS/MMH/IAS
Date of Data Reduction/Analyst: 4/18/00, 5/16/00, 05/08/01 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

WEEK 105 RAT LIVER

Concentration of PFOSA ug/g or % Rec	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	Group Dose	Sample #	Surrogate Verified	PFOSAA Old Purity Correction Factor (lot 98-0207-0303-3)	PFOSAA Purity Correction Factor	PFOSAA Conc. ug/g	PFOSAA Dilution Factor	PFOSAA Calc. Conc. ug/g	Filename	Concentration of PFOSAA ug/g or % Rec	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD	Surrogate Verified	EtFOSE Old Purity Correction Factor (lot SD013)	EtFOSE Purity Correction Factor	EtFOSE Conc. ug/g	EtFOSE Dilution Factor	EtFOSE Calc. Conc. ug/g
< LOQ (0.00644 ug/g)		NA	Method Blk	RBL041400-H2OBlk-1	NA	0.9760	Unknown	0.00	1	0.00	A041700004	< LOQ (0.0125 ug/g)		NA	NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)	<LOQ	NA		RBL041400-H2OBlk-2	NA	0.9760	Unknown	0.00	1	0.00	D041400004	< LOQ (0.0314 ug/g)	<LOQ	NA	NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.00644 ug/g)		NA	Matrix Blk	RBL041400-LiverBlk-1	NA	0.9760	Unknown	0.00	1	0.00	A041700005	< LOQ (0.0125 ug/g)		NA	NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)	<LOQ	NA		RBL041400-LiverBlk-2	NA	0.9760	Unknown	0.00	1	0.00	D041400005	< LOQ (0.0314 ug/g)	<LOQ	NA	NA	0.9770	0.8890	0.00	1	0.00
74%			QC	C92197/M/GB-MS-1	NA	0.9760	Unknown	224	1	215	D041400017	74%			NA	0.9770	0.8890	277	1	265
86%	82%	9%		C92197/M/GB-MSD-1	NA	0.9760	Unknown	260	1	249	D041400018	86%	80%	15%	NA	0.9770	0.8890	231	1	221
94%				C92195/M/GB-MS-1	NA	0.9760	Unknown	251	1	240	A041700017	82%			NA	0.9770	0.8890	472	1	452
63%	79%	39%		C92195/M/GB-MSD-1	NA	0.9760	Unknown	254	1	243	A041700018	83%	82%	2%	NA	0.9770	0.8890	459	1	439
92%				RBL041400-MS-1	NA	0.9760	Unknown	232	1	222	D041400075	73%			NA	0.9770	0.8890	NR	NR	NR
85%	88%	9%		RBL041400-MSD-1	NA	0.9760	Unknown	266	1	266	D041400076	87%	80%	18%	NA	0.9770	0.8890	NR	NR	NR
< LOQ (0.0322 ug/g)			Group 8	C92197M	NA	0.9760	Unknown	0.00	1	0.00	D041400019	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)			0.0 ppm	C92203M	NA	0.9760	Unknown	0.00	1	0.00	D041400020	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92208M	NA	0.9760	Unknown	0.00	1	0.00	D041400021	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92209M	NA	0.9760	Unknown	0.00	1	0.00	D041400024	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92218M	NA	0.9760	Unknown	0.00	1	0.00	D041400025	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92222M	NA	0.9760	Unknown	0.00	1	0.00	D041400026	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92231M	NA	0.9760	Unknown	0.00	1	0.00	D041400027	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92233M	NA	0.9760	Unknown	0.00	1	0.00	D041400028	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)		NA		C92239M	NA	0.9760	Unknown	0.00	1	0.00	D041400031	< LOQ (0.0314 ug/g)		NA	NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)	<LOQ	NA		C92252M	NA	0.9760	Unknown	0.00	1	0.00	D041400032	< LOQ (0.0314 ug/g)	<LOQ	NA	NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92333F	NA	0.9760	Unknown	0.00	1	0.00	D041400033	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92334F	NA	0.9760	Unknown	0.00	1	0.00	D041400034	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92348F	NA	0.9760	Unknown	0.00	1	0.00	D041400035	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92354F	NA	0.9760	Unknown	0.00	1	0.00	D041400038	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92358F	NA	0.9760	Unknown	0.00	1	0.00	D041400039	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92360F	NA	0.9760	Unknown	0.00	1	0.00	D041400040	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92362F	NA	0.9760	Unknown	0.00	1	0.00	D041400041	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)				C92369F	NA	0.9760	Unknown	0.00	1	0.00	D041400042	< LOQ (0.0314 ug/g)			NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)		NA		C92374F	NA	0.9760	Unknown	0.00	1	0.00	D041400045	< LOQ (0.0314 ug/g)		NA	NA	0.9770	0.8890	0.00	1	0.00
< LOQ (0.0322 ug/g)	<LOQ	NA		C92383F	NA	0.9760	Unknown	0.00	1	0.00	D041400046	< LOQ (0.0314 ug/g)	<LOQ	NA	NA	0.9770	0.8890	0.00	1	0.00
0.422			Group 9	C92261M	NA	0.9760	Unknown	574	1	591	D041400047	0.591			NA	0.9770	0.8890	0.00	1	0.00
0.280			1.0 ppm	C92266M	NA	0.9760	Unknown	383	1	396	D041400048	0.396			NA	0.9770	0.8890	0.00	1	0.00
0.829				C92270M	NA	0.9760	Unknown	112	20	2279	D051500019	2.28			NA	0.9770	0.8890	0.00	1	0.00
0.505				C92278M	NA	0.9760	Unknown	762	1	778	D041400052	0.778			NA	0.9770	0.8890	0.00	1	0.00
0.246				C92281M	NA	0.9760	Unknown	600	1	612	D041400053	0.612			NA	0.9770	0.8890	0.00	1	0.00
0.352				C92283M	NA	0.9760	Unknown	527	1	531	D041400054	0.531			NA	0.9770	0.8890	0.00	1	0.00
0.180				C92309M	NA	0.9760	Unknown	497	1	518	D041400055	0.518			NA	0.9770	0.8890	0.00	1	0.00
0.378				C92311M	NA	0.9760	Unknown	545	1	565	D041400056	0.565			NA	0.9770	0.8890	0.00	1	0.00
0.265		50.4		C92318M	NA	0.9760	Unknown	482	1	496	D041400059	0.496		74.9	NA	0.9770	0.8890	0.00	1	0.00
0.258	0.371	0.187		C92321M	NA	0.9760	Unknown	578	1	593	D041400060	0.593	0.736	0.551	NA	0.9770	0.8890	0.00	1	0.00
0.546				C92410F	NA	0.9760	Unknown	580	1	545	D041400061	0.545			NA	0.9770	0.8890	0.00	1	0.00
0.773				C92416F	NA	0.9760	Unknown	88.6	20	1811	D051500034	1.83			NA	0.9770	0.8890	0.00	1	0.00
0.512				C92419F	NA	0.9760	Unknown	420	1	424	D041400063	0.424			NA	0.9770	0.8890	0.00	1	0.00
0.624				C92423F	NA	0.9760	Unknown	813	1	835	D041400066	0.835			NA	0.9770	0.8890	60.4	1	55.1
0.402				C92430F	NA	0.9760	Unknown	482	1	496	D041400074	0.496			NA	0.9770	0.8890	0.00	1	0.00
0.484				C92431F	NA	0.9760	Unknown	698	1	707	D041400067	0.707			NA	0.9770	0.8890	0.00	1	0.00
0.551				C92439F	NA	0.9760	Unknown	404	1	414	D041400068	0.414			NA	0.9770	0.8890	0.00	1	0.00
0.671				C92443F	NA	0.9760	Unknown	753	1	780	D041400069	0.780			NA	0.9770	0.8890	0.00	1	0.00
0.490		20.3		C92451F	NA	0.9760	Unknown	862	1	885	D041400070	0.885		53.2	NA	0.9770	0.8890	0.00	1	0.00
0.446	0.550	0.112		C92452F	NA	0.9760	Unknown	772	1	796	D041400073	0.796	0.771	0.411	NA	0.9770	0.8890	0.00	1	0.00

A = Qualitative only

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PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
PFOSEA = Perfuoroctane sulfonyl ethylamide
M556 = C8F17SO2N((H)CH2COO)

Date Entered/Analyst: 05/01/00, 05/26/00 MMH, CSH
Date Verified/Analyst: 03/22/01 boj, 04/03/01 kjh, 5/22/01 mmh
Purity Entered/Verified: 03/29/01 LAC / 04/03/01 KJH

Covance# 6329-228

Filename	Concentration of EtFOSE ug/g or % Rec		Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD
A041700004	< LOQ (0.0559 ug/g)	A		NA
D041400004	< LOQ (0.0559 ug/g)	A	<LOQ	NA
A041700005	< LOQ (0.0559 ug/g)	A		NA
D041400005	< LOQ (0.0559 ug/g)	A	<LOQ	NA
D041400017	92%	A		
D041400018	77%	A	84%	18%
A041700017	154%	A		
A041700018	149%	A	151%	3%
NR	NR	A		
NR	NR	A	NR	NR
D041400019	< LOQ (0.0559 ug/g)	A		
D041400020	< LOQ (0.0559 ug/g)	A		
D041400021	< LOQ (0.0559 ug/g)	A		
D041400024	< LOQ (0.0559 ug/g)	A		
D041400025	< LOQ (0.0559 ug/g)	A		
D041400026	< LOQ (0.0559 ug/g)	A		
D041400027	< LOQ (0.0559 ug/g)	A		
D041400028	< LOQ (0.0559 ug/g)	A		
D041400031	< LOQ (0.0559 ug/g)	A		NA
D041400032	< LOQ (0.0559 ug/g)	A	<LOQ	NA
D041400033	< LOQ (0.0559 ug/g)	A		
D041400034	< LOQ (0.0559 ug/g)	A		
D041400035	< LOQ (0.0559 ug/g)	A		
D041400038	< LOQ (0.0559 ug/g)	A		
D041400039	< LOQ (0.0559 ug/g)	A		
D041400040	< LOQ (0.0559 ug/g)	A		
D041400041	< LOQ (0.0559 ug/g)	A		
D041400042	< LOQ (0.0559 ug/g)	A		
D041400045	< LOQ (0.0559 ug/g)	A		NA
D041400046	< LOQ (0.0559 ug/g)	A	<LOQ	NA
D041400047	< LOQ (0.0559 ug/g)	A		
D041400048	< LOQ (0.0559 ug/g)	A		
D041400049	< LOQ (0.0559 ug/g)	A		
D041400052	< LOQ (0.0559 ug/g)	A		
D041400053	< LOQ (0.0559 ug/g)	A		
D041400054	< LOQ (0.0559 ug/g)	A		
D041400055	< LOQ (0.0559 ug/g)	A		
D041400056	< LOQ (0.0559 ug/g)	A		
D041400059	< LOQ (0.0559 ug/g)	A		NA
D041400060	< LOQ (0.0559 ug/g)	A	<LOQ	NA
D041400061	< LOQ (0.0559 ug/g)	A		
D041400062	< LOQ (0.0559 ug/g)	A		
D041400063	< LOQ (0.0559 ug/g)	A		
D041400066	< LOQ (0.0559 ug/g)	A		
D041400074	< LOQ (0.0559 ug/g)	A		
D041400067	< LOQ (0.0559 ug/g)	A		
D041400068	< LOQ (0.0559 ug/g)	A		
D041400069	< LOQ (0.0559 ug/g)	A		
D041400070	< LOQ (0.0559 ug/g)	A		NA
D041400073	< LOQ (0.0559 ug/g)	A	<LOQ	NA

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats
Product Number(Test Substance):	T-6316 (EtFOSE-OH)
Matrix:	Rat Liver
Method/Revision:	ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number:	Ameba 062498, Davey 070799
Instrument Software/Version:	MassLynx 3.3 & 3.4
Date of Extraction/Analyst:	4/14/00 SAL/RWW
Date of Analysis/Analyst:	4/17/00, 5/15/00 IAS/MMH/IAS
Date of Data Reduction/Analyst:	4/18/00, 5/16/00, 05/08/01 IAS/MMH

Filename:	See Below
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments

Sample Data

Rat Liver week 104

lot NB1113047-80

Group Dose	Sample #	Surrogate Verified	M556 Old Purity Correction Factor	M556 Purity Correction Factor	M556 Conc. ug/g	M556 Dilution Factor	M556 Calc. Conc. ug/g	Filename	Concentration of M556 ug/g or % Rec	Mean M556 ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	RBL041400-H2OBlk-1	NA	0.9989	Unknown	0.00	1	0.00	A041700004	< LOQ (0.0307 ug/g)		NA
	RBL041400-H2OBlk-2	NA	0.9989	Unknown	0.00	1	0.00	D041400004	< LOQ (0.0307 ug/g)	<LOQ	NA
Matrix Blk	RBL041400-LiverBlk-1	NA	0.9989	Unknown	0.00	1	0.00	A041700005	< LOQ (0.0307 ug/g)		NA
	RBL041400-LiverBlk-2	NA	0.9989	Unknown	0.00	1	0.00	D041400005	< LOQ (0.0307 ug/g)	<LOQ	NA
QC	C92197/MGB-MS-1	NA	0.9989	Unknown	361	1	345	D041400017	119%		
	C92197/MGB-MSD-1	NA	0.9989	Unknown	370	1	354	D041400018	122%	121%	2.42%
	C92195/MGB-MS-1	NA	0.9989	Unknown	316	1	303	A041700017	103%		
	C92195/MGB-MSD-1	NA	0.9989	Unknown	331	1	317	A041700018	108%	105%	4.58%
	RBL041400-MS-1	NA	0.9989	Unknown	256	1	245	D041400075	80%		
	RBL041400-MSD-1	NA	0.9989	Unknown	257	1	257	D041400076	84%	82%	5%
Group 8 0.0 ppm	C92197M	NA	0.9989	Unknown	0.00	1	0.00	D041400019	< LOQ (0.0307 ug/g)		
	C92203M	NA	0.9989	Unknown	0.00	1	0.00	D041400020	< LOQ (0.0307 ug/g)		
	C92208M	NA	0.9989	Unknown	0.00	1	0.00	D041400021	< LOQ (0.0307 ug/g)		
	C92209M	NA	0.9989	Unknown	0.00	1	0.00	D041400024	< LOQ (0.0307 ug/g)		
	C92218M	NA	0.9989	Unknown	0.00	1	0.00	D041400025	< LOQ (0.0307 ug/g)		
	C92222M	NA	0.9989	Unknown	0.00	1	0.00	D041400026	< LOQ (0.0307 ug/g)		
	C92231M	NA	0.9989	Unknown	0.00	1	0.00	D041400027	< LOQ (0.0307 ug/g)		
	C92233M	NA	0.9989	Unknown	0.00	1	0.00	D041400028	< LOQ (0.0307 ug/g)		
	C92239M	NA	0.9989	Unknown	0.00	1	0.00	D041400031	< LOQ (0.0307 ug/g)		NA
	C92252M	NA	0.9989	Unknown	0.00	1	0.00	D041400032	< LOQ (0.0307 ug/g)	<LOQ	NA
	C92333F	NA	0.9989	Unknown	0.00	1	0.00	D041400033	< LOQ (0.0307 ug/g)		
	C92334F	NA	0.9989	Unknown	0.00	1	0.00	D041400034	< LOQ (0.0307 ug/g)		
	C92348F	NA	0.9989	Unknown	0.00	1	0.00	D041400035	< LOQ (0.0307 ug/g)		
	C92354F	NA	0.9989	Unknown	0.00	1	0.00	D041400038	< LOQ (0.0307 ug/g)		
	C92358F	NA	0.9989	Unknown	0.00	1	0.00	D041400039	< LOQ (0.0307 ug/g)		
	C92360F	NA	0.9989	Unknown	0.00	1	0.00	D041400040	< LOQ (0.0307 ug/g)		
	C92362F	NA	0.9989	Unknown	0.00	1	0.00	D041400041	< LOQ (0.0307 ug/g)		
	C92369F	NA	0.9989	Unknown	0.00	1	0.00	D041400042	< LOQ (0.0307 ug/g)		
	C92374F	NA	0.9989	Unknown	0.00	1	0.00	D041400045	< LOQ (0.0307 ug/g)		NA
	C92381F	NA	0.9989	Unknown	0.00	1	0.00	D041400046	< LOQ (0.0307 ug/g)	<LOQ	NA
Group 9 1.0 ppm	C92261M	NA	0.9989	Unknown	230	1	231	D041400047	0.231		
	C92266M	NA	0.9989	Unknown	314	1	317	D041400048	0.317		
	C92270M	NA	0.9989	Unknown	402	1	399	D041400049	0.399		
	C92278M	NA	0.9989	Unknown	313	1	312	D041400052	0.312		
	C92281M	NA	0.9989	Unknown	194	1	193	D041400053	0.193		
	C92283M	NA	0.9989	Unknown	306	1	300	D041400054	0.300		
	C92309M	NA	0.9989	Unknown	84	1	85.9	D041400055	0.0859		
	C92311M	NA	0.9989	Unknown	212	1	214	D041400056	0.214		
	C92318M	NA	0.9989	Unknown	240	1	241	D041400059	0.241		34.7
	C92321M	NA	0.9989	Unknown	201	1	202	D041400060	0.202	0.250	0.0865
	C92410F	NA	0.9989	Unknown	454	1	417	D041400061	0.417		
	C92416F	NA	0.9989	Unknown	486	1	491	D041400062	0.491		
	C92419F	NA	0.9989	Unknown	318	1	313	D041400063	0.313		
	C92423F	NA	0.9989	Unknown	385	1	386	D041400066	0.386		
	C92430F	NA	0.9989	Unknown	245	1	246	D041400074	0.246		
	C92431F	NA	0.9989	Unknown	267	1	265	D041400067	0.265		
	C92439F	NA	0.9989	Unknown	339	1	339	D041400068	0.339		
	C92443F	NA	0.9989	Unknown	302	1	306	D041400069	0.306		
	C92451F	NA	0.9989	Unknown	310	1	311	D041400070	0.311		22.2
	C92452F	NA	0.9989	Unknown	288	1	291	D041400073	0.291	0.336	0.0748

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Sample #	Surrogate Verified	PFOSEA Old Purity Correction Factor	PFOSEA Purity Correction Factor	PFOSEA Conc. ug/g	PFOSEA Dilution Factor	PFOSEA Calc. Conc. ug/g	Filename	Concentration of PFOSEA ug/g or % Rec
RBL041400-H2OBlk-1	NA	0.9930	Unknown	0.00	1	0.00	A041700004	< LOQ (0.0308 ug/g)
RBL041400-H2OBlk-2	NA	0.9930	Unknown	0.00	1	0.00	D041400004	< LOQ (0.0308 ug/g)
RBL041400-LiverBlk-1	NA	0.9930	Unknown	0.00	1	0.00	A041700005	< LOQ (0.0308 ug/g)
RBL041400-LiverBlk-2	NA	0.9930	Unknown	0.00	1	0.00	D041400005	< LOQ (0.0308 ug/g)
C92197/MGB-MS-1	NA	0.9930	Unknown	208	1	199	D041400017	69.2%
C92197/MGB-MSD-1	NA	0.9930	Unknown	254	1	243	D041400018	84.4%
C92195/MGB-MS-1	NA	0.9930	Unknown	442	1	423	A041700017	144%
C92195/MGB-MSD-1	NA	0.9930	Unknown	353	1	338	A041700018	115%
RBL041400-MS-1	NA	0.9930	Unknown	358	1	343	D041400075	112%
RBL041400-MSD-1	NA	0.9930	Unknown	393	1	376	D041400076	123%
C92197M	NA	0.9930	Unknown	0.00	1	0.00	D041400019	< LOQ (0.0308 ug/g)
C92203M	NA	0.9930	Unknown	0.00	1	0.00	D041400020	< LOQ (0.0308 ug/g)
C92208M	NA	0.9930	Unknown	0.00	1	0.00	D041400021	< LOQ (0.0308 ug/g)
C92209M	NA	0.9930	Unknown	0.00	1	0.00	D041400024	< LOQ (0.0308 ug/g)
C92218M	NA	0.9930	Unknown	0.00	1	0.00	D041400025	< LOQ (0.0308 ug/g)
C92222M	NA	0.9930	Unknown	0.00	1	0.00	D041400026	< LOQ (0.0308 ug/g)
C92231M	NA	0.9930	Unknown	0.00	1	0.00	D041400027	< LOQ (0.0308 ug/g)
C92233M	NA	0.9930	Unknown	0.00	1	0.00	D041400028	< LOQ (0.0308 ug/g)
C92239M	NA	0.9930	Unknown	0.00	1	0.00	D041400031	< LOQ (0.0308 ug/g)
C92252M	NA	0.9930	Unknown	0.00	1	0.00	D041400032	< LOQ (0.0308 ug/g)
C92333F	NA	0.9930	Unknown	0.00	1	0.00	D041400033	< LOQ (0.0308 ug/g)
C92334F	NA	0.9930	Unknown	0.00	1	0.00	D041400034	< LOQ (0.0308 ug/g)
C92348F	NA	0.9930	Unknown	0.00	1	0.00	D041400035	< LOQ (0.0308 ug/g)
C92354F	NA	0.9930	Unknown	0.00	1	0.00	D041400038	< LOQ (0.0308 ug/g)
C92358F	NA	0.9930	Unknown	0.00	1	0.00	D041400039	< LOQ (0.0308 ug/g)
C92360F	NA	0.9930	Unknown	0.00	1	0.00	D041400040	< LOQ (0.0308 ug/g)
C92362F	NA	0.9930	Unknown	0.00	1	0.00	D041400041	< LOQ (0.0308 ug/g)
C92369F	NA	0.9930	Unknown	0.00	1	0.00	D041400042	< LOQ (0.0308 ug/g)
C92374F	NA	0.9930	Unknown	0.00	1	0.00	D041400045	< LOQ (0.0308 ug/g)
C92381F	NA	0.9930	Unknown	0.00	1	0.00	D041400046	< LOQ (0.0308 ug/g)
C92261M	NA	0.9930	Unknown	0.00	1	0.00	D041400047	< LOQ (0.0308 ug/g)
C92266M	NA	0.9930	Unknown	0.00	1	0.00	D041400048	< LOQ (0.0308 ug/g)
C92270M	NA	0.9930	Unknown	0.00	1	0.00	D041400049	< LOQ (0.0308 ug/g)
C92278M	NA	0.9930	Unknown	0.00	1	0.00	D041400052	< LOQ (0.0308 ug/g)
C92281M	NA	0.9930	Unknown	0.00	1	0.00	D041400053	< LOQ (0.0308 ug/g)
C92283M	NA	0.9930	Unknown	0.00	1	0.00	D041400054	< LOQ (0.0308 ug/g)
C92309M	NA	0.9930	Unknown	0.00	1	0.00	D041400055	< LOQ (0.0308 ug/g)
C92311M	NA	0.9930	Unknown	0.00	1	0.00	D041400056	< LOQ (0.0308 ug/g)
C92318M	NA	0.9930	Unknown	0.00	1	0.00	D041400059	< LOQ (0.0308 ug/g)
C92321M	NA	0.9930	Unknown	0.00	1	0.00	D041400060	< LOQ (0.0308 ug/g)
C92410F	NA	0.9930	Unknown	0.00	1	0.00	D041400061	< LOQ (0.0308 ug/g)
C92416F	NA	0.9930	Unknown	0.00	1	0.00	D041400062	< LOQ (0.0308 ug/g)
C92419F	NA	0.9930	Unknown	0.00	1	0.00	D041400063	< LOQ (0.0308 ug/g)
C92423F	NA	0.9930	Unknown	0.00	1	0.00	D041400066	< LOQ (0.0308 ug/g)
C92430F	NA	0.9930	Unknown	0.00	1	0.00	D041400074	< LOQ (0.0308 ug/g)
C92431F	NA	0.9930	Unknown	0.00	1	0.00	D041400067	< LOQ (0.0308 ug/g)
C92439F	NA	0.9930	Unknown	0.00	1	0.00	D041400068	< LOQ (0.0308 ug/g)
C92443F	NA	0.9930	Unknown	0.00	1	0.00	D041400069	< LOQ (0.0308 ug/g)
C92451F	NA	0.9930	Unknown	0.00	1	0.00	D041400070	< LOQ (0.0308 ug/g)
C92452F	NA	0.9930	Unknown	0.00	1	0.00	D041400073	< LOQ (0.0308 ug/g)

A = Qualitative only

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
PFOSEA = Perfluorooctane sulfonyl ethylamide
M556 = C8F17SO2N((H)CH2COO)

Date Entered/Analyst:	05/01/00, 05/26/00 MMH, CSH
Date Verified/Analyst:	03/22/01 hoj, 04/03/01 kjh, 5/22/01 mmh
Purity Entered/Verified:	03/29/01 LAC / 04/03/01 KJH

Covance# 6329-228

Study:	TOX003, 104 Week Dietary Carcinogenicity Study with Narrow Range (98.1%) N-Ethyl Perfluorooctanesulfonamido Ethanol in Rats		
Product Number(Test Substance):	T-6316 (EtFOSE-OH)		
Matrix:	Rat Liver	Filename:	See Attachments
Method/Revision:	ETS-8-6.0 & ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Amelia 062498, Davey 070799	Slope:	See Attachments
Instrument Software/Version:	MassLynx 3.3 & 3.4	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	4/14/00 SAL/RWW		
Date of Analysis/Analyst:	4/17/00, 5/15/00 IAS/MMH/IAS		
Date of Data Reduction/Analyst:	4/18/00, 5/16/00, 05/08/01 IAS/MMH		

Sample Data

WEEK 105 RAT LIVER

Mean PFOSEA ug/g	RSD Std. Dev. MS/MSD RPD	Group Dose	Sample #	PFOS Calc. Conc. ug/g	Concentration of PFOS ug/g or % Rec	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	PFOSA Calc. Conc. ug/g	Concentration of PFOSA ug/g or % Rec	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	PFOSAA Calc. Conc. ug/g	Concentration of PFOSAA ug/g or % Rec	Mean PFOSAA ug/g	RSD Std. Dev. MS/MSD RPD	EtFOSE Calc. Conc. ug/g	Concentration of EtFOSE ug/g or % Rec		Mean EtFOSE ug/g	RSD Std. Dev. MS/MSD RPD
	NA	Method Blk	RBL041400-H2OBlk-1	0.00	< LOQ (0.0267 ug/g)			2.51	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
<LOQ	NA		RBL041400-H2OBlk-2	0.00	< LOQ (0.0107 ug/g)	<LOQ	NA	0.00	< LOQ (0.0322 ug/g)	<LOQ	NA	0.00	< LOQ (0.0314 ug/g)	<LOQ	NA	0.00	< LOQ (0.0559 ug/g)	A	<LOQ	NA
	NA	Matrix Blk	RBL041400-LiverBlk-1	0.00	< LOQ (0.0267 ug/g)			2.20	< LOQ (0.00644 ug/g)			0.00	< LOQ (0.0125 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
<LOQ	NA		RBL041400-LiverBlk-2	4.77	< LOQ (0.0107 ug/g)	<LOQ	NA	0.00	< LOQ (0.0322 ug/g)	<LOQ	NA	0.00	< LOQ (0.0314 ug/g)	<LOQ	NA	0.00	< LOQ (0.0559 ug/g)	A	<LOQ	NA
		QC - 300 ppb	C92197/M/G8-MS-1	226	78%			224	78%			215	74%			265	92%	A		
77%	20%		C92197/M/G8-MSD-1	224	78%	78%	1%	246	86%	82%	9%	249	86%	80%	15%	221	77%	A	84%	18%
			C92195/M/G8-MS-1	403	137%			280	94%			240	82%			452	154%	A		
130%	22%		C92195/M/G8-MSD-1	324	110%	124%	22%	188	63%	79%	39%	243	83%	82%	2%	439	149%	A	151%	3%
			RBL041400-MS-1	232	76%			282	92%			222	73%			NR	NR	A		
117%	9%		RBL041400-MSD-1	261	85%	80%	12%	259	85%	88%	9%	266	87%	80%	18%	NR	NR	A	NR	NR
		Group 8	C92197M	44.1	0.0441			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
		0.0 ppm	C92203M	2.94	< LOQ (0.0107 ug/g)			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92208M	8.06	< LOQ (0.0107 ug/g)			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92209M	0.00	< LOQ (0.0107 ug/g)			0.24	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92218M	73.2	0.0732			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92222M	3.43	< LOQ (0.0107 ug/g)			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92231M	2.41	< LOQ (0.0107 ug/g)			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92233M	0.00	< LOQ (0.0107 ug/g)			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
	NA		C92239M	225	0.225		141.4	0.00	< LOQ (0.0322 ug/g)		NA	0.00	< LOQ (0.0314 ug/g)		NA	0.00	< LOQ (0.0559 ug/g)	A		NA
<LOQ	NA		C92252M	246	0.246	0.065	0.092	0.00	< LOQ (0.0322 ug/g)	<LOQ	NA	0.00	< LOQ (0.0314 ug/g)	<LOQ	NA	0.00	< LOQ (0.0559 ug/g)	A	<LOQ	NA
			C92333F	109	0.109			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92334F	93.0	0.0930			0.37	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92348F	153	0.153			0.09	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92354F	13.8	0.0138			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92358F	6.42	< LOQ (0.0107 ug/g)			0.09	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92360F	88.4	0.0884			0.26	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92362F	29.9	0.0299			0.54	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
			C92369F	112	0.112			0.00	< LOQ (0.0322 ug/g)			0.00	< LOQ (0.0314 ug/g)			0.00	< LOQ (0.0559 ug/g)	A		
	NA		C92374F	111	0.111		59.9	0.46	< LOQ (0.0322 ug/g)		NA	0.00	< LOQ (0.0314 ug/g)		NA	0.00	< LOQ (0.0559 ug/g)	A		NA
<LOQ	NA		C92383F	150	0.150	0.08717	0.0522	0.32	< LOQ (0.0322 ug/g)	<LOQ	NA	0.00	< LOQ (0.0314 ug/g)	<LOQ	NA	0.00	< LOQ (0.0559 ug/g)	A	<LOQ	NA
		Group 9	C92261M	5037	5.04			422	0.422			591	0.591			0.00	< LOQ (0.0559 ug/g)	A		
		1.0 ppm	C92266M	7515	7.51			280	0.280			396	0.396			0.00	< LOQ (0.0559 ug/g)	A		
			C92270M	12702	12.7			829	0.829			2279	2.279			0.00	< LOQ (0.0559 ug/g)	A		
			C92278M	3781	3.78			505	0.505			778	0.778			0.00	< LOQ (0.0559 ug/g)	A		
			C92281M	4660	4.66			246	0.246			612	0.612			0.00	< LOQ (0.0559 ug/g)	A		
			C92283M	10841	10.8			352	0.352			531	0.531			0.00	< LOQ (0.0559 ug/g)	A		
			C92309M	568	0.568			180	0.180			518	0.518			0.00	< LOQ (0.0559 ug/g)	A		
			C92311M	2084	2.08			378	0.378			565	0.565			0.00	< LOQ (0.0559 ug/g)	A		
	NA		C92318M	8678	8.68		61.2	265	0.265		50.4	496	0.496		74.9	0.00	< LOQ (0.0559 ug/g)	A		NA
<LOQ	NA		C92321M	6249	6.25	6.21	3.80	258	0.258	0.371	0.187	593	0.593	0.736	0.551	0.00	< LOQ (0.0559 ug/g)	A	<LOQ	NA
			C92410F	10717	10.7			546	0.546			545	0.545			0.00	< LOQ (0.0559 ug/g)	A		
			C92416F	10911	10.9			773	0.773			1831	1.831			0.00	< LOQ (0.0559 ug/g)	A		
			C92419F	2177	2.18			512	0.512			424	0.424			0.00	< LOQ (0.0559 ug/g)	A		
			C92423F	5114	5.11			624	0.624			835	0.835			55.1	< LOQ (0.0559 ug/g)	A		
			C92430F	8351	8.35			402	0.402			496	0.496			0.00	< LOQ (0.0559 ug/g)	A		
			C92431F	6178	6.18			484	0.484			707	0.707			0.00	< LOQ (0.0559 ug/g)	A		
			C92439F	9900	9.90			551	0.551			414	0.414			0.00	< LOQ (0.0559 ug/g)	A		
			C92443F	16395	16.4			671	0.671			780	0.780			0.00	< LOQ (0.0559 ug/g)	A		
	NA		C92451F	11614	11.6		45.2	490	0.490		20.3	885	0.885		53.2	0.00	< LOQ (0.0559 ug/g)	A		NA
<LOQ	NA		C92452F	14968	15.0	9.63	4.35	446	0.446	0.550	0.112	796	0.796	0.771	0.411	0.00	< LOQ (0.0559 ug/g)	A	<LOQ	NA

A = Qualitative only

NR = Not Reported
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFOSAA = Perfluorooctanesulfonamidoacetate
EtFOSE = Narrow Range N-Ethyl Perfluorooctanesulfonamido ethyl alcohol
PFOSEA = Perfluorooctane sulfonyl ethylamide
M556 = C8F17SO2N((H)CH2COO)

Date Entered/Analyst:	05/01/00, 05/26/00 MMH, CSH
Date Verified/Analyst:	03/22/01 boj, 04/03/01 kjh, 5/22/01 mmh
Purity Entered/Verified:	03/29/01 LAC / 04/03/01 KJH

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Appendix F: Example Calculations

Formula Used for Sera Analyses in Study FACT TOX-003

$$\text{AR (ng/mL)} \times \text{DF} \times \frac{\text{PC2}}{\text{PC1}} \times \frac{\text{FV (mL)}}{\text{EV (mL)}} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \textbf{Reported Concentration (µg/mL)}$$

Calculation Used for Group 9, Week 105, Animal ID C92430/F

$$199\ \text{ng/mL} \times 10 \times \frac{0.8640}{0.9949} \times \frac{1\ \text{mL}}{1\ \text{mL}} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \mathbf{1.73\ \mu g/mL}$$

AR— Analytical result from MassLynx summary
DF— Dilution factor
PC1—Purity Correction–1
PC2—Purity Correction–2
FV—Final extract volume (1.0 mL unless otherwise noted)
EV—Volume of sera extracted

Formula Used for Liver Analyses in Study FACT TOX-003

$$\text{AR (ng/g)} \times \frac{\partial\ \text{curve}}{\partial\ \text{sample}}^{(1)} \times \text{SC} \times \text{DF} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \textbf{Reported Concentration (µg/g)}$$

(1) ∂ curve is assumed to be: $\frac{1\ \text{g liver}}{5\ \text{mL}\ H_2O}$

Calculation Used for Group 9, Week 53, Animal ID C92267M

$$497\ \text{ng/g} \times \frac{1\ \text{g/ 5 mL}}{1.0137\ \text{g/ 5mL}} \times \frac{0.8640}{0.9949} \times 100 \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \mathbf{42.6\ \mu g/g}$$

AR— Analytical result from MassLynx summary
∂ curve—Density of the liver standard curve, assumed to be 1g liver/ 5 ml water
∂ sample—Density of the liver sample (g sample/ 5 mL H_2O)
PC1—Purity Correction–1
PC2—Purity Correction–2
DF— Dilution factor

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Appendix F: Example Calculations

Formula Used for Sera Analyses in Study FACT TOX-003

$$\text{AR (ng/mL)} \times \text{DF} \times \frac{\text{PC2}}{\text{PC1}} \times \frac{\text{FV (mL)}}{\text{EV (mL)}} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \textbf{Reported Concentration (µg/mL)}$$

Calculation Used for Group 9, Week 105, Animal ID C92430/F

$$199\ \text{ng/mL} \times 10 \times \frac{0.8640}{0.9949} \times \frac{1\ \text{mL}}{1\ \text{mL}} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \mathbf{1.73\ \mu g/mL}$$

AR— Analytical result from MassLynx summary
DF— Dilution factor
PC1—Purity Correction–1
PC2—Purity Correction–2
FV—Final extract volume (1.0 mL unless otherwise noted)
EV—Volume of sera extracted

Formula Used for Liver Analyses in Study FACT TOX-003

$$\text{AR (ng/g)} \times \frac{\partial\ \text{curve}}{\partial\ \text{sample}}^{(1)} \times \text{SC} \times \text{DF} \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \textbf{Reported Concentration (µg/g)}$$

(1) ∂ curve is assumed to be: $\frac{1\ \text{g liver}}{5\ \text{mL}\ H_2O}$

Calculation Used for Group 9, Week 53, Animal ID C92267M

$$497\ \text{ng/g} \times \frac{1\ \text{g/ 5 mL}}{1.0137\ \text{g/ 5mL}} \times \frac{0.8640}{0.9949} \times 100 \times \frac{1.0\ \mu\text{g}}{1000\ \text{ng}} = \mathbf{42.6\ \mu g/g}$$

AR— Analytical result from MassLynx summary
∂ curve—Density of the liver standard curve, assumed to be 1g liver/ 5 ml water
∂ sample—Density of the liver sample (g sample/ 5 mL H_2O)
PC1—Purity Correction–1
PC2—Purity Correction–2
DF— Dilution factor

3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Appendix G: Interim Certificates of Analysis

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

INTERIM CERTIFICATE OF ANALYSIS

Revision 1(9/7/00)

Centre Analytical Laboratories COA Reference #: 023-018B
3M Product: PFOS, Lot 171
Reference #: SD-009
Purity: 86.4%

Test Name	Specifications	Result
Purity[1]		**86.4%**
Appearance	White Crystalline Powder	Conforms
Identification NMR		Positive
Metals (ICP/MS) 1. Calcium 2. Magnesium 3. Sodium 4. Potassium[2] 5. Nickel 6. Iron 7. Manganese		 1. 0.017 wt./wt.% 2. 0.007 wt./wt.% 3. 1.355 wt./wt.% 4. 6.552 wt./wt.% 5. 0.003 wt./wt.% 6. 0.004 wt./wt.% 7. <0.001 wt./wt.%
Total % Impurity (NMR)		1.00 wt./wt.%
Total % Impurity (LC/MS)		10.60 wt./wt.%
Total % Impurity (GC/MS)		None Detected
Related Compounds – POAA		0.30 wt./wt.%
Residual Solvents (TGA)		None Detected
Purity by DSC		Not Applicable[3]
Inorganic Anions (IC) 1. Chloride 2. Fluoride 3. Bromide 4. Nitrate 5. Nitrite 6. Phosphate 7. Sulfate[4]		 1. <0.015 wt./wt.% 2. 0.27 wt./wt.% 3. <0.040 wt./wt.% 4. <0.009 wt./wt.% 5. <0.006 wt./wt.% 6. <0.007 wt./wt.% 7. 8.82 wt./wt.%
Organic Acids[5] (IC) 1. TFA 2. PFPA 3. HFBA 4. NFPA		 1. <0.1 wt./wt.% 2. <0.1 wt./wt.% 3. <0.1 wt./wt.% 4. <0.25 wt./wt.%
Elemental Analysis[6]: 1. Carbon 2. Hydrogen 3. Nitrogen 4. Sulfur 5. Fluorine	 1. Theoretical Value = 17.8% 2. Theoretical Value = 0% 3. Theoretical Value = 0% 4. Theoretical Value = 5.95% 5. Theoretical Value = 60%	 1. 12.08 wt./wt.% 2. 0.794 wt./wt.% 3. 1.61 wt./wt.% 4. 10.1 wt./wt.% 5. 50.4 wt./wt.%

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-018B

Date of Last Analysis: 08/31/00

Expiration Date: 08/31/01

Storage Conditions: Frozen ≤-10°C

Re-assessment Date: 08/31/01

[1]Purity = 100% - (sum of metal impurities, 1.39% +LC/MS impurities, 10.60%+Inorganic Fluoride, 0.27%+NMR impurities, 1.00%+ POAA, 0.30%)

Total impurity from all tests = 13.56%

Purity = 100% - 13.56% = 86.4%

[2]Potassium is expected in this salt form and is therefore not considered an impurity.

[3]Purity by DSC is generally not applicable to materials of low purity. No endotherm was observed for this sample.

[4]Sulfur in the sample appears to be converted to SO_4 and hence detected using the inorganic anion method conditions. The anion result agrees well with the sulfur determination in the elemental analysis, lending confidence to this interpretation. Based on the results, the SO_4 is not considered an impurity.

[5]TFA	Trifluoroacetic acid
HFBA	Heptafluorobutyric acid
NFPA	Nonofluoropentanoic acid
PFPA	Pentafluoropropanoic acid

[6]Theoretical value calculations based on the empirical formula, $C_8F_{17}SO_3^-K^+$ (MW=538)

This work was conducted under EPA Good Laboratory Practice Standards (40 CFR 160).

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-018B

LC/MS Purity Profile:

Impurity	wt./wt. %
C4	1.03
C5	1.56
C6	6.38
C7	1.63
Total	**10.60**

Note: The C4 and C6 values were calculated using the C4 and C6 standard calibration curves, respectively. The C5 value was calculated using the average response factors from the C4 and C6 standard curves. Likewise, the C7 value was calculated using the average response factors from the C6 and C8 standard curves.

Prepared By: _____ 9/10/00
David S. Bell Date
Scientist, Centre Analytical Laboratories

Reviewed By: _____ 9/11/00
John Flaherty Date
Laboratory Manager, Centre Analytical Laboratories

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-2
3M Product: EtFOSE-OH
Test Control Reference #: TCR-00017-52
Purity: 97.4%

Test Name	Specifications	Result
Purity[1]		**97.4%**
Appearance	Yellow-white, waxy solid	Conforms
Identification NMR		Positive
Metals (ICP/MS) 1. Calcium 2. Magnesium 3. Sodium 4. Potassium 5. Nickel 6. Iron 7. Manganese		 1. <0.001 wt./wt.% 2. <0.001 wt./wt.% 3. <0.001 wt./wt.% 4. <0.001 wt./wt.% 5. <0.001 wt./wt.% 6. <0.001 wt./wt.% 7. <0.001 wt./wt.%
Total % Impurity (NMR)		1.26 wt./wt.%
Total % Impurity (LC/MS)		None Quantified
Total % Impurity (GC/MS)		1.29 wt./wt.%
Related Compounds – POAA		0.10 wt./wt.%
Residual Solvents (TGA)		None Detected
Purity by DSC		90.3 wt./wt.%.
Inorganic Anions (IC) 1. Chloride 2. Fluoride 3. Bromide 4. Nitrate 5. Nitrite 6. Phosphate 7. Sulfate		 1. <0.015 wt./wt.% 2. <0.005 wt./wt.% 3. <0.040 wt./wt.% 4. <0.009 wt./wt.% 5. <0.006 wt./wt.% 6. <0.007 wt./wt.% 7. <0.154 wt./wt.%
Organic Acids[2] (IC) 1. TFA 2. PFPA 3. HFBA 4. NFPA		 1. <0.1 wt./wt.% 2. <0.1 wt./wt.% 3. <0.1 wt./wt.% 4. <0.25 wt./wt.%
Elemental Analysis[3]: 1. Carbon 2. Hydrogen 3. Nitrogen 4. Sulfur 5. Fluorine	 1. Theoretical Value = 25.2% 2. Theoretical Value = 1.75% 3. Theoretical Value = 2.45% 4. Theoretical Value = 5.60% 5. Theoretical Value = 56.6%	 1. 25.04 wt./wt.% 2. 1.69 wt./wt.% 3. 2.61 wt./wt.% 4. 8.88 wt./wt.% 5. 56.8 wt./wt.%

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-2
3M Product: EtFOSE-OH
Test Control Reference #: TCR-00017-52

Date of Last Analysis: 11/26/00

Expiration Date: 11/26/01

Storage Conditions: <-10 °C

Re-assessment Date: 11/26/01

[1]Purity = 100% - (total NMR impurities, 1.26% + GC/MS impurities, 1.29 + POAA, 0.10%)

Total impurity from all tests = 2.65%
Purity = 100% - 2.65% = 97.4%

[2]TFA	Trifluoroacetic acid
HFBA	Heptafluorobutyric acid
NFPA	Nonafluoropentanoic acid
PFPA	Pentafluoropropanoic acid

[3]Theoretical value calculations based on the empirical formula, $C_{12}H_{10}F_{17}NO_3S$ (MW=571)

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-2
3M Product: EtFOSE-OH
Test Control Reference #: TCR-00017-52

GC/MS Purity Profile

Peak #	Retention Time (min)	Identity	% Impurity
1	13.934	PFOSDEA	0.36
2	17.307	C7	0.93
Total	-	-	**1.29**

This work was conducted under EPA Good Laboratory Practice Standards (40 CFR 160).

Prepared By: _____ 11/27/00
David S. Bell Date
Scientist
Centre Analytical Laboratories

Reviewed By: _____ 11/27/00
John Flaherty Date
Laboratory Manager
Centre Analytical Laboratories

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

INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-1
3M Product: EtFOSE-OH
Test Control Reference #: SD-013
Purity: 88.9%

Test Name	Specifications	Result
Purity[1]		**88.9%**
Appearance	Yellow-white, waxy solid	Conforms
Identification NMR		 Positive
Metals (ICP/MS) 1. Calcium 2. Magnesium 3. Sodium 4. Potassium 5. Nickel 6. Iron 7. Manganese		 1. <0.001 wt./wt.% 2. <0.001 wt./wt.% 3. <0.001 wt./wt.% 4. 0.002 wt./wt.% 5. <0.001 wt./wt.% 6. <0.001 wt./wt.% 7. <0.001 wt./wt.%
Total % Impurity (NMR)		0.90 wt./wt.%
Total % Impurity (LC/MS)		None Quantified
Total % Impurity (GC/MS)		10.21 wt./wt.%
Related Compounds – POAA		0.03 wt./wt.%
Residual Solvents (TGA)		None Detected
Purity by DSC		87.6 wt./wt.%.
Inorganic Anions (IC) 1. Chloride 2. Fluoride 3. Bromide 4. Nitrate 5. Nitrite 6. Phosphate 7. Sulfate		 1. <0.015 wt./wt.% 2. <0.005 wt./wt.% 3. <0.040 wt./wt.% 4. <0.009 wt./wt.% 5. <0.006 wt./wt.% 6. <0.007 wt./wt.% 7. <0.040 wt./wt.%
Organic Acids[2] (IC) 1. TFA 2. PFPA 3. HFBA 4. NFPA		 1. <0.1 wt./wt.% 2. <0.1 wt./wt.% 3. <0.1 wt./wt.% 4. <0.25 wt./wt.%
Elemental Analysis[3]: 1. Carbon 2. Hydrogen 3. Nitrogen 4. Sulfur 5. Fluorine	 1. Theoretical Value = 25.2% 2. Theoretical Value = 1.75% 3. Theoretical Value = 2.45% 4. Theoretical Value = 5.60% 5. Theoretical Value = 56.6%	 1. 24.42 wt./wt.% 2. 1.78 wt./wt.% 3. 2.72 wt./wt.% 4. 9.34 wt./wt.% 5. 58.4 wt./wt.%

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-1
3M Product: EtFOSE-OH
Test Control Reference #: SD-013

Date of Last Analysis: 11/26/00

Expiration Date: 11/26/01

Storage Conditions: <-10 °C

Re-assessment Date: 11/26/01

[1]Purity = 100% - (total metal impurities, 0.002% + total NMR impurities, 0.90% + GC/MS impurities, 10.21 + POAA, 0.03%)

Total impurity from all tests = 11.14%
Purity = 100% - 11.14% = 88.9%

[2]TFA	Trifluoroacetic acid
HFBA	Heptafluorobutyric acid
NFPA	Nonafluoropentanoic acid
PFPA	Pentafluoropropanoic acid

[3]Theoretical value calculations based on the empirical formula, $C_{12}H_{10}F_{17}NO_3S$ (MW=571)

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INTERIM CERTIFICATE OF ANALYSIS

Centre Analytical Laboratories COA Reference #: 023-022-1
3M Product: EtFOSE-OH
Test Control Reference #: SD-013

GC/MS Purity Profile

Peak #	Retention Time (min)	Identity	% Impurity
1	6.163	Unknown	0.12
2	8.011	Unknown	0.23
3	8.206	Unknown	0.51
4	9.065	Unknown	0.21
5	9.844	Unknown	0.34
6	13.93	Unknown	0.62
7	14.238	Unknown	0.11
8	15.130	C2	0.11
9	15.52	C3	1.11
10	15.941	C4	1.55
11	16.379	C5	1.07
12	16.801	C6	3.30
13	17.222	C7	0.93
Total	-	-	**10.21**

This work was conducted under EPA Good Laboratory Practice Standards (40 CFR 160).

Prepared By: _____ 11/27/00
David S. Bell Date
Scientist
Centre Analytical Laboratories

Reviewed By: _____ 11/27/00
John Flaherty Date
Laboratory Manager
Centre Analytical Laboratories

ANALYTICAL REPORT

STUDY TITLE

PURITY DETERMINATION OF SAMPLE LOTS OF PFOS
Sample # TCR-00065-022

DATA REQUIREMENTS

Test Article Characterization

STUDY DIRECTOR

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BY STE DATE 10/25/00

Kevin Lloyd

ANALYTICAL REPORT COMPLETION DATE

October 25, 2000

PERFORMING LABORATORIE / TESTING FACILITIES

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

STUDY SPONSOR

3M Environmental Technology and Safety Services
Building 2-3E-09
PO Box 33331
St. Paul, MN 55133-3331

PROJECT IDENTIFICATION

Centre Study Number: 023-045

Total Pages: 15

Centre Study No.: 023-045

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

Centre Study Number 023-045, entitled "Purity Determination of Sample Lots of PFOS, Sample # TCR-00065-022" conducted for 3M Environmental Laboratory, was performed in compliance with US EPA Good Laboratory Practice Standards (40 CFR Part 160) by Centre Analytical Laboratories, Inc. with the following exceptions:

> The automated data collection systems used in this study were not fully compliant with 21 CFR 58.130 (e).

Kevin Lloyd
Study Director
Centre Analytical Laboratories, Inc.

10/25/00
Date

James K. Lundberg, Ph.D.
Sponsor Representative
3M Environmental Technology and Safety Services

10/27/00
Date

STE 10/25/00

Centre Study No.: 023-045

QUALITY ASSURANCE STATEMENT

Centre Study Number 023-045, entitled "Purity Determination of Sample Lots of PFOS, Sample # TCR-00065-022" was reviewed by Centre Analytical Laboratories' Quality Assurance Unit. All reviewed phases were reviewed for conduct according to Centre Analytical Laboratories' Standard Operating Procedures, the Study Protocol, and all applicable Good Laboratory Practice Standards. All findings were reported to the Study Director and to management.

Phase	Date Inspected	Date Reported to Study Director and Centre Management	Date Reported to Sponsor Management
1. Protocol Review	10/19/00	10/25/00	10/25/00
2. Raw Data Review	10/19/00	10/25/00	10/25/00
3. Report Review	10/24/00	10/25/00	10/25/00

William Spare
Quality Assurance Officer

10/25/00
Date

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Centre Study No.: 023-045

CERTIFICATION OF AUTHENTICITY

This report, for Centre Study Number 023-045, is a true and complete representation of the raw data for the study.

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

Study Director, Centre:

10/25/00

Kevin Lloyd
Study Director
Centre Analytical Laboratories, Inc.

Date

Centre Analytical Laboratories, Inc. Facility Management:

25-OCT-00

Richard A. Grazzini, Ph.D.
President
Centre Analytical Laboratories, Inc.

Date

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Centre Study No.: 023-045

STUDY IDENTIFICATION

PURITY DETERMINATION OF SAMPLE LOTS OF PFOS
Sample # TCR-00065-022

TYPE OF STUDY:	Characterization
TEST SYSTEM:	Not Applicable
TEST ARTICLE:	PFOS, Test Control Reference # TCR-00065-022
SPONSOR:	3M Environmental Technology and Safety Services Building 2-3E-09 PO Box 33331 St. Paul, MN 55133-3331
STUDY DIRECTOR:	Kevin Lloyd Center Analytical Laboratories, Inc. Phone: (814) 231-8032
TESTING FACILITIES:	Centre Analytical Laboratories, Inc. (Centre) 3048 Research Drive State College, PA 16801 Phone: 814-231-8032
ANALYTICAL PHASE TIMETABLE:	Study Initiation Date: 09/28/00 Analytical Start Date: 10/05/00 Analytical Termination Date: 10/24/00

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Centre Study No.: 023-045

PROJECT PERSONNEL

The Study Director for this project was Kevin Lloyd at Centre Analytical Laboratories, Inc. The following personnel from Centre Analytical Laboratories, Inc., were associated with various phases of the study:

Name	Title
Gerry Shero	Scientist
David S. Bell	Scientist
Emily R. Stauffer	Scientist
Mark Ammerman	Sample Custodian

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Figure 1. PFOS Calibration Standard (C100500-4) at 269µg/L 12
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Appendix A: Study Protocol 00P-023-045: PURITY DETERMINATION OF SAMPLE LOTS OF PFOS, Sample # TCR-00065-022, Including Amendment 1 15

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1.0 SUMMARY

The purity of PFOS from sample # TCR-00065-022, was determined from the LC/MS test data to be 88.0% pure as compared with the PFOS control (TCR-00017-046, 97.9% purity).

2.0 INTRODUCTION

This report details the results of the analysis of PFOS, Test Control Reference# TCR-00065-022. The test was analysis by liquid chromatography – mass spectrometry (LC/MS). The LC/MS was conducted at Centre.

The study was initiated on September 28, 2000 when the Study Director signed protocol number 00P-023-045. The analytical start date was October 5, 2000, and the experimental termination date was October 9, 2000.

3.0 TEST SYSTEM

There is no test system associated with this characterization study, therefore the GLP requirement for test system description, justification, and identification do not apply. The route of administration, levels and frequency of administration also do not apply to characterization studies.

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4.0 TEST ARTICLE

The test article was PFOS, Test Control Reference# TCR-00065-022. 3M Environmental Laboratory supplied the test article and it was logged at Centre Analytical Laboratories, Inc. as follows:

Compound	Lot or NB Number	Centre Control No.	Purity	Date Received
PFOS TCR-00065-022	193	00-023-068	TBD	10/05/00

The sample received was a white, crystalline solid. It was received under frozen conditions and was stored in a temperature-monitored freezer kept at <-10°C.

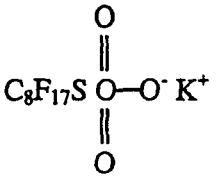
The analytical/control standard PFOS was:

Control Article	Lot Number	Centre Control No.	Purity	Expiration Date
PFOS	TCR-00017-046	00-023-042	97.9%	8/31/2001

The chemical and physical data for PFOS is as follows.

Common Name: PFOS
Molecular weight: 499 ($C_8F_{17}SO_3^-$)
CAS Number: 2795-39-3

Structure:



Note: the neutral molecule and standard form that the PFOS (anion) is derived from is potassium perfluorooctane sulfonate ($C_8F_{17}SO_3K$), molecular weight 538.

5.0 EXPERIMENTAL PROCEDURES

LC/MS Spectral Analysis

PFOS sample TCR-00065-022 was analyzed for purity using liquid chromatography/mass spectrometry (LC/MS). The sample was analyzed according to the following procedure.

The sample was prepared at about 250 µg/mL in methanol and analyzed by LC/MS using a Hewlett-Packard 1100 HPLC system interfaced with a Hewlett-Packard 1100 mass selective detector. The mass spectrometer was run in negative ionization mode using electrospray ionization (ESI) using Selective Ionization Monitoring for ion m/z 499. The liquid chromatography system was operated in reversed-phase mode using a C18 silica-based column.

Each resulting chromatogram was calculated against the analytical control article (TCR-00017-046) calibration curve.

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6.0 RESULTS AND DISCUSSION

LC/MS Spectral Analysis

Responses were observed for the PFOS sample in negative ion mode using an ESI interface. Quantitation was performed using SIM mode, m/z 499, and PFOS was detected at 7.8 minutes. TH-PFOS was used as the internal standard, m/z 427, and was detected at 7.5 minutes. The total percent purity, calculated against the analytical control article (TCR-00017-046) calibration curve was determined to be 88.0% from the average of three replicate analyses.

8.0 CIRCUMSTANCES THAT MAY HAVE AFFECTED THE DATA

Electronic records are not fully compliant with 21 CFR 11, "Electronic records; Electronic Signature." However, approved SOPs were in place and all instrumentation used in this study was fully calibrated and operational. All original raw data were printed as hard copies and fully audited by quality assurance. Verified exact copies and the electronic data will be stored in the archives at Centre Analytical Laboratories. Original raw data will be returned to the Sponsor.

9.0 RETENTION OF DATA AND SAMPLES

When the final report is final, all original paper data generated by Centre Analytical Laboratories, Inc. will be shipped to the sponsor. This does not include facility-specific raw data such as instrument logs, however exact copies of temperature logs will be submitted. Exact copies of all raw data, as well as a signed copy of the final analytical report and all original facility-specific raw data, will be retained in the Centre Analytical Laboratories, Inc. archives for the period of time specified in 40 CFR Part 160. Retained samples of reference substances are archived by the sponsor.

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Table I. Results From LC/MS Of TCR-00065-022, ESI Negative mode

3M ID	Retention Time (min)	Mass	Value µg/L	Determined µg/L	% Recovery
TCR-00065-022	7.79	499	250	219	88.0
TCR-00065-022	7.80	499	250	220	88.0
TCR-00065-022	7.80	499	250	220	88.0
				Average =	**88.0**
Standard	7.69	499	11	100*	*
Standard	7.73	499	11	17*	*
Standard	7.77	499	54	60	110
Standard	7.79	499	54	50	93
Standard	7.79	499	108	101	94
Standard	7.77	499	269	284	105
Standard	7.78	499	539	562	104
Standard	7.78	499	808	831	103
Standard	7.78	499	1078	1205	112
CCV	7.81	499	269	278	103
				Standards Average =	**103**
				Std Dev =	7

* First two injections during instrument warm up, not reported.
CCV = standard in run used to check calibration (C100500-4 at 269µg/L)

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Figure 1. PFOS Calibration Standard (C100500-4) at 269µg/L

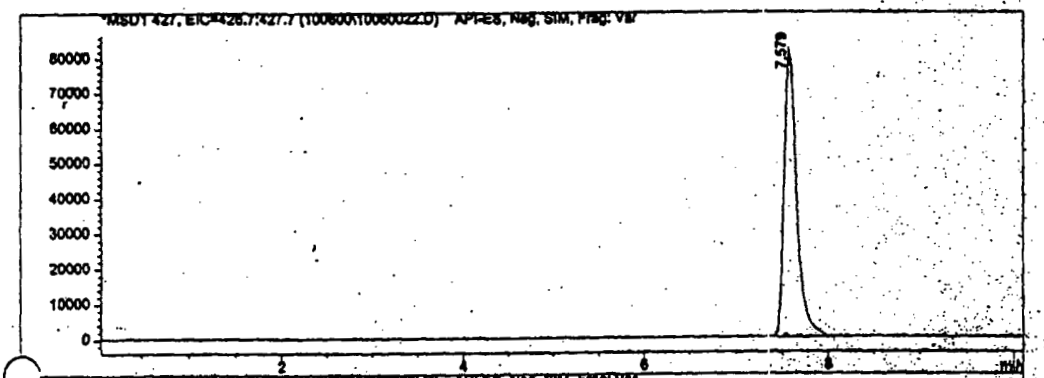
Sample Name:C100500-4
Sample Info: 1

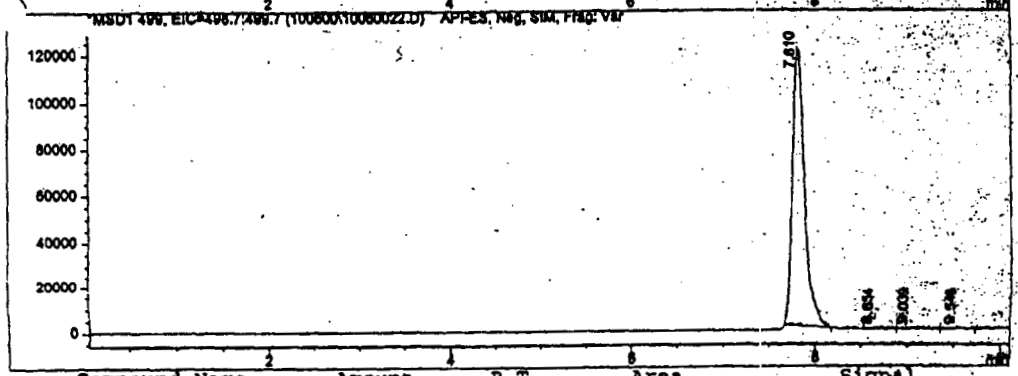
Data file :C:\HPCHEM\1\DATA\100600\10060022.D

Injection Date	: 10/6/2000 3:11:22 PM	Seq Line :	20
Acq Operator	: GS GS 10-23-00	Vial No. :	14
Instrument	: Instrument 1	Inj. No. :	1
Dilution	: 1	Inj. Vol. :	5 µl

Acq. Method : PFOS.M
Analysis Method : C:\HPCHEM\1\METHODS\100600.M
Last Changed : 10/23/2000 11:44:26 am

Analysis for PFOS





Compound Name	Amount	R.T.	Area	Signal
TH-PFOS	500.0	7.58	785730	MSD1 427, EIC=426.7:42
PFOS	261.5	7.81	1078963	MSD1 499, EIC=498.7:49

*** End of Report ***

Instrument 1 Mon, 23. Oct. 2000 00:12:06 pm Page 1 of 1

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Centre Study No.: 023-045

Figure 2. PFOS Calibration Standard (C100500-3) at 539µg/L

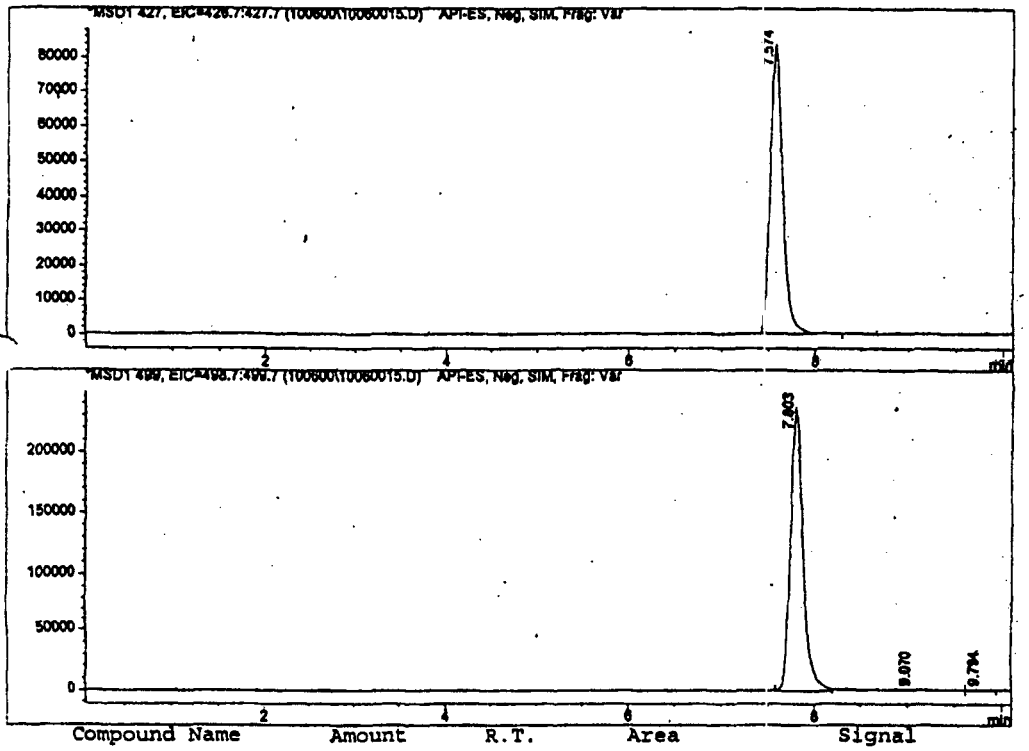
Sample Name:C100500-3
Sample Info: 1

Data file :C:\HPCHEM\1\DATA\100600\10060015.D

Injection Date	: 10/6/2000 1:13:21 PM	Seq Line	: 13
Acq Operator	: GS	Vial No.	: 15
Instrument	: Instrument 1	Inj. No.	: 1
Dilution	: 1	Inj. Vol.	: 5 µl

Acq. Method : PFOS.M
Analysis Method : C:\HPCHEM\1\METHODS\100600.M
Last Changed : 10/9/2000 01:45:43 pm
(modified after loading)

Analysis for PFOS



Compound Name	Amount	R.T.	Area	Signal
TH-PFOS	500.0	7.57	770327	MSD1 427, EIC=426.7:42
PFOS	499.8	7.80	2238071	MSD1 499, EIC=498.7:49

*** End of Report ***

Instrument 1 Mon, 9. Oct. 2000 01:46:00 pm Page 1 of 1

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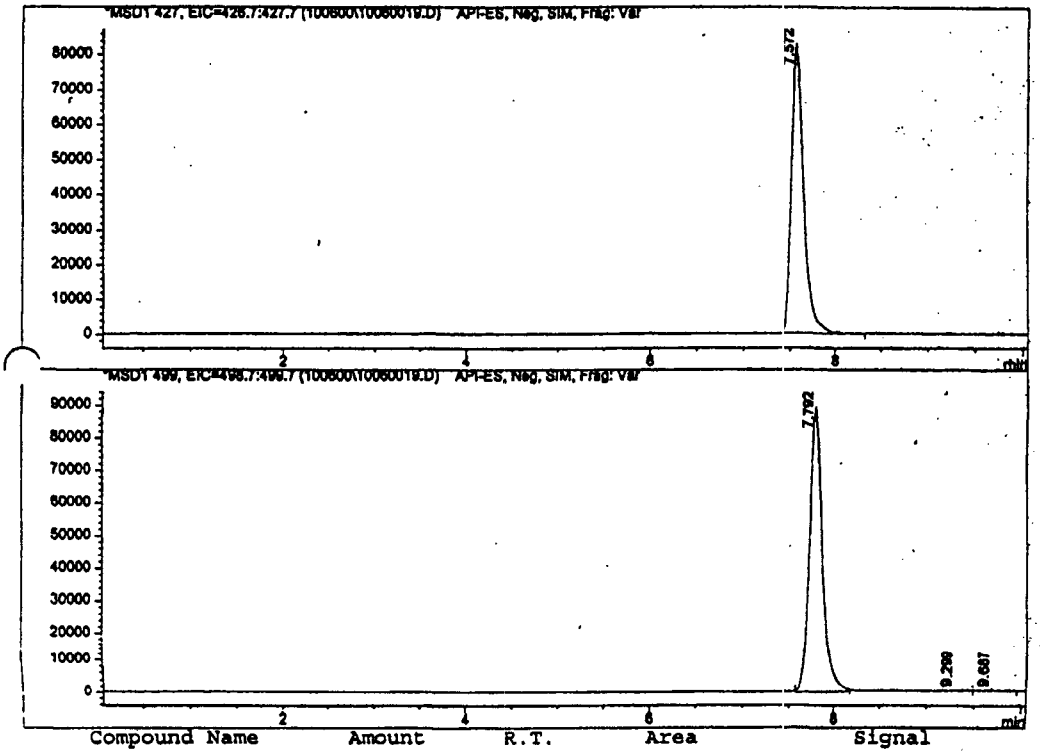
Figure 3. PFOS (TCR-00065-022) at 250µg/L, ESI Negative Ion Mode

```
Sample Name:L29092-1
Sample Info:C100500-8                                                    -> 1

Data file  :C:\HPCHEM\1\DATA\100600\10060019.D
=================================================================================
Injection Date  : 10/6/2000      2:20:49 PM      Seq Line    :          17
Acq Operator    : GS   GS 10-23-00               Vial No.    :          21
Instrument      : Instrument 1                   Inj. No.    :           1
Dilution        : 1                              Inj. Vol.   :        5 µl

Acq. Method     : PFOS.M
Analysis Method : C:\HPCHEM\1\METHODS\100600.M
Last Changed    : 10/23/2000          11:44:26 am

Analysis for PFOS
```



Compound Name	Amount	R.T.	Area	Signal
TH-PFOS	500.0	7.57	776592	MSD1 427, EIC=426.7:42
PFOS	218.7	7.79	894026	MSD1 499, EIC=498.7:49

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

Instrument 1     Mon, 23. Oct. 2000     00:10:19 pm          Page 1 of 1
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APPENDIX A

Study Protocol

Purity Determination of Sample Lots of PFOS

Including Amendment 1

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3M Medical Department Study: T-6316.1 Analytical Report: FACT TOX-003
LRN-U2104

Appendix H: Report Signature Page

John L. Butenhoff June 6, 2001

John L. Butenhoff, Ph.D., Ph.D., *Study Director* Date

Marvin T Case 6 June 2001

Marvin T. Case, D.V.M., Ph.D., *Sponsor Representative* Date

05/31/01

Kristen J. Hansen, Ph.D., *Principal Analytical Investigator* Date

05/31/01

William K. Reagen, Ph.D., *Laboratory Manager* Date