COMPOSITE ANALYTICAL LABORATORY REPORT

ON THE

Quantitative Analysis of Fluorochemicals in Environmental Samples

REPORT NO. FACT GEN-021, GEN-024, GEN-030, GEN-033
LRN—W2491, W2845, W3197, E00-1386

ANALYTICAL STUDY INITIATION

GEN021: 08/25/99
GEN024: 10/12/99
GEN030: 12/13/99
GEN033: 03/14/00

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ANALYTICAL STUDY PERSONNEL AND CONTRIBUTORS

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INTRODUCTION

Purpose

The purpose of this composite report is to provide a summary of the analytical data collected for 3M studies Gen-021, Gen-024, Gen-030, and Gen-033. All of the samples included in these studies are tissue samples collected from fish, birds, mammals, and amphibians; Dr. John Geisy of Michigan State University has supplied all samples to 3M. These analyses have been conducted to support studies designed by Dr. Geisy.

The target analytes for these four studies were perfluorooctane sulfonate (PFOS; CAS# 2795-39-3), perfluorooctanesulfonylamide (PFOSA; CAS# 754-91-6), perfluorooctanoate (PFOA or POAA; CAS# 3825-26-1), and perfluorohexane sulfonate (PFHS; no CAS# available).

Due to the variety of matrices analyzed (with respect to both species and tissues), and due to evolving analytical methods, some analytical data quality objectives, such as the limit of quantitation (LOQ) were quite variable. A summary of the achieved LOQ (by specie, tissue and study number) is presented in Table 2 of this report. The stated data quality is based on results of data collection quality controls, sample prep quality controls, and recovery of target analytes from prepared matrix spike samples. More specific data quality objectives and parameters for these analytical studies are outlined later in this report.

Test and Control Article

The test articles for each study consisted of various tissues from various species and are listed below, in Table 1. For all studies, the control article consisted of rabbit sera and rabbit liver, as appropriate. Rabbit tissues were chosen as the control articles because previous studies have indicated very low levels of endogenous fluorochemicals in these matrices. Samples of the control articles were provided by the 3M Environmental Laboratory.

This report does not include details for the collection of the test articles; these details should be obtained from Dr. Geisy.

Table 1. Description of Samples, by Study

STUDY NUMBER	SERA/PLASMA/BLOOD	LIVER	OTHER
Gen-021	Cormorant Blood, Caspian Seal Blood, Sea Otter Blood	California Sea Lion, Elephant Seal, Harbor Seal, Gozzi, Mink, River Otter, Sea Otter, Turtle	Sea Otter Brain, Sea Otter Kidney
Gen-024	Albatross sera, Albatross plasma, Cormorant plasma, Herring Gull Plasma, Bald Eagle plasma, Cormorant blood, Herring Gull blood	Loon, Brown Pelican, Albatross	Albatross kidney, Cormorant yolk, Gull yolk
Gen-030	Northern Fur Seal blood (juvenile, sub-adult, adult), Polar Bear blood, Stellar Sea Lion blood	Northern Fur Seal, Polar Bear, Mink, Map Turtle, Terrapin, Tuna, Green Frog, Chinook Salmon, Lake Whitefish, Brown Trout	Carp body, Frog muscle, Frog body, Green Frog eggs, Lake Whitefish eggs, Brown Trout eggs, Carp muscle, Chinook Salmon muscle, Lake Whitefish muscle, Brown Trout muscle
Gen-033	None Submitted	Mink, Baikal Seal, Ganges Dolphin, Cormorant (adult and juvenile), Bottlenose Dolphin, Striped Dolphin, Weddell Seal, Swordfish, Tuna, Blacktailed Gull	None Submitted

Following analysis, extracts generated from these samples have been retained in cold storage.

Sample Collection and Analysis

Tissue samples were submitted to the Environmental Laboratory- Fluorine Analytical Chemistry Team by Kurunthachalan Kannan of Michigan State University. Details of the sample receipt are documented on the chain of custody forms located in appendices of this report.

SAMPLE RECEIPT AND MAINTENANCE

Samples were received in the Environmental Lab cold or frozen on the following dates: Gen-021 (8/24/99), Gen-024 (10/11/99), Gen-030 (12/13/99), and Gen-033 (3/13/00). Sample receipt, identification, and chain of custody information are located in the study folder for each report; the folders are located in the 3M archives.

The sample extracts will be maintained in cold storage at the 3M Environmental Laboratory until the quality of preparation no longer affords preservation.

CHEMICAL CHARACTERIZATION

The target analytes characterized in the samples include PFOS, PFOSA, PFOA, and PFHS. Procurement details of the reference standards used for analysis are summarized below.

Procurement

Table 2. Procurement Information for Reference Materials in the Analysis of Environmental Samples

REFERENCE MATERIAL	LOT NUMBER	SOURCE
PFOS (potassium salt)	171	3M ICP/PCP Division
PFOSA	Gen-021: L-2353; all others: L-15709	3M Specialty Chemicals (R. Buckanin)
PFHS (potassium salt)	NB116638-16	3M Specialty Chemicals (G. Moore)
PFOA (ammonium salt)	Gen-024: 245; all others: commercial	Gen-024: 3M Specialty Chemicals; all others: Aldrich

Full chemical characterization studies, including purity and stability determination, have not been completed at this time. Upon completion of these studies, a report will be archived in the 3M Environmental Lab.

METHOD SUMMARIES

Following is a brief description of the methods used during this analytical study by the 3M Environmental Laboratory. Copies of the actual methods used for these studies are located in attachment H.

PREPARATORY AND ANALYTICAL METHODS

- ETS-8-004.1, "Extraction of PFOS or Other Fluorochemical Compounds from Serum for Analysis using HPLC-Electrospray Mass Spectrometry" with some modifications, described below.

  Because the matrices were so variable and sample size extremely limited, it was not possible to prepare extracted standard curves. All extracts were evaluated versus unextracted standard curves. When sample size permitted, two matrix spikes were prepared in each tissue sample from each specie tested to provide some level of extraction efficiency determination.

  For some samples, less than 1 mL of sample was available. For these samples, the available volume was extracted according to the method with the exception that the final volume of extraction solvent was adjusted to match the volume of the initial sample.

  This method was used for the extraction of sera, plasma, and whole blood samples.

- ETS-8-005.1, "Analysis of PFOS or Other Fluorochemical Compounds in Serum Extracts Using HPLC-Electrospray Mass Spectrometry" with some modifications, described below.

  Because the matrices were so variable and sample size extremely limited, it was not possible to prepare extracted standard curves. All extracts were evaluated versus unextracted standard curves; as a result, all sample concentrations were adjusted by a factor of 1.25 to adjust for the removal of 4/5 of the MTBE from the extract. The factor is unnecessary when an extracted curve is used for evaluation.

- ETS-8-006, "Analysis of PFOS or Other Fluorochemical Compounds in Liver Extracts using HPLC-Electrospray Mass Spectrometry" with some modifications, described below.

  Because the matrices were so variable and sample size extremely limited, it was not possible to prepare extracted standard curves. All extracts were evaluated versus unextracted standard curves. When sample size permitted, two matrix spikes were prepared in each tissue sample from each specie tested to provide some level of extraction efficiency determination.

  For some samples, less than 1 g of sample (as called for in the method) was available. For these samples, the available mass of tissue was extracted according to the method.

  Samples of kidney, brain, egg, and muscle were extracted by this method.

- ETS-8-007, "Extraction of PFOS or Other Fluorochemical Compounds from Liver for Analysis using HPLC-Electrospray Mass Spectrometry" with some modifications, described below.

  Because the matrices were so variable and sample size extremely limited, it was not possible to prepare extracted standard curves. All extracts were evaluated versus unextracted standard curves; as a result, all sample concentrations were adjusted by a factor of 1.25 to adjust for the removal of 4/5 of the MTBE from the extract. The factor is unnecessary when an extracted curve is used for evaluation.

  For Gen-030 and Gen-033 only: Due to the lack of excess test material for method development, all samples determined to contain greater than 0.015 µg/g of PFOS were subject to an additional PFOS verification process. Each sample was analyzed separately with respect to the 499 → 99 transition and the 499 → 80 transitions. The quantitative results

obtained from each transition analysis were compared. When these results agreed to with 30%, the identity of PFOS was confirmed (see Reference 1). Those samples where the identity of PFOS could not be confirmed are noted in the data table.

In Gen-021, no PFHS standard was available. In these samples, qualitative determination of PFHS was conducted based on reasonable retention time and a known PFHS transition (399 → 99).

Specific instrumental parameters are available in appendix I-L of this report, stored in the 3M Environmental Lab archives.

ANALYTICAL EQUIPMENT

For HPLC-Electrospray Tandem Mass Spectrometry:
Liquid Chromatograph: Hewlett-Packard® Series 1100 Liquid Chromatograph system
Analytical column:
1×30 mm C18 Betasil™
Column temperature: 30 degrees C
Cycle Time: 10 minutes

Mobile phase components:
Component A: 2mM ammonium acetate
Component B: Methyl alcohol
Flow rate: 300 µL/min

Injection volume: 10 µL
Solvent Gradient:

Time (min)	%B
0	10
1	10
5.5	95
7.5	95
8	10
10	10

For Detection:
Mass Spectrometer: Micromass® API/Mass Spectrometer Quattro Ultima Triple Quadrapole system or Micromass® API/Mass Spectrometer Quattro II Triple Quadrapole system
Acquisition Mode: MRM (refer to Table 3)
Software: Mass Lynx™ 3.3
Mode: Electrospray Negative
Source Block Temperature: 125-150°C
Source: Z-spray

Table 3. Ions Monitored in the Analyses of Extracts of Groundwater

TARGET ANALYTE	PRIMARY ION (amu)	PRODUCT ION (amu)
PFOS	499.0	80, 99*
PFOSA	498	78
PFOA	413	169
PFHS	399	99

* Indicates the ion used for quantitation

Refer to the analytical methods and equipment logs found in the raw data for details on the actual analytical equipment settings used in the present study. These settings may have varied somewhat during actual data collection. However, slight variations in the instrument settings will not adversely affect the quality of the data. Exact settings during all phases of data collection are recorded and presented in the appendix of this report.

DATA SUMMARY, ANALYSES, AND RESULTS

Summary of Quality Control Analyses Results

- **Standard Curves:** The coefficient of determination (r^2) for all 1/X weighted curves bracketing useable data was $\geq$ 0.982. High or low curve points may have been excluded to provide a better fit over the linear range appropriate to the data. High or low curve points were deactivated if the calculated concentration varied from the theoretical concentration by more than 30%. Acceptable data was evaluated versus a standard curve containing at least 5 points. All actions are acceptable and are documented in specific data sets. All standard curves used to evaluate quantitative data were acceptable.
- **Continuing Calibration Verifications:** On average, one calibration check is analyzed for every five samples. Acceptable data is bracketed by calibration checks quantitated to be within 30% of the theoretical value, evaluated at least every ten samples. All quantitative data is bracketed by acceptable calibration checks, as required.
- **Blanks:** Extraction blanks were compliant if no target analyte was detected above the limit of quantitation (LOQ) for a specific analyte. In this study, extraction blanks were often higher than low curve points. Because analyte levels in the blank are used to determine the LOQ, by default, all blanks were determined to be below the limit of quantitation for the compounds of interest.
- **Internal Standards:** Internal standard response was monitored in Gen-030 and Gen-033 only. Internal standard response was required to be within ±50% of the theoretical value. If samples showed an internal standard response that deviated more than ±50%, the samples were reanalyzed. If the deviant IS response was confirmed, the analyte data was reported, but noted in the data table.

Summary of Sample Results

- **GEN-021:**
  - PFOS was detected in at least one sample from the following matrices: California Sea Lion liver, Harbor Seal liver, Gozzi liver, Mink liver, River Otter liver, Turtle liver, Cormorant blood, Otter blood, and Caspian Seal blood.
  - PFOSA was tentatively identified in at least one sample from the following matrices: California Sea Lion liver, River Otter liver, Sea Otter liver, Sea Otter brain, and Otter blood.
  - PFOA was tentatively identified in at least one sample from the following matrices: California Sea Lion liver and Caspian Seal blood.
  - PFHS was tentatively identified in at least one sample from the following matrices: California Sea Lion liver, Gozzi liver, Mink liver, River Otter liver, Sea Otter liver, Turtle liver, Cormorant blood, Caspian Seal blood, and Otter blood.
- **GEN-024:**
  - PFOS was detected identified in at least one sample from the following matrices: Albatross plasma, Albatross sera, Cormorant plasma, Cormorant blood, Herring Gull plasma, Herring Gull blood, Bald Eagle plasma, Loon liver, Albatross liver, Brown Pelican liver, Albatross kidney, Cormorant yolk, and Gull yolk.
  - PFOSA was tentatively identified in at least one sample from the following matrices: Cormorant blood, Bald Eagle plasma, Loon liver, Brown Pelican liver, and Albatross liver.
  - PFOA was tentatively identified in at least one sample from the following matrices: Cormorant blood, Albatross liver, Cormorant yolk, and Gull yolk.
  - PFHS was tentatively identified in at least one sample from the following matrices: Herring Gull plasma and Bald Eagle plasma, Loon liver, Albatross liver, Brown Pelican liver, Albatross kidney, Cormorant yolk, and Gull yolk.
- **GEN-030:**
  - PFOS was detected in at least one sample from the following matrices: Polar Bear blood, Polar Bear liver, Mink liver, Northern Fur Seal liver, Map Turtle liver, Tuna liver, Green Frog liver, Chinook Salmon liver, Lake Whitefish liver, Brown Trout liver, Whole Carp, Frog muscle, Lake Whitefish eggs, Brown Trout eggs, Carp muscle, Chinook Salmon muscle, Lake Whitefish muscle, and Brown Trout muscle.
  - PFOSA was tentatively identified in at least one sample from the following matrices: Mink liver.
  - PFOA was not tentatively identified in any sample analyzed.
  - PFHS was not tentatively identified in any sample analyzed.
- **GEN-033:**
  - PFOS was detected in at least one sample from the following matrices: Mink liver, Baikal Seal liver, Cormorant liver, Bottle Nosed Dolphin liver, Ganges Dolphin liver, Striped Dolphin liver, Swordfish Liver, Tuna liver, and Black Tailed Gull liver.
  - PFOSA was tentatively identified in at least one sample from the following matrices: Mink liver, Cormorant liver, and Bottle Nosed Dolphin liver.

- PFOA was tentatively identified in at least one sample from the following matrices: Cormorant liver.
- PFHS was tentatively identified in at least one sample from the following matrices: Mink liver, Striped Dolphin liver, and Swordfish Liver.

Appendices contain data summary tables.

DATA QUALITY OBJECTIVES

No circumstances existed during the present study that would have affected the quality or integrity of the data. The data quality objectives (DQOs) followed during the present are indicated below.

- **Linearity:** The coefficient of determination (r^2) of the standard curve was equal to or greater than 0.985 with at least 5 active points using a linear regression curve with 1/x weighting.
- **Instrument Quantitation Limit (IQL):** The IQL is equal to the lowest acceptable standard in the calibration curve (acceptable standard is defined as a standard within 30% of the theoretical value). As this value is not useful in consideration of the sample data, the IQL was not specifically determined or stated for every study.
- **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 extraction blanks. The LOQ may vary due to the amount of sample available for analysis (particularly for samples extracted according to ETS-8-006) or to day-to-day variations in the analytical system. The ranges of LOQs for various tissues are listed in Table 4 (sera, plasma, and blood) and Table 5 (liver, kidney, muscle, egg, and brain).

Table 4. Range of LOQs for Sera, by Study

ANALYTE	GEN-021	GEN024	GEN-030	GEN-033
PFOS	0.0116 µg/mL	0.00116 µg/mL	0.0029-0.0579 µg/mL	NA
PFOSA	0.00625µg/mL	0.00626 µg/mL	0.000625 µg/mL	NA
PFHS	NA	0.00114 µg/mL	0.00114 µg/mL	NA
PFOA	0.00599 µg/mL	0.0299 µg/mL	0.00240-0.00958 µg/mL	NA

Table 5. Range of LOQs for Liver and Other Tissues, by Study

ANALYTE	GEN-021	GEN024	GEN-030	GEN-033
PFOS	0.0348 µg/g	0.0348 µg/g	0.00696-0.0696 µg/g	0.00696- 0.0694µg/g
PFOSA	0.0375 µg/g	0.00750 µg/g	0.0188 µg/g	0.0376 µg/g
PFHS	NA	0.00683 µg/g	0.00683-0.0342 µg/g	0.00683 µg/g
PFOA	0.0359 µg/g	0.180 µg/g	0.0180-0.0719 µg/g	0.00719-0.0718 µg/g

NA = not applicable

- **Duplicate/acceptable precision (extraction):** Spikes conducted on samples of control tissues were reproducible to within 15%
- **Quality Control Response:** A continuing calibration verification (CCV) was analyzed every 5–10 samples. Acceptable CCV response was within ±30% of the theoretical value. No more than 10 samples were analyzed between acceptable CCVs.
- **Spike/acceptable recoveries:** Due to the number of different matrices analyzed, there was great variability in spike recoveries. For any given matrix (specie and tissue), spike recoveries within 70–130% of the expected concentration indicate quantitative data (good to ±30%); spike recoveries between 50–150% indicate semi-quantitative data for that matrix (good to ±50%). Spike recoveries outside of this range indicate that sample data should be used for qualitative purposes only. Due to sample limitations, matrix spike studies were not conducted for all matrices. For PFOS analyses, sample data that is not supported by matrix spike studies should be considered for qualitative purposes only. Since no identity verification experiments were performed for PFOA, PFHS, and PFOSA, for these analytes, all analyses that are not supported by matrix spike studies should be considered to provide unconfirmed qualitative data only.
- **Use of Internal Standards:** Tetrahydro-perfluorooctane sulfonate (THPFOS) was spiked into the extracts post-extraction and used as an internal standard for samples in Gen-030 and Gen-033. For all samples in these studies, THPFOS levels were monitored to verify the analytical soundness of the data. THPFOS levels that were determined to be deviant from expected values by more than ±50% were reanalyzed. If the deviant THPFOS levels were confirmed, analyte levels were reported but are noted in the results table.
- **Use of confirmatory methods:** Given the selectivity of the analytical tool used (HPLC-ESMSMS) and lack of a viable alternative for analysis, no confirmatory methods were used.
- **Demonstration of specificity:** Specificity was demonstrated by chromatographic retention time (matched to standards to within 3%) and the response of at least one characteristic product ion arising from collisions of an analyte-specific parent ion.

Assuming spike recovery studies form a suitable indication of endogenous analyte recovery, matrix spike studies have been used as an indicator of data quality (see above). The validity of this assumption has not been verified by other techniques.

STATEMENT OF CONCLUSION

Under the conditions of the present studies, the presence of fluorochemicals was observed in the quantitative analysis of a selection of environmental matrices.

REFERENCES

1) "Acceptance Criteria for Ultratrace HPLC-Tandem Mass Spectrometry: Quantitative and Qualitative Determination of Sulfonylurea Herbicides in Soil"; Li, L.Y.; Campbell, D.A.; Bennet, P.K.; Henion, J.; *Anal. Chem.*, **68** (19), 3397-3404, 1996

ATTACHMENTS

- Attachment A: Gen-021 Sera/Plasma/Blood Results
- Attachment B: Gen-021 Liver/Miscellaneous Results
- Attachment C: Gen-024 Sera/Plasma/Blood Results
- Attachment D: Gen-024 Liver/Miscellaneous Results
- Attachment E: Gen-030 Sera/Plasma/Blood Results
- Attachment F: Gen-030 Liver/Miscellaneous Results
- Attachment G: Gen-033 Liver Results
- Attachment H: Analytical Methods
- Attachment I – L (additional bound document available in the 3M Environmental Lab archives): Analytical Details for Gen-021, Gen-024, Gen-030, and Gen-033

REPORT SIGNATURE

Kristen Hansen 5/10/00

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

Dale L. Bacon 5/10/2000

Dale L. Bacon, Sponsor Representative — *Date*

Study:	GEN021 Various Matrices from MSU
Product Number(Test Substance):	None
Matrix:	Blood
Method/Revision:	ETS-8-4.1 & ETS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.2
Filename:	See list to right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	08/25/99 MCH/KK/SAL/SEE
Dates of Analysis/Analyst:	08/26/99, 08/27/99, 08/28/99, 12/09/99 MEE/IAS
Date of Data Reduction/Analyst:	08/27/99, 08/30/99, 08/31/99, 12/10/99, 01/20/00 MEE/IAS

Sample Data

BLOOD

Group Dose	Sample #	Concentration of PFOS ug/mL	Mean PFOS ug/mL	RSD Std. Dev.	Concentration of PFOSA ug/mL	Mean PFOSA ug/mL	RSD Std. Dev.
Method Blk	H2O Blk-1 8/25/99	<LOQ (0.0116)			<LOQ (0.00625)		
	H2O Blk-2 8/25/99	<LOQ (0.0116)			<LOQ (0.00625)		
	H2O Blk-3 8/25/99	<LOQ (0.0116)		NA	<LOQ (0.00625)		NA
	H2O Blk-4 8/25/99	<LOQ (0.0116)	<LOQ	NA	<LOQ (0.00625)	<LOQ	NA
Caspian Seal Blood	W2491-40,J 53	0.0180			<LOQ (0.00625)		
	W2491-41,J 11	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-42,J 46	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-43,J 17	0.0166			<LOQ (0.00625)		
	W2491-44,J 13	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-45,J 8	0.0131			<LOQ (0.00625)		
	W2491-46,J 12	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-47,J 9	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-48,J 14	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-49,J 18	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-50,J 52	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-51,J 55	<LOQ (0.0116)			<LOQ (0.00625)		
	W2491-52,J 10	<LOQ (0.0116)		NA	<LOQ (0.00625)		NA
	W2491-53,J 15	<LOQ (0.0116)	<LOQ - 3 Outliers	NA	<LOQ (0.00625)	<LOQ	NA

No curve analyzed for PFHS, PFDS. PFHS based on PFOS response.
No PFOS qualitative confirmation performed. Identifications are preliminary.
Deviant Surrogate levels are not noted and were not confirmed.
Date Entered/By: 08/27/99, 09/01/99, 12/30/99, 01/20/00, 02/14/00 LAC
Date Verified/ By: 02/22/00 MEE

LOQ = Limit of Quantitation
NA = Not Applicable
RSD = Relative Standard Deviation
ND = Not Detected
D = Detected

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
POAA = Perfluorooctanoate
PFHS = Perfluorohexanesulfonate

Study:	GEN021 Various Matrices from MSU
Product Number(Test Substance):	None
Matrix:	Blood
Method/Revision:	ETS-8-4.1 & ETS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.2
Filename:	See list to right
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	08/25/99 MCH/KK/SAL/SEE
Dates of Analysis/Analyst:	08/26/99, 08/27/99, 08/28/99, 12/09/99 MEE/IAS
Date of Data Reduction/Analyst:	08/27/99, 08/30/99, 08/31/99, 12/10/99, 01/20/00 MEE/IAS

Sample Data

BLOOD

Group Dose	Sample #	Concentration of POAA ug/mL	Mean POAA ug/mL	RSD Std. Dev.	Concentration of PFHS ug/mL	Mean PFHS ug/mL	RSD Std. Dev.
Method Blk	H2O Blk-1 8/25/99	0.00629			ND		
	H2O Blk-2 8/25/99	<LOQ (0.00599)			ND		
	H2O Blk-3 8/25/99	<LOQ (0.00599)		NA	ND		NA
	H2O Blk-4 8/25/99	<LOQ (0.00599)	<LOQ - 1 Outlier	NA	ND	ND	NA
Caspian Seal Blood	W2491-40,J 53	<LOQ (0.00599)			D		
	W2491-41,J 11	0.00759			D		
	W2491-42,J 46	<LOQ (0.00599)			ND		
	W2491-43,J 17	<LOQ (0.00599)			D		
	W2491-44,J 13	<LOQ (0.00599)			ND		
	W2491-45,J 8	<LOQ (0.00599)			ND		
	W2491-46,J 12	<LOQ (0.00599)			ND		
	W2491-47,J 9	<LOQ (0.00599)			ND		
	W2491-48,J 14	0.00728			ND		
	W2491-49,J 18	<LOQ (0.00599)			ND		
	W2491-50,J 52	0.0108			ND		
	W2491-51,J 55	0.0234			ND		
	W2491-52,J 10	<LOQ (0.00599)		NA	ND		NA
	W2491-53,J 15	<LOQ (0.00599)	<LOQ - 4 Outliers	NA	ND	ND - 3 Outliers	NA

No curve analyzed for PFHS, PFDS. PFHS based on PFOS response.
No PFOS qualitative confirmation performed. Identifications are preliminary.
Deviant Surrogate levels are not noted and were not confirmed.
Date Entered/By: 08/27/99, 09/01/99, 12/30/99, 01/20/00, 02/14/00 LAC
Date Verified/ By: 02/22/00 MEE

LOQ = Limit of Quantitation
NA = Not Applicable
RSD = Relative Standard Deviation
ND = Not Detected
D = Detected

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
POAA = Perfluorooctanoate
PFHS = Perfluorohexanesulfonate

Study:	GEN021 Various Matrices from MSU
Product Number(Test Substance):	None
Matrix:	Blood
Method/Revision:	BTS-8-4.1 & BTS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.2, 3.3
Filename:	08/28/99, 12/09/99 IAS
R-Squared Value:	08/28/99, 12/09/99 IAS
Slope:	08/30/99, 12/10/99 IAS
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	08/25/99 MCH/KK/SAL/SEE
Dates of Analysis/Analyst:	08/28/99, 12/09/99 IAS
Date of Data Reduction/Analyst:	08/30/99, 12/10/99 IAS

Sample Data

BLOOD **QC**

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL or % Rec	RSD Std. Dev.	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL or % Rec	RSD Std. Dev.
Method Blk	H2O Blk-1 8/25/99	NA			NA		
	H2O Blk-2 8/25/99	NA			NA		
	H2O Blk-3 8/25/99	NA		NA	NA		NA
	H2O Blk-4 8/25/99	NA	NA	NA	NA	NA	NA
Caspian Seal Blood	W2491-40,J 53-MS	164%			106%		
	W2491-41,J 11-MS	115%			98%		
	W2491-42,J 46-MS	101%			73%		
	W2491-43,J 17-MS	2431%	*		1991%	*	
	W2491-44,J 13-MS	67%			50%		
	W2491-45,J-8-MS	103%			79%		
	W2491-46,J 12-MS	150%			116%		
	W2491-47,J 9-MS	103%			85%		
	W2491-48,J 14-MS	103%			90%		
	W2491-49,J 18-MS	73%			47%		
	W2491-50,J 52-MS	65%			49%		
	W2491-51,J 55-MS	17%			10%		
	W2491-52,J 10-MS	12%	* outlier excluded	58%	10%	* outlier excluded	58%
	W2491-53,J 15-MS	17%	84%	48%	12%	63%	37%

No curve analyzed for PFHS, PFDS. PFHS based on PFOS response.
No PFOS qualitative confirmation performed. Identifications are preliminary.
Deviant Surrogate levels are not noted and were not confirmed.
Date Entered/By: 02/16/00, 02/17/00 LAC
Date Verified/ By: 02/22/00 MEE

LOQ = Limit of Quantitation
NA = Not Applicable
RSD = Relative Standard Deviation
ND = Not Detected
D = Detected
NS = Not Spiked

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
POAA = Perfluorooctanoate
PFHS = Perfluorohexanesulfonate

Study:	GEN021 Various Matrices from MSU
Product Number(Test Substance):	None
Matrix:	Blood
Method/Revision:	ETS-8-4.1 & ETS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.2, 3.3
Filename:	08/28/99, 12/09/99 IAS
R-Squared Value:	08/28/99, 12/09/99 IAS
Slope:	08/30/99, 12/10/99 IAS
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	08/25/99 MCH/KK/SAL/SEE
Dates of Analysis/Analyst:	08/28/99, 12/09/99 IAS
Date of Data Reduction/Analyst:	08/30/99, 12/10/99 IAS

Sample Data

BLOOD **QC**

Group Dose	Sample #	Concentration of POAA ug/mL or % Rec	Mean POAA ug/mL or % Rec	RSD Std. Dev.	Concentration of PFHS ug/mL or % Rec	Mean PFHS ug/mL or % Rec	RSD Std. Dev.
Method Blk	H2O Blk-1 8/25/99	NA			NS		
	H2O Blk-2 8/25/99	NA			NS		
	H2O Blk-3 8/25/99	NA		NA	NS		NS
	H2O Blk-4 8/25/99	NA	NA	NA	NS	NS	NS
Caspian Seal Blood	W2491-40,J 53-MS	157%			NS		
	W2491-41,J 11-MS	115%			NS		
	W2491-42,J 46-MS	100%			NS		
	W2491-43,J 17-MS	2056%	*		NS		
	W2491-44,J 13-MS	89%			NS		
	W2491-45,J 8-MS	108%			NS		
	W2491-46,J 12-MS	174%			NS		
	W2491-47,J 9-MS	111%			NS		
	W2491-48,J 14-MS	112%			NS		
	W2491-49,J 18-MS	91%			NS		
	W2491-50,J 52-MS	78%			NS		
	W2491-51,J 55-MS	14%			NS		
	W2491-52,J 10-MS	19%	* outlier excluded	54%	NS		NS
	W2491-53,J 15-MS	19%	91%	50%	NS	NS	NS

No curve analyzed for PFHS, PFDS. PFHS based on PFOS response.
No PFOS qualitative confirmation performed. Identifications are preliminary.
Devient Surrogate levels are not noted and were not confirmed.
Date Entered/By: 02/16/00, 02/17/00 LAC
Date Verified/ By: 02/22/00 MEE

LOQ = Limit of Quantitation
NA = Not Applicable
RSD = Relative Standard Deviation
ND = Not Detected
D = Detected
NS = Not Spiked

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
POAA = Perfluorooctanoate
PFHS = Perfluorohexanesulfonate

Study:	GEN021 Various Matrices from MSU
Product Number(Test Substance):	None
Matrix:	Various Matrices
Method/Revision:	ETS-8-6.0 & ETS-8-7.0 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.2
Date of Extraction/Analyst:	08/25/99 MCH/EKE/SAL/SEE
Date of Analysis/Analyst:	08/26/99, 08/27/99, 08/28/99, 12/09/99 MEE/IAS
Date of Data Reduction/Analyst:	08/27/99, 08/30/99, 08/31/99, 12/10/99, 01/20/00 MEE/IAS

Sample Data

LIVER/WHOLE BLOOD

Group Dose	Sample #	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev.	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std. Dev.
Method Blk	H2O Blk-1 8/25/99	<LOQ (0.0348)			<LOQ (0.0375)		
	H2O Blk-2 8/25/99	<LOQ (0.0348)			<LOQ (0.0375)		
	H2O Blk-3 8/25/99	<LOQ (0.0348)		<LOQ	<LOQ (0.0375)		NA
	H2O Blk-4 8/25/99	<LOQ (0.0348)	<LOQ	<LOQ	<LOQ (0.0375)	<LOQ	NA
California Sea Lion Liver	W2491-3,CSL 3448	0.0384			0.0443		
	W2491-4,CSL 3395	0.0494			<LOQ (0.0375)		
	W2491-6,CSL 3020	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-7,CSL 2169	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-9,CSL 2839	<LOQ (0.0348)		NA	<LOQ (0.0375)		NA
	W2491-10,CSL 2367	<LOQ (0.0348)	<LOQ - 2 Outliers	0.00773	<LOQ (0.0375)	<LOQ - 1 Outlier	NA
Elephant Seal Liver	W2491-2,ES 1500	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-5,ES 1552	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-11,ES 808	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-12,ES 772	<LOQ (0.0348)		NA	<LOQ (0.0375)		NA
	W2491-14,ES 782	<LOQ (0.0348)	<LOQ	NA	<LOQ (0.0375)	<LOQ	NA
Harbor Seal Liver	W2491-4,HS	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-13,HS 1191	<LOQ (0.0348)		NA	<LOQ (0.0375)		NA
	W2491-15,HS 1199	0.0571	<LOQ - 1 Outlier	NA	<LOQ (0.0375)	<LOQ	NA
Canal Liver	W2491-1,NFS 100	0.133	0.133	NA	<LOQ (0.0375)	<LOQ	NA
Mink Liver	W2491-34,D1030 USFWS	4.85			<LOQ (0.0375)		
	W2491-35,D1146 USFWS	2.41		81.7	<LOQ (0.0375)		NA
	W2491-36,D1158 USFWS	0.587	2.62	2.14	<LOQ (0.0375)	<LOQ	NA
River Otter Liver	W2491-29,RAG 066	0.279			0.0371		
	W2491-30,RAG 028	0.994			0.0448		
	W2491-31,RAG 148	0.189			0.0716		
	W2491-32,RAG 230	0.0336		116	<LOQ (0.0375)		33.1
	W2491-33,RAG 237	0.151	0.329	0.382	0.0393	0.0482 - 1 Outlier	0.0160
Sea Otter Liver	W2491-16,SO 12593-001	<LOQ (0.0348)			0.0806		
	W2491-19,SO 11494-001	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-22,SO 11940-001	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-24,SO 11309-001	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-25,SO 12797-001	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-26,SO 13110-001	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-27,SO 12679-001	<LOQ (0.0348)		NA	<LOQ (0.0375)		NA
	W2491-28,SO 12707-001	<LOQ (0.0348)	<LOQ	NA	<LOQ (0.0375)	<LOQ - 1 Outlier	NA
Turtle Liver	W2491-37,Male Turtle (-2,8),Turtle Liver	0.239			<LOQ (0.0375)		
	W2491-38,Male Turtle (2,12),Turtle Liver	0.358		56.0	<LOQ (0.0375)		NA
	W2491-39,Female Turtle (-3,9),Turtle Liver	0.099	0.232	0.130	<LOQ (0.0375)	<LOQ	NA
Sea Otter Brain	W2491-18,SO 12593-001,Sea Otter Brain	<LOQ (0.0348)		NA	0.0664		NA
	W2491-21,SO 11494-001,Sea Otter Brain	<LOQ (0.0348)	<LOQ	NA	<LOQ (0.0375)	NA	NA
Sea Otter Kidney	W2491-17,SO 12593-001,Sea Otter Kidney	<LOQ (0.0348)			<LOQ (0.0375)		
	W2491-20,SO 11494-001,Sea Otter Kidney	<LOQ (0.0348)		NA	<LOQ (0.0375)		NA
	W2491-23,SO 11940-001,Sea Otter Kidney	<LOQ (0.0348)	<LOQ	NA	<LOQ (0.0375)	<LOQ	NA
Whole Blood	W2491-54,Cormorant DCCO L Charity	0.190			<LOQ (0.0375)		
	W2491-55,Cormorant DCCO Hyrn Island, Lake Sup	0.0422		95.2	<LOQ (0.0375)		NA
	W2491-56,Otter DCCO Great Lakes	0.0592	0.0904	0.0861	0.112	<LOQ - 1 Outlier	NA

PFDS/PFHS = no curve analyzed PFHS based on PFOS response.
No PFOS qualitative confirmation performed. Identifications are preliminary.
Deviant Surrogate levels are not noted and were not confirmed.

LOQ = Limit of Quantitation
RSD = Relative Standard Deviation
NA = Not Applicable
ND = Not Detected
D = Detected

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
POAA = Perfluorooctanoate
PFHS = Perfluorohexanesulfonate

Date Entered/Analyst: 08/27/99, 09/01/99, 12/30/99, 01/20/00, 02/14/00 LAC
Date Verified/Analyst: 02/22/00 MEE

Study: GEN021 Various Matrices from MSU
Product Number(Test Substance): None
Matrix: Various Matrices
Method/Revision: ETS-8-6.0 & ETS-8-7.0 using unextracted curves
Analytical Equipment System Number: Amelia 062498
Instrument Software/Version: Masslynx 3.2
Date of Extraction/Analyst: 08/25/99 MCH/KK/SAL/SEE
Date of Analysis/Analyst: 08/26/99, 08/27/99, 08/28/99, 12/09/99 MEE/IAS
Date of Data Reduction/Analyst: 08/27/99, 08/30/99, 08/31/99, 12/10/99, 01/20/00 MEE/IAS

Sample Data

LIVER/WHOLE BLOOD

Group Dose	Sample #	Concentration of POAA ug/g or % Rec.	Mean POAA ug/g	RSD Std. Dev.	Concentration of PFHS ug/g or % Rec.	Mean PFHS ug/g	RSD Std. Dev.
Method Blk	H2O Blk-1 8/25/99	0.00602			ND		
	H2O Blk-2 8/25/99	<LOQ (0.0359)			ND		
	H2O Blk-3 8/25/99	<LOQ (0.0359)		NA	ND		NA
	H2O Blk-4 8/25/99	<LOQ (0.0359)	<LOQ - 1 Outlier	NA	ND	ND	NA
California Sea Lion Liver	W2491-3,CSL 3448	<LOQ (0.0359)			ND		
	W2491-4,CSL 3395	0.0409			ND		
	W2491-6,CSL 3020	<LOQ (0.0359)			ND		
	W2491-7,CSL 2169	<LOQ (0.0359)			ND		
	W2491-9,CSL 2839	<LOQ (0.0359)		NA	D		NA
	W2491-10,CSL 2367	<LOQ (0.0359)	<LOQ - 1 Outlier	NA	ND	ND - 1 Outlier	NA
Elephant Seal Liver	W2491-2,ES 1500	<LOQ (0.0359)			ND		
	W2491-5,ES 1552	<LOQ (0.0359)			ND		
	W2491-11,ES 806	<LOQ (0.0359)			ND		
	W2491-13,ES 772	<LOQ (0.0359)		NA	ND		NA
	W2491-14,ES 782	<LOQ (0.0359)	<LOQ	NA	ND	ND	NA
Harbor Seal Liver	W2491-8,HS	<LOQ (0.0359)			ND		
	W2491-13,HS 1191	<LOQ (0.0359)		NA	ND		NA
	W2491-15,HS 1199	<LOQ (0.0359)	<LOQ	NA	ND	ND	NA
Goat Liver	W2491-1,MFS 100	<LOQ (0.0359)	<LOQ	NA	D	D	NA
Mink Liver	W2491-34,D1080 USFWS	<LOQ (0.0359)			D		
	W2491-35,D1146 USFWS	<LOQ (0.0359)		NA	ND		NA
	W2491-36,D1158 USFWS	<LOQ (0.0359)	<LOQ	NA	ND	ND - 1 Outlier	NA
River Otter Liver	W2491-29,RAG 066	<LOQ (0.0359)			D		
	W2491-30,RAG 028	<LOQ (0.0359)			D		
	W2491-31,RAG 148	<LOQ (0.0359)			D		
	W2491-32,RAG 230	<LOQ (0.0359)		NA	ND		NA
	W2491-33,RAG 237	<LOQ (0.0359)	<LOQ	NA	D	D - 1 Outlier	NA
Sea Otter Liver	W2491-16,SO 12593-001	<LOQ (0.0359)			ND		
	W2491-19,SO 11494-001	<LOQ (0.0359)			D		
	W2491-22,SO 11940-001	<LOQ (0.0359)			ND		
	W2491-24,SO 11309-001	<LOQ (0.0359)			ND		
	W2491-25,SO 12797-001	<LOQ (0.0359)			D		
	W2491-26,SO 13110-001	<LOQ (0.0359)			ND		
	W2491-27,SO 12679-001	<LOQ (0.0359)		NA	ND		NA
	W2491-28,SO 12707-001	<LOQ (0.0359)	<LOQ	NA	ND	ND - 2 Outliers	NA
Turtle Liver	W2491-37,Male Turtle (-2,8),Turtle Liver	<LOQ (0.0359)			ND		
	W2491-38,Male Turtle (2,12),Turtle Liver	<LOQ (0.0359)		NA	D		NA
	W2491-39,Female Turtle (-3,9),Turtle Liver	<LOQ (0.0359)	<LOQ	NA	D	D - 1 Outlier	NA
Sea Otter Brain	W2491-18,SO 12593-001,Sea Otter Brain	<LOQ (0.0359)		NA	ND		NA
	W2491-21,SO 11494-001,Sea Otter Brain	<LOQ (0.0359)	<LOQ	NA	ND	ND	NA
Sea Otter Kidney	W2491-17,SO 12593-001,Sea Otter Kidney	<LOQ (0.0359)			ND		
	W2491-20,SO 11494-001,Sea Otter Kidney	<LOQ (0.0359)		NA	ND		NA
	W2491-23,SO 11940-001,Sea Otter Kidney	<LOQ (0.0359)	<LOQ	NA	ND	ND	NA
Whole Blood	W2491-54,Cormorant DCCO L Charity	<LOQ (0.0359)			ND		
	W2491-55,Cormorant DCCO Hyrn Island, Lake Sup	<LOQ (0.0359)		NA	D		NA
	W2491-56,Otter DCCO Great Lakes	<LOQ (0.0359)	<LOQ	NA	D	D - 1 Outlier	NA

PFDS/PFHS = no curve analyzed PFHS based on PFOS response.
No PFOS qualitative confirmation performed. Identifications are preliminary.
Deviant Surrogate levels are not noted and were not confirmed.

LOQ = Limit of Quantitation
RSD = Relative Standard Deviation
NA = Not Applicable
ND = Not Detected
D = Detected

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
POAA = Perfluorooctanoate
PFHS = Perfluorohexanesulfonate

Data Entered/Analyst: 08/27/99, 09/01/99, 12/30/99, 01/20/00, 02/14/00 LAC
Data Verified/Analyst: 02/22/00 MEE

Study: GEN021 Various Matrices from MSU
Product Number(Test Substance): None
Matrix: Various Matrices
Method/Revision: ETS-8-6.0 & ETS-8-7.0 using unextracted curves
Analytical Equipment System Number: Amelia 062498
Instrument Software/Version: Masslynx 3.2, 3.3
Date of Extraction/Analyst: 08/25/99 MCH/KK/SAL/SEE
Date of Analysis/Analyst: 08/28/99, 12/09/99 IAS
Date of Data Reduction/Analyst: 08/30/99, 12/10/99 IAS

Sample Data

LIVER/WHOLE BLOOD **QC**

Group Dose	Sample #	Concentration of PFOS ug/g or % Rec.	Mean PFOS Recovery	RSD Std. Dev.	Concentration of PFOSA ug/g or % Rec.	Mean PFOSA ug/g	RSD Std. Dev.
Method Blk	H2O Blk-1 8/25/99	NA			NA		
	H2O Blk-2 8/25/99	NA			NA		
	H2O Blk-3 8/25/99	NA		NA	NA		NA
	H2O Blk-4 8/25/99	NA	NA	NA	NA	NA	NA
California Sea Lion Liver	W2491-3,CSL 3448-MS	91%			66%		
	W2491-4,CSL 3395-MS	78%			52%		
	W2491-6,CSL 3020-MS	69%			48%		
	W2491-7,CSL 2169-MS	45%			30%		
	W2491-9,CSL 2839-MS	31%		35%	15%		42%
	W2491-10,CSL 2367-MS	64%	63%	22%	43%	42%	18%
Elephant Seal Liver	W2491-2,ES 1500-MS	105%			78%		
	W2491-5,ES 1552-MS	77%			65%		
	W2491-11,ES 808-MS	51%			35%		
	W2491-12,ES 772-MS	44%		47%	18%		56%
	W2491-14,ES 782-MS	33%	62%	29%	29%	45%	25%
Harbor Seal Liver	W2491-8,HS-MS	35%			42%		
	W2491-13,HS 1191-MS	81%		42%	79%		39%
	W2491-15,HS 1199-MS	50%	55%	23%	42%	54%	21%
Gonzi Liver	W2491-1,NFS 100-MS	57%	NA	NA	63%	NA	NA
Mink Liver	W2491-34,D1030 USFWS-MS	NR			NR		
	W2491-35,D1146 USFWS-MS	NR		NA	NR		NA
	W2491-36,D1158 USFWS-MS	NR	NR	NA	NR	NR	NA
River Otter Liver	W2491-29,RAG 066-MS	NR			NR		
	W2491-30,RAG 028-MS	NR			NR		
	W2491-31,RAG 148-MS	48%			47%		
	W2491-32,RAG 230-MS	38%		15%	34%		26%
	W2491-33,RAG 237-MS	37%	41%	6%	29%	36%	9%
Sea Otter Liver	W2491-16,SO 12593-001-MS	42%			11%		
	W2491-19,SO 11494-001-MS	61%			50%		
	W2491-22,SO 11940-001-MS	44%			35%		
	W2491-24,SO 11309-001-MS	27%			16%		
	W2491-25,SO 12797-001-MS	34%			22%		
	W2491-26,SO 13110-001-MS	69%			44%		
	W2491-27,SO 12679-001-MS	36%		33%	26%		45%
	W2491-28,SO 12707-001-MS	65%	47%	16%	33%	30%	13%
Turtle Liver	W2491-37,Male Turtle (-2,8)-MS	37%			42%		
	W2491-38,Male Turtle (2,12)-MS	35%		20%	51%		15%
	W2491-39,Female Turtle (-3,9)-MS	51%	41%	8%	39%	44%	6%
Sea Otter Brain	W2491-18,SO 12593-001-MS	42%		7%	-3%		171%
	W2491-21,SO 11494-001-MS	46%	44%	3%	27%	12%	21%
Sea Otter Kidney	W2491-17,SO 12593-001-MS	61%			66%		
	W2491-20,SO 11494-001-MS	75%		49%	57%		61%
	W2491-23,SO 11940-001-MS	24%	53%	26%	14%	46%	28%
Whole Blood	W2491-54,Cormorant DCCO L Charity-MS	63%			65%		
	W2491-55,Cormorant DCCO Hyrn Island, Lake Sup-MS	69%		34%	64%		29%
	W2491-56,Otter DCCO Great Lakes-MS	115%	82%	28%	104%	77%	23%

PFDS/PFHS = no curve analyzed, PFHS based on PFOS response.
No PFOS qualitative confirmation performed. Identifications are preliminary.
NR = Not reported, appears the spike wasn't detectable from endogenous levels.

LOQ = Limit of Quantitation
RSD = Relative Standard Deviation
NA = Not Applicable
NS = Not spiked

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
POAA = Perfluorooctanoate
PFHS = Perfluorohexanesulfonate

Date Entered/Analyst: 02/16/00, 02/17/00 LAC
Date Verified/Analyst: 20/22/00 MEE

Study:	GEN021 Various Matrices from MSU
Product Number(Test Substance):	None
Matrix:	Various Matrices
Method/Revision:	ETS-8-6.0 & ETS-8-7.0 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.2, 3.3
Date of Extraction/Analyst:	08/25/99 MCH/KK/SAL/SEB
Date of Analysis/Analyst:	08/28/99, 12/09/99 LAS
Date of Data Reduction/Analyst:	08/30/99, 12/10/99 LAS

Sample Data

LIVER/WHOLE BLOOD **QC**

Group Dose	Sample #	Concentration of POAA ug/g or % Rec.	Mean POAA ug/g	RSD Std. Dev.	Concentration of PFHS ug/g or % Rec.	Mean PFHS ug/g	RSD Std. Dev.
Method Blk	H2O Blk-1 8/25/99	NA			NS		
	H2O Blk-2 8/25/99	NA			NS		
	H2O Blk-3 8/25/99	NA		NA	NS		NA
	H2O Blk-4 8/25/99	NA	NA	NA	NS	NS	NA
California Sea Lion Liver	W2491-3,CSL 3448-MS	109%			NS		
	W2491-4,CSL 3395-MS	85%			NS		
	W2491-6,CSL 3020-MS	68%			NS		
	W2491-7,CSL 2169-MS	45%			NS		
	W2491-9,CSL 2839-MS	20%		47%	NS		NA
	W2491-10,CSL 2367-MS	64%	65%	31%	NS	NS	NA
Elephant Seal Liver	W2491-2,ES 1500-MS	100%			NS		
	W2491-5,ES 1552-MS	80%			NS		
	W2491-11,ES 808-MS	44%			NS		
	W2491-12,ES 772-MS	31%		52%	NS		NA
	W2491-14,ES 782-MS	36%	58%	30%	NS	NS	NA
Harbor Seal Liver	W2491-8,HS-MS	48%			NS		
	W2491-13,HS 1191-MS	94%		37%	NS		NA
	W2491-15,HS 1199-MS	58%	67%	25%	NS	NS	NA
Canal Liver	W2491-1,NFS 100-MS	91%	NA	NA	NS	NS	NA
Mink Liver	W2491-34,D1030 USFWS-MS	NR			NS		
	W2491-35,D1146 USFWS-MS	NR		NA	NS		NA
	W2491-36,D1158 USFWS-MS	NR	NR	NA	NS	NS	NA
River Otter Liver	W2491-29,RAG 066-MS	21%			NS		
	W2491-30,RAG 028-MS	46%			NS		
	W2491-31,RAG 148-MS	69%			NS		
	W2491-32,RAG 230-MS	52%		39%	NS		NA
	W2491-33,RAG 237-MS	70%	52%	20%	NS	NS	NA
Sea Otter Liver	W2491-16,SO 12593-001-MS	47%			NS		
	W2491-19,SO 11494-001-MS	64%			NS		
	W2491-22,SO 11940-001-MS	47%			NS		
	W2491-24,SO 11309-001-MS	22%			NS		
	W2491-25,SO 12797-001-MS	30%			NS		
	W2491-26,SO 13110-001-MS	83%			NS		
	W2491-27,SO 12679-001-MS	44%		39%	NS		NA
	W2491-28,SO 12707-001-MS	64%	50%	20%	NS	NS	NA
Turtle Liver	W2491-37,Male Turtle (-2,8)-MS	48%			NS		
	W2491-38,Male Turtle (2,12)-MS	71%		29%	NS		NA
	W2491-39,Female Turtle (-3,9)-MS	42%	54%	15%	NS	NS	NA
Sea Otter Brain	W2491-18,SO 12593-001-MS	41%		13%	NS		NA
	W2491-21,SO 11494-001-MS	50%	46%	6%	NS	NS	NA
Sea Otter Kidney	W2491-17,SO 12593-001-MS	94%			NS		
	W2491-20,SO 11494-001-MS	95%		75%	NS		NA
	W2491-23,SO 11940-001-MS	9%	66%	49%	NS	NS	NA
Whole Blood	W2491-54,Cormorant DCCO L Charity-MS	118%			NS		
	W2491-55,Cormorant DCCO Hyrn Island, Lake Sup-MS	122%		2%	NS		NA
	W2491-56,Otter DCCO Great Lakes-MS	123%	121%	3%	NS	NS	NA

PFDS/PFHS = no curve analyzed, PFHS based on PFOS response.
No PFOS qualitative confirmation performed. Identifications are preliminary.
NR = Not reported, appears the spike wasn't detectable from endogenous levels.

LOQ = Limit of Quantitation
RSD = Relative Standard Deviation
NA = Not Applicable
NS = Not spiked

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
POAA = Perfluorooctanoate
PFHS = Perfluorohexanesulfonate

Date Entered/Analyst: 02/16/00, 02/17/00 LAC
Date Verified/Analyst: 20/22/00 MEE

C

Study:	GEN024 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
Method/Revision:	BTS-8-4.1 & BTS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.3
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	10/12/99 SAL/KK
Dates of Analysis/Analyst:	10/15/99, 10/19/99 IAS/MMH, 12/13/99 IAS
Date of Data Reduction/Analyst:	10/18/99, 10/20/99 HOJ, 12/14/99 MMH

Sample Data

SERA

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec	Mean PFOS ug/mL	RSD Std. Dev. RPD-MS/MSD	Concentration of PFHS ug/mL or % Rec	Mean PFHS ug/mL	RSD Std. Dev. RPD-MS/MSD
Method Blk	Bird10129-WBlk-5-1	<LOQ (0.00116 ug/mL)	<LOQ	<LOQ	<LOQ (0.00114 ug/mL)	<LOQ	<LOQ
Matrix Blk	NE	NE			NE		
	NE	NE	NE	NE	NE	NE	NE
MS/MSD	Bird 040-Alb sera-MS-250 ppb-5-1	81%			68%		
250 ppb	Bird 040-Alb sera-MSD-250 ppb-5-1	96%	89%	17%	81%	75%	17%
	Bird 060-E plasma-MS-250 ppb-5-2	11%			59%		
	Bird 060-E plasma-MSD-250 ppb-5-2	23%	17%	69%	65%	62%	11%
	Bird 054-C blood-MS-250 ppb-5	84%			76%		
	Bird 054-C blood-MSD-250 ppb-5	-46%	19%	678%	14%	45%	139%
Albatross	Bird 034-Albatross Chick sera	0.0393			<LOQ (0.00114 ug/mL)		
	Bird 035-Albatross plasma	0.0256			<LOQ (0.00114 ug/mL)		
	Bird 036-Albatross sera	0.00755			<LOQ (0.00114 ug/mL)		
	Bird 037-Albatross plasma	0.0196			<LOQ (0.00114 ug/mL)		
	Bird 038-Albatross sera	0.00548			<LOQ (0.00114 ug/mL)		
	Bird 039-Albatross sera	0.00677			<LOQ (0.00114 ug/mL)		
	Bird 040-Albatross sera	0.00621			<LOQ (0.00114 ug/mL)		
	Bird 041-Albatross sera	0.00430	Plasma	47.0	<LOQ (0.00114 ug/mL)	Plasma	NA
	Bird 042-Albatross sera	0.00402	0.0180	0.00847	<LOQ (0.00114 ug/mL)	<LOQ	NA
	Bird 043-Albatross sera	0.00546			<LOQ (0.00114 ug/mL)		
	Bird 044-Albatross sera	0.00897			<LOQ (0.00114 ug/mL)		
	Bird 045-Albatross sera	0.00657	Sera	115	<LOQ (0.00114 ug/mL)	Sera	NA
	Bird 046-Albatross plasma	0.00889	0.00926	0.0107	<LOQ (0.00114 ug/mL)	<LOQ	NA
Cormorant	Bird 047-Cormorant plasma	0.431			<LOQ (0.00114 ug/mL)		
	Bird 048-Cormorant plasma	0.242		61.8	<LOQ (0.00114 ug/mL)		NA
	Bird 049-Cormorant plasma	0.110	0.261	0.161	<LOQ (0.00114 ug/mL)	<LOQ	NA
	Bird 050-Cormorant blood	0.0725			<LOQ (0.00114 ug/mL)		
	Bird 051-Cormorant blood	0.218			<LOQ (0.00114 ug/mL)		
	Bird 052-Cormorant blood	0.153			<LOQ (0.00114 ug/mL)		
	Bird 053-Cormorant blood	<LOQ (0.00116 ug/mL)			<LOQ (0.00114 ug/mL)		
	Bird 054-Cormorant blood	0.144		44.5	<LOQ (0.00114 ug/mL)		NA
	Bird 055-Cormorant blood	0.273	0.172 - 1 Outlier	0.0764	<LOQ (0.00114 ug/mL)	<LOQ	NA
Herring	Bird 056-Herring Gull plasma	0.453		34.1	0.00243		NA
	Bird 057-Herring Gull plasma	0.277	0.365	0.125	<LOQ (0.00114 ug/mL)	NA	NA
	Bird 058-Herring Gull blood	0.0790		12.2	<LOQ (0.00114 ug/mL)		NA
	Bird 059-Herring Gull blood	0.0664	0.0727	0.00888	<LOQ (0.00114 ug/mL)	<LOQ	NA
Bald Eagle	Bird 060-Bald Eagle plasma	0.449			<LOQ (0.00114 ug/mL)		
	Bird 061-Bald Eagle plasma	0.316			<LOQ (0.00114 ug/mL)		
	Bird 062-Bald Eagle plasma	0.185			0.00148		
	Bird 063-Bald Eagle plasma	0.167			<LOQ (0.00114 ug/mL)		
	Bird 064-Bald Eagle plasma	0.0402			<LOQ (0.00114 ug/mL)		
	Bird 065-Bald Eagle plasma	0.0147			<LOQ (0.00114 ug/mL)		
	Bird 066-Bald Eagle plasma	0.122			<LOQ (0.00114 ug/mL)		
	Bird 067-Bald Eagle plasma	0.0865			<LOQ (0.00114 ug/mL)		
	Bird 068-Bald Eagle plasma	2.57			<LOQ (0.00114 ug/mL)		
	Bird 069-Bald Eagle plasma	2.35			<LOQ (0.00114 ug/mL)		
	Bird 070-Bald Eagle plasma	0.0324			<LOQ (0.00114 ug/mL)		
	Bird 071-Bald Eagle plasma	0.0908			<LOQ (0.00114 ug/mL)		
	Bird 072-Bald Eagle plasma	0.217			<LOQ (0.00114 ug/mL)		
	Bird 073-Bald Eagle plasma	0.168			0.00131		
	Bird 074-Bald Eagle plasma	0.163			<LOQ (0.00114 ug/mL)		
	Bird 075-Bald Eagle plasma	0.0822			<LOQ (0.00114 ug/mL)		
	Bird 076-Bald Eagle plasma	0.0238			<LOQ (0.00114 ug/mL)		
	Bird 077-Bald Eagle plasma	0.0573			<LOQ (0.00114 ug/mL)		
	Bird 078-Bald Eagle plasma	<LOQ (0.00116 ug/mL)			<LOQ (0.00114 ug/mL)		
	Bird 079-Bald Eagle plasma	0.286			<LOQ (0.00114 ug/mL)		
	Bird 080-Bald Eagle plasma	0.281			<LOQ (0.00114 ug/mL)		
	Bird 081-Bald Eagle plasma	0.649			0.00617		
	Bird 082-Bald Eagle plasma	0.335			<LOQ (0.00114 ug/mL)		
	Bird 083-Bald Eagle plasma	0.0718			<LOQ (0.00114 ug/mL)		
	Bird 084-Bald Eagle plasma	0.0381		183	<LOQ (0.00114 ug/mL)		NA
	Bird 085-Bald Eagle plasma	0.118	0.356 - 1 Outlier	0.651	<LOQ (0.00114 ug/mL)	<LOQ - 3 Outliers	NA

No PFOS qualitative confirmation performed. Identifications are preliminary.

* Appears to not have been spiked. LAC 10/19/99

NE = Not extracted
NA = Not Applicable
NS = Not Spiked
LOQ = Limit of Quantitation
RSD = Relative Standard Deviation
RPD = Relative Percent Difference

PFOS = Perfluorooctanesulfonate
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate
PFOSA = Perfluorooctanesulfonamide

Date Entered/By:	10/18/99, 10/20/99 LAC 12/30/99 MMH
Date Verified/ By:	2/17/00 mmh

Study:	GEN024 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
Method/Revision:	ETS-8-4.1 & ETS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.3
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	10/12/99 SAL/KK
Dates of Analysis/Analyst:	10/15/99, 10/19/99 IAS/MMH, 12/13/99 IAS
Date of Data Reduction/Analyst:	10/18/99, 10/20/99 HOJ, 12/14/99 MMH

Sample Data

SERA

Group Dose	Sample #	Concentration of POAA ug/mL or % Rec	Mean POAA ug/mL	RSD Std. Dev. RPD-MS/MSD	Concentration of PFOSA ug/mL or % Rec	Mean PFOSA ug/mL	RSD Std. Dev. RPD-MS/MSD
Method Blk	Bird10129-WBlk-S-1	<LOQ (0.0299 ug/mL)	<LOQ	<LOQ	<LOQ (0.00625 ug/mL)	<LOQ	<LOQ
Matrix Blk	NE	NE			NE		
	NE	NE	NE	NE	NE	NE	NE
MS/MSD	Bird 040-Alb sera-MS-250 ppb-S-1	89%			70%		
250 ppb	Bird 040-Alb sera-MSD-250 ppb-S-1	105%	97%	17%	80%	75%	14%
	Bird 060-E plasma-MS-250 ppb-S-2	77%			59%		
	Bird 060-E plasma-MSD-250 ppb-S-2	84%	80%	9%	65%	62%	9%
	Bird 054-C blood-MS-250 ppb-S	90%			77%		
	Bird 054-C blood-MSD-250 ppb-S	23%	56%	119%	14%	45%	140%
Albatross	Bird 034-Albatross Chick sera	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 035-Albatross plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 036-Albatross sera	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 037-Albatross plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 038-Albatross sera	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 039-Albatross sera	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 040-Albatross sera	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 041-Albatross sera	<LOQ (0.0299 ug/mL)	Plasma	NA	<LOQ (0.00625 ug/mL)	Plasma	NA
	Bird 042-Albatross sera	<LOQ (0.0299 ug/mL)	<LOQ	NA	<LOQ (0.00625 ug/mL)	<LOQ	NA
	Bird 043-Albatross sera	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 044-Albatross sera	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 045-Albatross sera	<LOQ (0.0299 ug/mL)	Sera	NA	<LOQ (0.00625 ug/mL)	Sera	NA
	Bird 046-Albatross plasma	<LOQ (0.0299 ug/mL)	<LOQ	NA	<LOQ (0.00625 ug/mL)	<LOQ	NA
Cormorant	Bird 047-Cormorant plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 048-Cormorant plasma	<LOQ (0.0299 ug/mL)		NA	<LOQ (0.00625 ug/mL)		NA
	Bird 049-Cormorant plasma	<LOQ (0.0299 ug/mL)	<LOQ	NA	<LOQ (0.00625 ug/mL)	<LOQ	NA
	Bird 050-Cormorant blood	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 051-Cormorant blood	<LOQ (0.0299 ug/mL)			0.0145		
	Bird 052-Cormorant blood	<LOQ (0.0299 ug/mL)			0.00871		
	Bird 053-Cormorant blood	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 054-Cormorant blood	<LOQ (0.0299 ug/mL)		NA	<LOQ (0.00625 ug/mL)		82.7
	Bird 055-Cormorant blood	0.0489	<LOQ - 1 Outlier	NA	0.0426	0.0219 - 3 Outliers	0.0181
Herring	Bird 056-Herring Gull plasma	<LOQ (0.0299 ug/mL)		NA	<LOQ (0.00625 ug/mL)		NA
	Bird 057-Herring Gull plasma	<LOQ (0.0299 ug/mL)	<LOQ	NA	<LOQ (0.00625 ug/mL)	<LOQ	NA
	Bird 058-Herring Gull blood	<LOQ (0.0299 ug/mL)		NA	<LOQ (0.00625 ug/mL)		NA
	Bird 059-Herring Gull blood	<LOQ (0.0299 ug/mL)	<LOQ	NA	<LOQ (0.00625 ug/mL)	<LOQ	NA
Bald Eagle	Bird 060-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 061-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 062-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 063-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 064-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 065-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 066-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 067-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 068-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			0.0751		
	Bird 069-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			0.0996		
	Bird 070-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 071-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 072-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 073-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 074-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 075-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 076-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 077-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 078-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 079-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 080-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 081-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 082-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 083-Bald Eagle plasma	<LOQ (0.0299 ug/mL)			<LOQ (0.00625 ug/mL)		
	Bird 084-Bald Eagle plasma	<LOQ (0.0299 ug/mL)		NA	<LOQ (0.00625 ug/mL)		NA
	Bird 085-Bald Eagle plasma	<LOQ (0.0299 ug/mL)	<LOQ	NA	<LOQ (0.00625 ug/mL)	<LOQ - 2 Outliers	NA

No PFOS qualitative confirmation performed. Identifications are preliminary.

* Appears to not have been spiked. LAC 10/19/99

NE = Not extracted
NA = Not Applicable
NS = Not Spiked
LOQ = Limit of Quantitation
RSD = Relative Standard Deviation
RPD = Relative Percent Difference

PFOS = Perfluorooctanesulfonate
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate
PFOSA = Perfluorooctanesulfonamide

Date Entered/By: 10/18/99, 10/20/99 LAC 12/30/99 MMH
Date Verified/ By: 2/17/00 mmh

Study:	GEN024 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
Method/Revision:	ETS-8-6.0 & ETS-8-7.0 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.3
Date of Extraction/Analyst:	10/12/99 SAL/KK
Date of Analysis/Analyst:	10/14/99, 12/13/99 HOJ/IAS
Date of Data Reduction/Analyst:	10/15/99, 11/15/99, 12/14/99 HOJ/MMH

Sample Data

LIVER, KIDNEY, YOLK

Group Dose	Sample #	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/g or % Rec.	Mean PFHS ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	Bird10129-wblk-5-1	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird10129-wblk-6-1	<LOQ (0.0348 ug/g)	<LOQ	NA	<LOQ (0.0683 ug/g)	<LOQ	NA
QC 250 ng/g	Bird001-Loon Lvr-MS	28%			54%		
	Bird001-Loon Lvr-MSD	28%	28%	3%	65%	60%	18%
	Bird023-Albatross Kdny-MS	71%			63%		
	Bird023-Albatross Kdny-MSD	87%	79%	20%	77%	70%	20%
	Bird030-Cormorant Yolk-MS	121%			71%		
	Bird030-Cormorant Yolk-MSD	134%	128%	10%	75%	73%	5%
Liver	Bird001-Loon	0.345			<LOQ (0.0683 ug/g)		
	Bird002-Loon	0.689			<LOQ (0.0683 ug/g)		
	Bird003-Loon	0.185			<LOQ (0.0683 ug/g)		
	Bird004-Loon	0.199			<LOQ (0.0683 ug/g)		
	Bird005-Loon	0.202			<LOQ (0.0683 ug/g)		
	Bird006-Loon	0.105			<LOQ (0.0683 ug/g)		
	Bird007-Loon	<LOQ (0.0348 ug/g)		73.6	<LOQ (0.0683 ug/g)		
	Bird008-Loon	<LOQ (0.0348 ug/g)	0.287 - 2 Outliers	0.212	<LOQ (0.0683 ug/g)	<LOQ	NA
Liver	Bird009-Brown Pelican	0.0460		103	<LOQ (0.0683 ug/g)		
	Bird010-Brown Pelican	0.294	0.170	0.175	<LOQ (0.0683 ug/g)	<LOQ	NA
Liver	Bird011-Albatross	0.617			<LOQ (0.0683 ug/g)		
	Bird012-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird013-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird014-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird015-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird016-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird020-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird021-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird022-Albatross	<LOQ (0.0348 ug/g)	<LOQ - 1 Outlier	NA	<LOQ (0.0683 ug/g)	<LOQ	NA
Kidney	Bird017-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird018-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird019-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird023-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird024-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird025-Albatross	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird026-Albatross	<LOQ (0.0348 ug/g)	<LOQ	NA	<LOQ (0.0683 ug/g)	<LOQ	NA
Yolk	Bird027-Cormorant	<LOQ (0.0348 ug/g)			<LOQ (0.0683 ug/g)		
	Bird028-Cormorant	0.134			<LOQ (0.0683 ug/g)		
	Bird029-Cormorant	0.317		39.5	<LOQ (0.0683 ug/g)		
	Bird030-Cormorant	0.254	0.235 - 1 Outlier	0.0930	<LOQ (0.0683 ug/g)	<LOQ	NA
Yolk	Bird031-Gull	0.146			<LOQ (0.0683 ug/g)		
	Bird032-Gull	<LOQ (0.0348 ug/g)		64.8	<LOQ (0.0683 ug/g)		
	Bird033-Gull	0.0541	0.0999 - 1 Outlier	0.0648	<LOQ (0.0683 ug/g)	<LOQ	NA

No PFOS qualitative confirmation performed. Identifications are preliminary.
LOQ = Limit of Quantitation
RSD = Relative Standard Deviation
RPD = Relative Percent Difference
NA = Not Applicable

PFOS = Perfluorooctanesulfonate
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate
PFOSA = Pefluorooctane sulfonamide

Date Entered/Analyst: 10/23/99 LAC, 12/1/99 GML, 12/30/99 MMH
Date Verified/Analyst: 2/17/00 MMH

Study:	GEN024 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
Method/Revision:	ETS-8-6.0 & ETS-8-7.0 using unextracted curves
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.3
Date of Extraction/Analyst:	10/12/99 SAL/KK
Date of Analysis/Analyst:	10/14/99, 12/13/99 HOJ/IAS
Date of Data Reduction/Analyst:	10/15/99, 11/15/99, 12/14/99 HOJ/MMH

Sample Data

LIVER, KIDNEY, YOLK

Group Dose	Sample #	Concentration of POAA ug/g or % Rec.	Mean POAA 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
Method Blk	Bird10129-wblk-5-1	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird10129-wblk-6-1	<LOQ (0.180 ug/g)	<LOQ	NA	<LOQ (0.00750 ug/g)	<LOQ	NA
QC 250 ng/g	Bird001-Loon Lvr-MS	101%			53%		
	Bird001-Loon Lvr-MSD	108%	104%	6%	51%	52%	4%
	Bird023-Albatross Kdny-MS	113%			56%		
	Bird023-Albatross Kdny-MSD	121%	117%	7%	58%	57%	5%
	Bird030-Cormorant Yolk-MS	105%			62%		
	Bird030-Cormorant Yolk-MSD	130%	117%	22%	72%	67%	15%
Liver	Bird001-Loon	<LOQ (0.180 ug/g)			0.0153		
	Bird002-Loon	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird003-Loon	<LOQ (0.180 ug/g)			0.0147		
	Bird004-Loon	<LOQ (0.180 ug/g)			0.0262		
	Bird005-Loon	<LOQ (0.180 ug/g)			0.0213		
	Bird006-Loon	<LOQ (0.180 ug/g)			0.0242		
	Bird007-Loon	<LOQ (0.180 ug/g)		NA	<LOQ (0.00750 ug/g)		25.5
	Bird008-Loon	<LOQ (0.180 ug/g)	< LOQ	NA	<LOQ (0.00750 ug/g)	0.0204 - 3 Outliers	0.00520
Liver	Bird009-Brown Pelican	<LOQ (0.180 ug/g)		NA	<LOQ (0.00750 ug/g)		
	Bird010-Brown Pelican	<LOQ (0.180 ug/g)	< LOQ	NA	0.178	0.178 - 1 Outlier	NA
Liver	Bird011-Albatross	<LOQ (0.180 ug/g)			0.527		
	Bird012-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird013-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird014-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird015-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird016-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird020-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird021-Albatross	<LOQ (0.180 ug/g)		NA	<LOQ (0.00750 ug/g)		
	Bird022-Albatross	0.182	< LOQ - 1 Outlier	NA	<LOQ (0.00750 ug/g)	<LOQ	NA
Kidney	Bird017-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird018-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird019-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird023-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird024-Albatross	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird025-Albatross	<LOQ (0.180 ug/g)		NA	<LOQ (0.00750 ug/g)		
	Bird026-Albatross	<LOQ (0.180 ug/g)	< LOQ	NA	<LOQ (0.00750 ug/g)	<LOQ	NA
Yolk	Bird027-Comorant	<LOQ (0.180 ug/g)			<LOQ (0.00750 ug/g)		
	Bird028-Comorant	0.245			<LOQ (0.00750 ug/g)		
	Bird029-Comorant	<LOQ (0.180 ug/g)		17.2	<LOQ (0.00750 ug/g)		
	Bird030-Comorant	0.192	0.218- 2 outliers	0.0374	<LOQ (0.00750 ug/g)	<LOQ	NA
Yolk	Bird031-Gull	0.197			<LOQ (0.00750 ug/g)		
	Bird032-Gull	<LOQ (0.180 ug/g)		0.528	<LOQ (0.00750 ug/g)		
	Bird033-Gull	0.196	0.196 - 1 Outlier	0.00104	<LOQ (0.00750 ug/g)	<LOQ	NA

No PFOS qualitative confirmation performed. Identifications are preliminary.
LOQ = Limit of Quantitation
RSD = Relative Standard Deviation
RPD = Relative Percent Difference
NA = Not Applicable
PFOS = Perfluorooctanesulfonate
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate
PFOSA = Perfluorooctane sulfonamide

Date Entered/Analyst: 10/23/99 LAC, 12/1/99 GML, 12/30/99 MMH
Date Verified/Analyst: 2/17/00 MMH

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various Blood
Method/Revision:	ETS-8-4.1 & ETS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Soup020199
Instrument Software/Version:	Masslynx 3.3
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	12/14/99 SAL/SRP/KK
Dates of Analysis/Analyst:	01/06/00, 01/07/00 MMH/IAS
Date of Data Reduction/Analyst:	01/07/00, 01/10/00 IAS/MMH

Sample Data

BLOOD

Group Dose	Sample #	Concentration of PFOS ug/mL or % Rec.	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/mL or % Rec.	Mean PFHS ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	MSU12129-H2Oblk unfiltered 5-3	<LOQ (0.00290 ug/mL)		NA	<LOQ (0.00114 ug/mL)		NA
	MSU12129-H2Oblk filtered 5-4	<LOQ (0.00290 ug/mL)	<LOQ	NA	<LOQ (0.00114 ug/mL)	<LOQ	NA
Matrix Blk	HMB12129-blood blk-5-1	0.0253			<LOQ (0.00114 ug/mL)		
	HMB12129-blood blk-5-2	0.0248			<LOQ (0.00114 ug/mL)		
	HMB12129-blood blk-5-3	0.0262			<LOQ (0.00114 ug/mL)		
	HMB12129-blood blk-5-4	0.0243			<LOQ (0.00114 ug/mL)		
	HMB12129-blood blk-5-5	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	HMB12129-blood blk-5-6*	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	HMB12129-blood blk-5-7	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	HMB12129-blood blk-5-8	<LOQ (0.00579 ug/mL)		NA	<LOQ (0.0114 ug/mL)		NA
	HMB12129-blood blk-5-9	<LOQ (0.00579 ug/mL)	<LOQ - 4 outliers	0.000790	<LOQ (0.0114 ug/mL)	<LOQ	NA
QC	PBB-6255-250MS-5-1-2	74%			76%		
	PBB-6255-250MSD-5-1-2	91%	82%	21%	77%	76%	2%
	FSB-S009-250ppb MS-5-1-2	-6%			-1%		
	FSB-S009-250ppb MSD-5-1-2	-5%	-6%	25%	-1%	-1%	20%
	SSB-SSL49-250ppb MS-5-1-2	77%			83%		
	SSB-SSL49-250ppb MSD-5-1-2	65%	71%	17%	77%	80%	9%
	HMB-FES2189-250 MS-5-1-2	-1%			1%		
	HMB-FES2189-250 MSD-5-1-2	77%	38%	208%	74%	37%	196%
Blood Northern Fur Seal Pups	P205	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P206	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P207	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P208	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P209	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P210	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P211	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P212	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P215	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P217	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P219	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P220	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P221	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P222	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P223	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P224	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P226	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P229	<LOQ (0.00579 ug/mL)		NA	<LOQ (0.0114 ug/mL)		NA
	P230	<LOQ (0.00579 ug/mL)	<LOQ	NA	<LOQ (0.0114 ug/mL)	<LOQ	NA
Blood Northern Fur Seal Adult Females	M104	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	M105	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	M106	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	M107	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	M112	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	M115	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	M116	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	M118	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	M119	<LOQ (0.00579 ug/mL)		NA	<LOQ (0.0114 ug/mL)		NA
	M122	<LOQ (0.00579 ug/mL)	<LOQ	NA	<LOQ (0.0114 ug/mL)	<LOQ	NA
Blood Northern Fur Seal Subadult Males	S001	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	S002	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	S003	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	S006	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	S007	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	S008	<LOQ (0.00579 ug/mL)		NA	<LOQ (0.0114 ug/mL)		NA
	S009	<LOQ (0.00579 ug/mL)	<LOQ	NA	<LOQ (0.0114 ug/mL)	<LOQ	NA
Blood Northern Fur Seal	P298B	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P236A	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	368C	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P406	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	P411	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	98 CUKB 3*	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	98 CUKB 7	<LOQ (0.00579 ug/mL)		NA	<LOQ (0.0114 ug/mL)		NA
NA2 EDTA	98 CUKB 9	<LOQ (0.00579 ug/mL)	<LOQ	NA	<LOQ (0.0114 ug/mL)	<LOQ	NA

* Surrogate >50% deviation, not confirmed

I = May need to rerun all samples for PFOSA, interferent present in both analyses.

** PFOS NOT confirmed; MS transitions variation > 30%

Date Entered/By: 01/21/00 LAC

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Pefluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various Blood
Method/Revision:	ETS-8-4.1 & ETS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Soup020199
Instrument Software/Version:	Masslynx 3.3
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	12/14/99 SAL/SRP/KK
Dates of Analysis/Analyst:	01/06/00, 01/07/00 MMH/IAS
Date of Data Reduction/Analyst:	01/07/00, 01/10/00 IAS/MMH

Sample Data

BLOOD

Group Dose	Sample #	Concentration of POAA ug/mL or % Rec.	Mean POAA 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
Method Blk	MSU12129-H2Oblk unfiltered 5-3	<LOQ (0.00240 ug/mL)		NA	<LOQ (0.000625 ug/mL)		NA
	MSU12129-H2Oblk filtered 5-4	<LOQ (0.00240 ug/mL)	<LOQ	NA	<LOQ (0.000625 ug/mL)	<LOQ	NA
Matrix Blk	HMB12129-blood blk-5-1	<LOQ (0.00240 ug/mL)			0.00195		
	HMB12129-blood blk-5-2	<LOQ (0.00240 ug/mL)			0.00190		
	HMB12129-blood blk-5-3	<LOQ (0.00240 ug/mL)			0.00225		
	HMB12129-blood blk-5-4	<LOQ (0.00240 ug/mL)			0.00189		
	HMB12129-blood blk-5-5	<LOQ (0.00958 ug/mL)			I		
	HMB12129-blood blk-5-6*	<LOQ (0.00958 ug/mL)			I		
	HMB12129-blood blk-5-7	<LOQ (0.00958 ug/mL)			I		
	HMB12129-blood blk-5-8	<LOQ (0.00958 ug/mL)		NA	I		NA
	HMB12129-blood blk-5-9	<LOQ (0.00958 ug/mL)	<LOQ	NA	I	I	NA
QC	PBB-6255-250MS-5-1-2	76%			50%		
	PBB-6255-250MSD-5-1-2	75%	76%	1%	47%	49%	6%
	FSB-S009-250ppb MS-5-1-2	0%			I		
	FSB-S009-250ppb MSD-5-1-2	1%	1%	36%	I	I	NA
	SSB-SSL49-250ppb MS-5-1-2	76%			I		
	SSB-SSL49-250ppb MSD-5-1-2	71%	74%	7%	I	I	NA
	HMB-FE52189-250 MS-5-1-2	1%			I		
	HMB-FE52189-250 MSD-5-1-2	70%	35%	195%	I	I	NA
Blood Northern Fur Seal Pups	P205	<LOQ (0.00958 ug/mL)			I		
	P206	<LOQ (0.00958 ug/mL)			I		
	P207	<LOQ (0.00958 ug/mL)			I		
	P208	<LOQ (0.00958 ug/mL)			I		
	P209	<LOQ (0.00958 ug/mL)			I		
	P210	<LOQ (0.00958 ug/mL)			I		
	P211	<LOQ (0.00958 ug/mL)			I		
	P212	<LOQ (0.00958 ug/mL)			I		
	P215	<LOQ (0.00958 ug/mL)			I		
	P217	<LOQ (0.00958 ug/mL)			I		
	P219	<LOQ (0.00958 ug/mL)			I		
	P220	<LOQ (0.00958 ug/mL)			I		
	P221	<LOQ (0.00958 ug/mL)			I		
	P222	<LOQ (0.00958 ug/mL)			I		
	P223	<LOQ (0.00958 ug/mL)			I		
	P224	<LOQ (0.00958 ug/mL)			I		
	P226	<LOQ (0.00958 ug/mL)			I		
	P229	<LOQ (0.00958 ug/mL)		NA	I		NA
	P230	<LOQ (0.00958 ug/mL)	<LOQ	NA	I	I	NA
Blood Northern Fur Seal Adult Females	M104	<LOQ (0.00958 ug/mL)			I		
	M105	<LOQ (0.00958 ug/mL)			I		
	M106	<LOQ (0.00958 ug/mL)			I		
	M107	<LOQ (0.00958 ug/mL)			I		
	M112	<LOQ (0.00958 ug/mL)			I		
	M115	<LOQ (0.00958 ug/mL)			I		
	M116	<LOQ (0.00958 ug/mL)			I		
	M118	<LOQ (0.00958 ug/mL)			I		
	M119	<LOQ (0.00958 ug/mL)		NA	I		NA
	M122	<LOQ (0.00958 ug/mL)	<LOQ	NA	I	I	NA
Blood Northern Fur Seal Subadult Males	S001	<LOQ (0.00958 ug/mL)			I		
	S002	<LOQ (0.00958 ug/mL)			I		
	S003	<LOQ (0.00958 ug/mL)			I		
	S006	<LOQ (0.00958 ug/mL)			I		
	S007	<LOQ (0.00958 ug/mL)			I		
	S008	<LOQ (0.00958 ug/mL)		NA	I		NA
	S009	<LOQ (0.00958 ug/mL)	<LOQ	NA	I	I	NA
Blood Northern Fur Seal	P298B	<LOQ (0.00958 ug/mL)			I		
	P236A	<LOQ (0.00958 ug/mL)			I		
	368C	<LOQ (0.00958 ug/mL)			I		
	P406	<LOQ (0.00958 ug/mL)			I		
	P411	<LOQ (0.00958 ug/mL)			I		
	98 CUKB 3*	<LOQ (0.00958 ug/mL)			I		
	98 CUKB 7	<LOQ (0.00958 ug/mL)		NA	I		NA
NA2 EDTA	98 CUKB 9	<LOQ (0.00958 ug/mL)	<LOQ	NA	I	I	NA

* Surrogate >50% deviation, not confirmed

I = May need to rerun all samples for PFOSA, interferent present in both analyses.

** PFOS NOT confirmed; MS transitions variation > 30%

Date Entered/By: 01/21/00 LAC

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various Blood
Method/Revision:	ETS-8-4.1 & ETS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Soup020199
Instrument Software/Version:	Masslynx 3.3
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	12/14/99 SAL/SRP/KKK
Dates of Analysis/Analyst:	01/06/00, 01/07/00 MMH/IAS
Date of Data Reduction/Analyst:	01/07/00, 01/10/00 IAS/MMH

Sample Data

BLOOD

Group Dose	Sample #	PFOS Verified	Concentration of PFOS ug/mL or % Rec.	Mean PFOS ug/mL	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/mL or % Rec.	Mean PFHS ug/mL	RSD Std. Dev. MS/MSD RPD
Method Blk	MSU12129-H2Oblk unfiltered 5-3	NA	<LOQ (0.00290 ug/mL)		NA	<LOQ (0.00114 ug/mL)		NA
	MSU12129-H2Oblk filtered 5-4	NA	<LOQ (0.00290 ug/mL)	<LOQ	NA	<LOQ (0.00114 ug/mL)	<LOQ	NA
Matrix Blk	HMB12129-blood blk-5-1	NA	0.0253			<LOQ (0.00114 ug/mL)		
	HMB12129-blood blk-5-2	NA	0.0248			<LOQ (0.00114 ug/mL)		
	HMB12129-blood blk-5-3	NA	0.0262			<LOQ (0.00114 ug/mL)		
	HMB12129-blood blk-5-4	NA	0.0243			<LOQ (0.00114 ug/mL)		
	HMB12129-blood blk-5-5	NA	<LOQ (0.0579 ug/mL)			<LOQ (0.0114 ug/mL)		
	HMB12129-blood blk-5-6*	NA	<LOQ (0.0579 ug/mL)			<LOQ (0.0114 ug/mL)		
	HMB12129-blood blk-5-7	NA	<LOQ (0.0579 ug/mL)			<LOQ (0.0114 ug/mL)		
	HMB12129-blood blk-5-8	NA	<LOQ (0.0579 ug/mL)		NA	<LOQ (0.0114 ug/mL)		NA
	HMB12129-blood blk-5-9	NA	<LOQ (0.0579 ug/mL)	<LOQ - 4 outliers	0.000790	<LOQ (0.0114 ug/mL)	<LOQ	NA
QC	PBB-6255-250MS-5-1-2	NA	74%			76%		
	PBB-6255-250MSD-5-1-2	NA	91%	82%	21%	77%	76%	2%
	FSB-S009-250ppb MS-5-1-2	NA	-6%			-1%		
	FSB-S009-250ppb MSD-5-1-2	NA	-5%	-6%	25%	-1%	-1%	20%
	SSB-SSL49-250ppb MS-5-1-2	NA	77%			83%		
	SSB-SSL49-250ppb MSD-5-1-2	NA	65%	71%	17%	77%	80%	9%
	HMB-FE52189-250 MS-5-1-2	NA	-1%			1%		
	HMB-FE52189-250 MSD-5-1-2	NA	77%	38%	208%	74%	37%	196%
Blood Polar Bear	6255 (Heparin)	X	0.0518			<LOQ (0.00114 ug/mL)		
	20436 (NA2 EDTA)	X	0.0381			<LOQ (0.00114 ug/mL)		
	20467 (NA2 EDTA)	X	0.0358			<LOQ (0.00114 ug/mL)		
	20468 (EDTA)	X	0.0315			<LOQ (0.00114 ug/mL)		
	20470	X	0.0281			<LOQ (0.00114 ug/mL)		
	20472	X	0.0327			<LOQ (0.00114 ug/mL)		
	20473*	X	0.0309			<LOQ (0.00114 ug/mL)		
	20474 (NA2 EDTA)	X	0.0256			<LOQ (0.00114 ug/mL)		
	20475 (NA2 EDTA)	X	<LOQ (0.00290 ug/mL)			<LOQ (0.00114 ug/mL)		
	20476 (NA2 EDTA)	X	0.0345			<LOQ (0.00114 ug/mL)		
	20477	X	0.0272			<LOQ (0.00114 ug/mL)		
	20485 (Heparin)	X	0.0429			<LOQ (0.00114 ug/mL)		
	20486 (Heparin)	X	0.0316		0.214	<LOQ (0.00114 ug/mL)		NA
	20487 (Heparin)	X	<LOQ (0.00290 ug/mL)	0.0342 - 2 outliers	0.00731	<LOQ (0.00114 ug/mL)	<LOQ	NA
Blood Stellar Sealion	SSL49 (7.2 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL50 (7.2 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL51 (7.2 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL52 (7.2 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL53 (7.2 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL54 (7.2 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL55 (5.4 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL56 (5.4 mg K2 EDTA)**	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL57 (5.4 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL58 (5.4 mg K2 EDTA)*	NA	<LOQ (0.00579 ug/mL)			<LOQ (0.0114 ug/mL)		
	SSL59 (5.4 mg K2 EDTA)	NA	<LOQ (0.00579 ug/mL)		NA	<LOQ (0.0114 ug/mL)		NA
	SSL60 (5.4 mg K2 EDTA)*	NA	<LOQ (0.00579 ug/mL)	<LOQ	NA	<LOQ (0.0114 ug/mL)	<LOQ	NA

I = May need to rerun all samples for PFOSA, interferent present in both analyses.
** Surrogate >50% deviation confirmed
* Surrogate >50% deviation, not confirmed
** PFOS NOT confirmed; MS transitions variation > 30%
Date Entered/By: 1/13/00, 01/21/00 MMH/LAC
Date Verified/ By: 02/10/00 kjh

NR = Not Extracted
E = Lost during extraction
NA = Not Applicable
NV = Not Verified
LOQ = Limit of quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various Blood
Method/Revision:	ETS-8-4.1 & ETS-8-5.1 using unextracted curves
Analytical Equipment System Number:	Soup020199
Instrument Software/Version:	Masslynx 3.3
Filename:	See Attachments
R-Squared Value:	See Attachments
Slope:	See Attachments
Y-Intercept:	See Attachments
Dates of Extraction/Analyst:	12/14/99 SAL/SRP/KKK
Dates of Analysis/Analyst:	01/06/00, 01/07/00 MMH/IAS
Date of Data Reduction/Analyst:	01/07/00, 01/10/00 IAS/MMH

Sample Data

BLOOD

Group Dose	Sample #	Concentration of POAA ug/mL or % Rec.	Mean POAA 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
Method Blk	MSU12129-H2Oblk unfiltered 5-3	<LOQ (0.00240 ug/mL)		NA	<LOQ (0.000625 ug/mL)		NA
	MSU12129-H2Oblk filtered 5-4	<LOQ (0.00240 ug/mL)	<LOQ	NA	<LOQ (0.000625 ug/mL)	<LOQ	NA
Matrix Blk	HMB12129-blood blk-5-1	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	HMB12129-blood blk-5-2	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	HMB12129-blood blk-5-3	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	HMB12129-blood blk-5-4	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	HMB12129-blood blk-5-5	<LOQ (0.00958 ug/mL)			I		
	HMB12129-blood blk-5-6*	<LOQ (0.00958 ug/mL)			I		
	HMB12129-blood blk-5-7	<LOQ (0.00958 ug/mL)			I		
	HMB12129-blood blk-5-8	<LOQ (0.00958 ug/mL)		NA	I		NA
	HMB12129-blood blk-5-9	<LOQ (0.00958 ug/mL)	<LOQ	NA	I	I	NA
QC	PBB-6255-250MS-5-1-2	76%			50%		
	PBB-6255-250MSD-5-1-2	75%	76%	1%	47%	49%	6%
	FSB-S009-250ppb MS-5-1-2	0%			I		
	FSB-S009-250ppb MSD-5-1-2	1%	1%	36%	I	I	NA
	SSB-SSL49-250ppb MS-5-1-2	76%			I		
	SSB-SSL49-250ppb MSD-5-1-2	71%	74%	7%	I	I	NA
	HMB-FE52189-250 MS-5-1-2	1%			I		
	HMB-FE52189-250 MSD-5-1-2	70%	35%	195%	I	I	NA
Blood Polar Bear	6255 (Heparin)	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20436 (NA2 EDTA)	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20467 (NA2 EDTA)	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20468 (EDTA)	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20470	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20472	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20473*	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20474 (NA2 EDTA)	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20475 (NA2 EDTA)	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20476 (NA2 EDTA)	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20477	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20485 (Heparin)	<LOQ (0.00240 ug/mL)			<LOQ (0.000625 ug/mL)		
	20486 (Heparin)	<LOQ (0.00240 ug/mL)		NA	<LOQ (0.000625 ug/mL)		NA
	20487 (Heparin)	<LOQ (0.00240 ug/mL)	<LOQ	NA	<LOQ (0.000625 ug/mL)	<LOQ	NA
Blood Stellar Sealion	SSL49 (7.2 mg K2 EDTA)	<LOQ (0.00958 ug/mL)			I		
	SSL50 (7.2 mg K2 EDTA)	<LOQ (0.00958 ug/mL)			I		
	SSL51 (7.2 mg K2 EDTA)	<LOQ (0.00958 ug/mL)			I		
	SSL52 (7.2 mg K2 EDTA)	<LOQ (0.00958 ug/mL)			I		
	SSL53 (7.2 mg K2 EDTA)	<LOQ (0.00958 ug/mL)			I		
	SSL54 (7.2 mg K2 EDTA)	<LOQ (0.00958 ug/mL)			I		
	SSL55 (5.4 mg K2 EDTA)	<LOQ (0.00958 ug/mL)			I		
	SSL56 (5.4 mg K2 EDTA)**	<LOQ (0.00958 ug/mL)			I		
	SSL57 (5.4 mg K2 EDTA)	<LOQ (0.00958 ug/mL)			I		
	SSL58 (5.4 mg K2 EDTA)*	<LOQ (0.00958 ug/mL)			I		
	SSL59 (5.4 mg K2 EDTA)	<LOQ (0.00958 ug/mL)		NA	I		NA
	SSL60 (5.4 mg K2 EDTA)*	<LOQ (0.00958 ug/mL)	<LOQ	NA	I	I	NA

I = May need to rerun all samples for PFOSA, interferent present in both analyse
** Surrogate >50% deviation confirmed
* Surrogate >30% deviation, not confirmed
** PFOS NOT confirmed; MS transitions variation > 30%

NB = Not Extracted
E = Lost during extraction
NA = Not Applicable
NV = Not Verified
LOQ = Limit of quantitation

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Date Entered/By:	1/13/00, 01/21/00 MMH/LAC
Date Verified/ By:	02/10/00 kjh

FACT-GEN-030
MSU Environmental Samples

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
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:	12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	01/10/00 MMH
Date of Data Reduction/Analyst:	01/12/00 LAS

Sample Data

MUSCLE

Group Dose	Sample #	PFOS Verified	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/g or % Rec.	Mean PFHS ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	MSU12149-H2Ounfil 5-1*	NA	0.0163		NA	<LOQ (0.0342)		NA
Unfiltered water	MSU12149-H2Ounfil 5-2*	NA	<LOQ (0.00696)	NA	NA	<LOQ (0.0342)	<LOQ	NA
Method Blk	MSU12149-H2Ofil 5-1*	NA	<LOQ (0.00696)		NA	<LOQ (0.0342)		NA
Filtered water	MSU12149-H2Ofil 5-2*	NA	0.0257	NA	NA	<LOQ (0.0342)	<LOQ	NA
Matrix Blk	MSU12149-Fishblk 5-1	NA	0.00948			<LOQ (0.0342)		
Fish Liver	MSU12149-Fishblk 5-2	NA	0.00985		37.2	<LOQ (0.0342)		NA
	MSU12149-Fishblk 5-3*	NA	0.00454	0.00796	0.00296	<LOQ (0.0342)	<LOQ	NA
QC	CPM-B1N7-MS 5-1-2	NA	59%			60%		
250 ng/g	CPM-B2N6-MS 5-1-2	NA	31%	45%	63%	45%	53%	28%
	CSM-1999030-03-01-MS 5-1-2	NA	160%			148%		
	CSM-1999030-03-01-MSD 5-1-2	NA	127%	143%	23%	183%	166%	21%
	LWM-1999029-16-MS 5-1-2	NA	67%			129%		
	LWM-1999029-11-MS 5-1-3	NA	122%	95%	59%	130%	130%	0%
	BTM-1999040-09-MS 5-1-2	NA	148%			144%		
	BTM-1999040-10-MS 5-1-2	NA	166%	157%	11%	168%	156%	15%
Muscle	B1N1	X	0.101			<LOQ (0.0342)		
Carp	B1N2	X	0.0784			<LOQ (0.0342)		
	B1N7	X	0.0905			<LOQ (0.0342)		
	B1N10	X	0.0878			<LOQ (0.0342)		
	B2N2	X	0.105			<LOQ (0.0342)		
	B2N6	X	0.0894			<LOQ (0.0342)		
	B2N8	X	0.0596			<LOQ (0.0342)		
	B2N10	X	0.0838			<LOQ (0.0342)		
	Carp1*	X	0.297		64.2	<LOQ (0.0342)		NA
	Carp2*	X	0.243	0.124	0.0793	<LOQ (0.0342)	<LOQ	NA
Muscle	1999030-01	NA	0.189			<LOQ (0.0342)		
Chinook Salmon	1999030-02	NA	0.126			<LOQ (0.0342)		
	1999030-02-01	X	<LOQ (0.00696)			<LOQ (0.0342)		
	1999030-02-04	X	0.113			<LOQ (0.0342)		
	1999030-03-01	X	0.0514		0.524	<LOQ (0.0342)		NA
	1999030-03-04	X	0.0573	0.107 - 1 outlier	0.0562	<LOQ (0.0342)	<LOQ	NA
Muscle	1999029-11*	X	0.168			<LOQ (0.0342)		
Lake Whitefish	1999029-12*	X	0.130			<LOQ (0.0342)		
	1999029-13	X	0.0967			<LOQ (0.0342)		
	1999029-14	X	0.0983		26.4	<LOQ (0.0342)		NA
	1999029-16	X	0.1659	0.132	0.0348	<LOQ (0.0342)	<LOQ	NA
Muscle	1999040-01	X	<LOQ (0.00696)			<LOQ (0.0342)		
Brown Trout	1999040-02 **	X **	<LOQ (0.00696)			<LOQ (0.0342)		
	1999040-03	NA	<LOQ (0.00696)			<LOQ (0.0342)		
	1999040-04	NA	<LOQ (0.00696)			<LOQ (0.0342)		
	1999040-05	X	<LOQ (0.00696)			<LOQ (0.0342)		
	1999040-06	X	<LOQ (0.00696)			<LOQ (0.0342)		
	1999040-07	NA	<LOQ (0.00696)			<LOQ (0.0342)		
	1999040-08	X	0.0460			<LOQ (0.0342)		
	1999040-09	NA	<LOQ (0.00696)		NA	<LOQ (0.0342)		NA
	1999040-10	NA	<LOQ (0.00696)	<LOQ - 1 outlier	NA	<LOQ (0.0342)	<LOQ	NA

* High (>50%) surrogate deviations

** PFOS NOT confirmed; MS transitions variation > 30%

Date Entered/Analyst: 01/24/00, 01/25/00, 01/28/00 LAC

Date Verified/Analyst: 02/10/00 kjh

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation
X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

FACT-GEN-030
MSU Environmental Samples

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
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:	12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	01/10/00 MMH
Date of Data Reduction/Analyst:	01/12/00 IAS

Sample Data

MUSCLE

Group Dose	Sample #	Concentration of POAA ng/g or % Rec.	Mean POAA ng/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ng/g or % Rec.	Mean PFOSA ng/g	RSD Std. Dev. MS/MSD RPD
Method Blk	MSU12149-H2Ounfil 5-1*	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
Unfiltered water	MSU12149-H2Ounfil 5-2*	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Method Blk	MSU12149-H2Ofil 5-1*	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
Filtered water	MSU12149-H2Ofil 5-2*	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Matrix Blk	MSU12149-Fishblk 5-1	<LOQ (0.0359)			<LOQ (0.0188)		
Fish Liver	MSU12149-Fishblk 5-2	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
	MSU12149-Fishblk 5-3*	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
QC	CPM-B1N7-MS 5-1-2	6%			11%		
250 ng/g	CPM-B2N6-MS 5-1-2	39%	23%	148%	13%	12%	19%
	CSM-1999030-03-01-MS 5-1-2	139%			77%		
	CSM-1999030-03-01-MSD 5-1-2	178%	158%	25%	69%	73%	11%
	LWM-1999029-16-MS 5-1-2	146%			61%		
	LWM-1999029-11-MS 5-1-3	105%	125%	33%	76%	68%	22%
	BTM-1999040-09-MS 5-1-2	147%			80%		
	BTM-1999040-10-MS 5-1-2	152%	150%	3%	87%	83%	8%
Muscle	B1N1	<LOQ (0.0359)			<LOQ (0.0188)		
Carp	B1N2	<LOQ (0.0359)			<LOQ (0.0188)		
	B1N7	<LOQ (0.0359)			<LOQ (0.0188)		
	B1N10	<LOQ (0.0359)			<LOQ (0.0188)		
	B2N2	<LOQ (0.0359)			<LOQ (0.0188)		
	B2N6	<LOQ (0.0359)			<LOQ (0.0188)		
	B2N8	<LOQ (0.0359)			<LOQ (0.0188)		
	B2N10	<LOQ (0.0359)			<LOQ (0.0188)		
	Carp1*	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
	Carp2*	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Muscle	1999030-01	<LOQ (0.0359)			<LOQ (0.0188)		
Chinook Salmon	1999030-02	<LOQ (0.0359)			<LOQ (0.0188)		
	1999030-02-01	<LOQ (0.0359)			<LOQ (0.0188)		
	1999030-02-04	<LOQ (0.0359)			<LOQ (0.0188)		
	1999030-03-01	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
	1999030-03-04	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Muscle	1999029-11*	<LOQ (0.0359)			<LOQ (0.0188)		
Lake Whitefish	1999029-12*	<LOQ (0.0359)			<LOQ (0.0188)		
	1999029-13	<LOQ (0.0359)			<LOQ (0.0188)		
	1999029-14	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
	1999029-16	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Muscle	1999040-01	<LOQ (0.0359)			<LOQ (0.0188)		
Brown Trout	1999040-02 **	<LOQ (0.0359)			<LOQ (0.0188)		
	1999040-03	<LOQ (0.0359)			<LOQ (0.0188)		
	1999040-04	<LOQ (0.0359)			<LOQ (0.0188)		
	1999040-05	<LOQ (0.0359)			<LOQ (0.0188)		
	1999040-06	<LOQ (0.0359)			<LOQ (0.0188)		
	1999040-07	<LOQ (0.0359)			<LOQ (0.0188)		
	1999040-08	<LOQ (0.0359)			<LOQ (0.0188)		
	1999040-09	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
	1999040-10	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA

* High (>50%) surrogate deviations

** PFOS NOT confirmed; MS transitions variation > 30%

Date Entered/Analyst: 01/24/00, 01/25/00, 01/28/00 LAC

Date Verified/Analyst: 02/10/00 kjh

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation
X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
Method/Revision:	BTS-8-6.0 & BTS-8-7.0
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.3
Date of Extraction/Analyst:	12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	01/10/00 MMH
Date of Data Reduction/Analyst:	01/11/00 IAS

Sample Data

MUSCLE and EGGS

Group Dose	Sample #	PFOS Verified	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/g or % Rec.	Mean PFHS ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	MSU12159-H2OBlk-unfiltered-5-9	NA	<LOQ (0.0174)		NA	<LOQ (0.0342)		NA
Water	MSU12159-H2OBlk-filtered-5-9	NA	<LOQ (0.0174)	<LOQ	NA	<LOQ (0.0342)	<LOQ	NA
Matrix Blk	EggBlk-5-1	NA	<LOQ (0.0174)			<LOQ (0.0342)		
Egg	EggBlk-5-2	NA	<LOQ (0.0174)		NA	<LOQ (0.0342)		NA
	EggBlk-5-3	NA	<LOQ (0.0174)	<LOQ	NA	<LOQ (0.0342)	<LOQ	NA
QC	FGW-ALSWCR-TD2 6/25/98-MS-5-1-2	X	44%			16%		
250 ng/g	FGW-ALSWCR-TD2 6/25/98-MSD-5-1-2	X	47%	46%	8%	16%	16%	3%
	FGE-AL118-HP26/25/98-MS-5-1-2	X	13%			10%		
	FGE-AL118-HP26/25/98-MSD-5-1-2	X	15%	14%	8%	12%	11%	13%
	LWE-19999029-13-MS-5-1-2	X	146%			30%		
	LWE-19999029-13-MSD-5-1-2	X	104%	125%	33%	35%	33%	16%
	BTE-19999040-01-MS-5-1-2	X	121%			57%		
	BTE-19999040-01-MSD-5-1-2	X	167%	144%	32%	57%	57%	1%
Carp	Diet 1 (Carp5) **	X **	<LOQ (0.0174)			<LOQ (0.0342)		
	Diet 2 (Carp6)	X	<LOQ (0.0174)			<LOQ (0.0342)		
	Diet 3 (Carp4)	X	0.0267		NA	<LOQ (0.0342)		NA
	Diet 4 (Carp3)	X	0.0278	0.0272 - 2 outliers	NA	<LOQ (0.0342)	<LOQ	NA
Frog Muscle	AL-118-YOY 08/26/98 **	X **	0.00243			<LOQ (0.0342)		
Wholebody	ALSWCR-TD 06/03/98 **	X **	<LOQ (0.0174)			<LOQ (0.0342)		
	ALSWCR-TD2 06/25/99 **	X **	<LOQ (0.0174)			<LOQ (0.0342)		
	KZCKDM-JUV 08/26/98 **	X **	<LOQ (0.0174)			<LOQ (0.0342)		
	KZCKDM-TD 06/05/98 **	X **	0.0216			<LOQ (0.0342)		
	KZCKDM-TD-2 06/25/98 **	X **	<LOQ (0.0174)			<LOQ (0.0342)		
	SJ0002-TD-2 06/05/98 **	X **	<LOQ (0.0174)		NA	<LOQ (0.0342)		NA
	SJ0002-TD-2 06/25/98 **	X **	<LOQ (0.0174)	<LOQ - 2 outliers	NA	<LOQ (0.0342)	<LOQ	NA
Green Frog Eggs	AL-118-HP99 06/25/98	X	<LOQ (0.0174)			<LOQ (0.0342)		
	AL-118-HP94 06/25/98	X	<LOQ (0.0174)			<LOQ (0.0342)		
	SJ0001 06/03/98 **	X **	<LOQ (0.0174)		NA	<LOQ (0.0342)		NA
	SJ0001 06/05/98	X	<LOQ (0.0174)	<LOQ	NA	<LOQ (0.0342)	<LOQ	NA
Lake Whitefish Eggs	1999029-13	X	0.145		63.5	<LOQ (0.0342)		NA
	1999029-14	X	0.381	0.263	0.167	<LOQ (0.0342)	<LOQ	NA
Brown Trout Eggs	1999040-01	X	0.0749			<LOQ (0.0342)		
	1999040-04	X	0.0675		21.1	<LOQ (0.0342)		NA
	1999040-06	X	0.0488	0.0637	0.0134	<LOQ (0.0342)	<LOQ	NA

* High (>50%) surrogate deviations
** PFOS NOT confirmed; MS transitions variation > 30%
Date Entered/Analyst: 01/21/00, 01/24/00 LAC
Date Verified/Analyst: 02/10/00 kjh

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation
X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
Method/Revision:	BTS-8-6.0 & BTS-8-7.0
Analytical Equipment System Number:	Amelia 062498
Instrument Software/Version:	Masslynx 3.3
Date of Extraction/Analyst:	12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	01/10/00 MMH
Date of Data Reduction/Analyst:	01/11/00 IAS

Sample Data

MUSCLE and EGGS

Group Dose	Sample #	Concentration of POAA ug/g or % Rec.	Mean POAA 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
Method Blk	MSU12159-H2OBlk-unfiltered-5-9	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
Water	MSU12159-H2OBlk-filtered-5-9	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Matrix Blk	EggBlk-5-1	<LOQ (0.0359)			<LOQ (0.0188)		
Egg	EggBlk-5-2	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
	EggBlk-5-3	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
QC	FGW-ALSWCR-TD2 6/25/98-MS-5-1-2	34%			49%		
250 ng/g	FGW-ALSWCR-TD2 6/25/98-MSD-5-1-2	33%	34%	4%	49%	49%	1%
	FGE-AL118-HP26/25/98-MS-5-1-2	22%			46%		
	FGE-AL118-HP26/25/98-MSD-5-1-2	26%	24%	15%	54%	50%	16%
	LWE-19999029-13-MS-5-1-2	90%			68%		
	LWE-19999029-13-MSD-5-1-2	83%	86%	9%	64%	66%	6%
	BTE-19999040-01-MS-5-1-2	98%			104%		
	BTE-19999040-01-MSD-5-1-2	112%	105%	13%	108%	106%	4%
Carp	Diet 1 (Carp5) **	<LOQ (0.0359)			<LOQ (0.0188)		
	Diet 2 (Carp6)	<LOQ (0.0359)			<LOQ (0.0188)		
	Diet 3 (Carp4)	<LOQ (0.0359)		NA	<LOQ (0.0188)		NA
	Diet 4 (Carp3)	<LOQ (0.0359)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Frog Muscle	AL-118-YOY 08/26/98 **	<LOQ (0.0180)			<LOQ (0.0188)		
Wholebody	ALSWCR-TD 06/03/98 **	<LOQ (0.0180)			<LOQ (0.0188)		
	ALSWCR-TD2 06/25/99 **	<LOQ (0.0180)			<LOQ (0.0188)		
	KZCKDM-JUV 08/26/98 **	<LOQ (0.0180)			<LOQ (0.0188)		
	KZCKDM-TD 06/05/98 **	<LOQ (0.0180)			<LOQ (0.0188)		
	KZCKDM-TD-2 06/25/98 **	<LOQ (0.0180)			<LOQ (0.0188)		
	SJ0002-TD-2 06/05/98 **	<LOQ (0.0180)		NA	<LOQ (0.0188)		NA
	SJ0002-TD-2 06/25/98 **	<LOQ (0.0180)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Green Frog Eggs	AL-118-HP80 06/25/98	<LOQ (0.0180)			<LOQ (0.0188)		
	AL-118-HP94 06/25/98	<LOQ (0.0180)			<LOQ (0.0188)		
	SJ0001 06/03/98 **	<LOQ (0.0180)		NA	<LOQ (0.0188)		NA
	SJ0001 06/05/98	<LOQ (0.0180)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Lake Whitefish Eggs	1999029-13	<LOQ (0.0180)		NA	<LOQ (0.0188)		NA
	1999029-14	<LOQ (0.0180)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Brown Trout Eggs	1999040-01	<LOQ (0.0180)			<LOQ (0.0188)		
	1999040-04	<LOQ (0.0180)		NA	<LOQ (0.0188)		NA
	1999040-06	<LOQ (0.0180)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA

* High (>50%) surrogate deviations
** PFOS NOT confirmed; MS transitions variation > 30%
Date Entered/Analyst: 01/21/00, 01/24/00 LAC
Date Verified/Analyst: 02/10/00 kjh

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POAA = Perfluorooctanoate

FACT-GEN-030
MSU Environmental Samples

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance)	Various
Matrix:	Various
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:	12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	12/17/99, 12/20/99, 12/28/99, 12/29/99, 01/03/00, 01/06/00 IAS/MMH
Date of Data Reduction/Analyst:	12/20/99, 12/21/99, 12/22/99, 12/30/99, 01/03/00, 01/05/00, 01/07/00 MMH/IAS

Sample Data

Miscellaneous Liver

Group Dose	Sample #	PFOS Verified	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/g or % Rec.	Mean PFHS ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	MSU12129-H2OBlk-unfiltered-5-1	NA	NA			NA		
Unfiltered water	MSU12129-H2OBlk-unfiltered-5-2	NA	NA			NA		
	MSU12129-H2OBlk-unfiltered-5-3	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-H2OBlk-unfiltered-5-4	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-H2OBlk-unfiltered-5-5	NA	E			E		
	MSU12129-H2OBlk-unfiltered-5-6	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	MSU12129-H2OBlk-unfiltered-5-7	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	MSU12129-H2OBlk-unfiltered-5-8	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
Method Blk	MSU12129-H2OBlk-filtered-5-1	NA	NA			NA		
Filtered water	MSU12129-H2OBlk-filtered-5-2	NA	NA			NA		
	MSU12129-H2OBlk-filtered-5-3	NA	E			E		
	MSU12129-H2OBlk-filtered-5-4	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-H2OBlk-filtered-5-5	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-H2OBlk-filtered-5-6	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	MSU12129-H2OBlk-filtered-5-7	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	MSU12129-H2OBlk-filtered-5-8	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
Matrix Blk	FSH12129-LvrBlk-5-1	NA	0.0305			<LOQ (0.0171)		
Fish Liver	FSH12129-LvrBlk-5-2	NA	0.0331		32.1	<LOQ (0.0171)		NA
	FSH12129-LvrBlk-5-3	NA	0.0170	0.0269	0.00862	<LOQ (0.0171)	<LOQ	NA
Matrix Blk	RBL12129-LvrBlk-5-1	NA	<LOQ (0.0347)			<LOQ (0.00683)		
Rabbit Liver	RBL12129-LvrBlk-5-2	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	RBL12129-LvrBlk-5-3	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	RBL12129-LvrBlk-5-4	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	RBL12129-LvrBlk-5-5*	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	RBL12129-LvrBlk-5-6*	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
QC	Mink Liver, D530, MS-5-1-1	NA	145%			61%		
250 ng/g	Mink Liver, D530, MSD-5-1-2*	NA	538%	342%	115%	50%	55%	21%
	CSL-1999030-03-01-MS-5-1-2*	NA	196%			116%		
	CSL-1999030-03-01-MSD-5-1-2*	NA	140%	168%	33%	112%	114%	4%
	LWL-1999029-12-MS-5-1-2*	NA	321%			57%		
	LWL-1999029-12-MSD-5-1-2*	NA	287%	304%	11%	68%	63%	18%
	BTL-1999040-01-MS-5-1-2*	NA	138%			81%		
	BTL-1999040-01-MSD-5-1-2*	NA	132%	135%	5%	84%	83%	4%
	TNL-TU54-MS-5-1-2	NA	68%			55%		
	TNL-TU54-MSD-5-1-2	NA	E	68%	NA	E	55%	NA
	FSL-P295-MS-5-1	NA	91%			97%		
	FSL-P295-MSD-5-1	NA	86%	88%	6%	84%	90%	15%
	PBL-980390LB-MS-5-1	NA	18%			70%		
	PBL-980390LB-MSD-5-2*	NA	179%	99%	163%	76%	73%	9%
	GFL-KZCKDM-D1-MS-5-1-2	NA	93%			82%		
	GFL-KZCKDM-D1-MSD-5-1-	NA	105%	99%	12%	89%	86%	9%
	TTL-LCPTR99503C-MS-5-1-2*	NA	90%			66%		
	TTL-LCPTR99503C-MSD-5-1-2	NA	100%	95%	11%	72%	69%	8%
	MTL-10Vancleave98-MS-5-1-2	NA	79%			74%		
	MTL-10Vancleave98-MSD-5-1-2	NA	89%	84%	11%	84%	79%	13%
Liver	1999030-01	X	0.109			<LOQ (0.0171)		
Chinook Salmon	1999030-02	X	0.169			<LOQ (0.0171)		
	1999030-02-01	NA	0.0328			<LOQ (0.0171)		
	1999030-02-04	NA	0.126			<LOQ (0.0171)		
	1999030-03-01	NA	0.173		56.1	<LOQ (0.0171)		NA
	1999030-03-04	NA	0.0405	0.108	0.0608	<LOQ (0.0171)	<LOQ	NA
Liver	1999029-11	NA	0.0679			<LOQ (0.0171)		
Lake Whitefish	1999029-12	NA	0.0812			<LOQ (0.0171)		
	1999029-13	X	0.0738			<LOQ (0.0171)		
	1999029-14	X	0.0329		29.3	<LOQ (0.0171)		NA
	1999029-16	NA	0.0778	0.0667	0.0195	<LOQ (0.0171)	<LOQ	NA
Liver	1999040-01	NA	<LOQ (0.0174)			<LOQ (0.0171)		
Brown Trout	1999040-02	NA	<LOQ (0.0174)			<LOQ (0.0171)		
	1999040-03	NA	<LOQ (0.0174)			<LOQ (0.0171)		
	1999040-04	NA	<LOQ (0.0174)			<LOQ (0.0171)		
	1999040-05	NA	0.0255			<LOQ (0.0171)		
	1999040-06	NA	<LOQ (0.0174)			<LOQ (0.0171)		
	1999040-07	NA	<LOQ (0.0174)			<LOQ (0.0171)		
	1999040-08	NA	<LOQ (0.0174)			<LOQ (0.0171)		
	1999040-09	NA	<LOQ (0.0174)		NA	<LOQ (0.0171)		NA
	1999040-10	NA	<LOQ (0.0174)	<LOQ - 1 outlier	NA	<LOQ (0.0171)	<LOQ	NA

* High (>50%) surrogate deviations

Date Entered/Analyst: 12/22/99, 12/28/99, 12/29/99, 12/30/99, 01/12/00, 01/17/00, 01/18/00 LAC

Date Verified/Analyst: 0

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation
X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
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:	12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	12/17/99, 12/20/99, 12/28/99, 12/29/99, 01/03/00, 01/06/00 IAS/MMH
Date of Data Reduction/Analyst:	12/20/99, 12/21/99, 12/22/99, 12/30/99, 01/03/00, 01/05/00, 01/07/00 MMH/IAS

Sample Data

Miscellaneous Liver

Group Dose	Sample #	Concentration of POAA ug/g or % Rec.	Mean POAA 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
Method Blk	MSU12129-H2OBlk-unfiltered-5-1	NA			NA		
Unfiltered water	MSU12129-H2OBlk-unfiltered-5-2	NA			NA		
	MSU12129-H2OBlk-unfiltered-5-3	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-5	E			E		
	MSU12129-H2OBlk-unfiltered-5-6	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-7	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	MSU12129-H2OBlk-unfiltered-5-8	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Method Blk	MSU12129-H2OBlk-filtered-5-1	NA			NA		
Filtered water	MSU12129-H2OBlk-filtered-5-2	NA			NA		
	MSU12129-H2OBlk-filtered-5-3	E			E		
	MSU12129-H2OBlk-filtered-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-5	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-6	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-7	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	MSU12129-H2OBlk-filtered-5-8	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Matrix Blk	FSH12129-LvrBlk-5-1	<LOQ (0.0719)			<LOQ (0.0188)		
Fish Liver	FSH12129-LvrBlk-5-2	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	FSH12129-LvrBlk-5-3	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Matrix Blk	RBL12129-LvrBlk-5-1	<LOQ (0.0719)			<LOQ (0.0188)		
Rabbit Liver	RBL12129-LvrBlk-5-2	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-3	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-5*	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	RBL12129-LvrBlk-5-6*	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
QC	Mink Liver, D530, MS-5-1-1	86%			43%		
250 ng/g	Mink Liver, D530, MSD-5-1-2*	84%	85%	3%	81%	62%	60%
	CSL-1999030-03-01-MS-5-1-2*	131%			64%		
	CSL-1999030-03-01-MSD-5-1-2*	111%	121%	17%	52%	58%	21%
	LWL-1999029-12-MS-5-1-2*	133%			77%		
	LWL-1999029-12-MSD-5-1-2*	145%	139%	9%	83%	80%	7%
	BTL-1999040-01-MS-5-1-2*	127%			94%		
	BTL-1999040-01-MSD-5-1-2*	120%	124%	6%	92%	93%	3%
	TNL-TU54-MS-5-1-2	84%			71%		
	TNL-TU54-MSD-5-1-2	E	84%	NA	E	71%	NA
	FSL-P295-MS-5-1	72%			87%		
	FSL-P295-MSD-5-1	80%	76%	10%	82%	85%	7%
	PBL-980390LB-MS-5-1	85%			71%		
	PBL-980390LB-MSD-5-2*	129%	107%	41%	65%	68%	9%
	GFL-KZCKDM-D1-MS-5-1-2	82%			82%		
	GFL-KZCKDM-D1-MSD-5-1-	103%	92%	23%	93%	88%	12%
	TTL-LCPTR99503C-MS-5-1-2*	73%			78%		
	TTL-LCPTR99503C-MSD-5-1-2	64%	69%	13%	71%	74%	10%
	MTL-10Vancleave98-MS-5-1-2	100%			74%		
	MTL-10Vancleave98-MSD-5-1-2	75%	88%	29%	74%	74%	0%
Liver	1999030-01	<LOQ (0.0719)			<LOQ (0.0188)		
Chinook Salmon	1999030-02	<LOQ (0.0719)			<LOQ (0.0188)		
	1999030-02-01	<LOQ (0.0719)			<LOQ (0.0188)		
	1999030-02-04	<LOQ (0.0719)			<LOQ (0.0188)		
	1999030-03-01	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	1999030-03-04	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Liver	1999029-11	<LOQ (0.0719)			<LOQ (0.0188)		
Lake Whitefish	1999029-12	<LOQ (0.0719)			<LOQ (0.0188)		
	1999029-13	<LOQ (0.0719)			<LOQ (0.0188)		
	1999029-14	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	1999029-16	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Liver	1999040-01	<LOQ (0.0719)			<LOQ (0.0188)		
Brown Trout	1999040-02	<LOQ (0.0719)			<LOQ (0.0188)		
	1999040-03	<LOQ (0.0719)			<LOQ (0.0188)		
	1999040-04	<LOQ (0.0719)			<LOQ (0.0188)		
	1999040-05	<LOQ (0.0719)			<LOQ (0.0188)		
	1999040-06	<LOQ (0.0719)			<LOQ (0.0188)		
	1999040-07	<LOQ (0.0719)			<LOQ (0.0188)		
	1999040-08	<LOQ (0.0719)			<LOQ (0.0188)		
	1999040-09	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	1999040-10	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA

* High (>50%) surrogate deviations

Date Entered/Analyst: 12/22/99, 12/28/99, 12/29/99, 12/30/99, 01/12/00, 01/17/00, 01/18/00 LAC

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Study:	GEN-030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
Method/Revision:	ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number:	Sonp 020199, Aurelia 062498
Instrument Software/Version:	Masslynx 3.3
Date of Extraction/Analyst:	12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	12/17/99, 12/20/99, 12/28/99, 12/29/99, 01/03/00, 01/05/00, 01/06/00, 01/08/00 LAS/MMH
Date of Data Reduction/Analyst:	12/20/99, 12/21/99, 12/22/99, 12/30/99, 01/03/00, 01/05/00, 01/06/00, 01/07/00, 01/11/00 MMH/LAS

Sample Data

Miscellaneous Liver

Group Dose	Sample #	PFOS Verified	Concentration of PFOS ng/g or % Rec.	Mean PFOS ng/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ng/g or % Rec.	Mean PFHS ng/g	RSD Std. Dev. MS/MSD RPD
Method Blk Unfiltered water	MSU12129-HDOBlk-unfiltered-5-1	NA	NA			NA		
	MSU12129-HDOBlk-unfiltered-5-2	NA	NA			NA		
	MSU12129-HDOBlk-unfiltered-5-3	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-HDOBlk-unfiltered-5-4	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-HDOBlk-unfiltered-5-5	NA	E			E		
	MSU12129-HDOBlk-unfiltered-5-6	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	MSU12129-HDOBlk-unfiltered-5-7	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	MSU12129-HDOBlk-unfiltered-5-8	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
Method Blk Filtered water	MSU12129-HDOBlk-filtered-5-1	NA	NA			NA		
	MSU12129-HDOBlk-filtered-5-2	NA	NA			NA		
	MSU12129-HDOBlk-filtered-5-3	NA	E			E		
	MSU12129-HDOBlk-filtered-5-4	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-HDOBlk-filtered-5-5	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-HDOBlk-filtered-5-6	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	MSU12129-HDOBlk-filtered-5-7	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	MSU12129-HDOBlk-filtered-5-8	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
Matrix Blk Fish Liver	FSH12129-LvrBlk-5-1	NA	0.0305			<LOQ (0.0171)		
	FSH12129-LvrBlk-5-2	NA	0.0331		32.1	<LOQ (0.0171)		NA
	FSH12129-LvrBlk-5-3	NA	0.0170	0.0269	0.00862	<LOQ (0.0171)	<LOQ	NA
Matrix Blk Rabbit Liver	RBL12129-LvrBlk-5-1	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	RBL12129-LvrBlk-5-2	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	RBL12129-LvrBlk-5-3	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	RBL12129-LvrBlk-5-4	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	RBL12129-LvrBlk-5-5*	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	RBL12129-LvrBlk-5-6*	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
QC 250 ng/g	Mink Liver, D530, MS-5-1-1	NA	143%			61%		
	Mink Liver, D530, MSD-5-1-2*	NA	538%	342%	115%	50%	55%	21%
	CSL-1999030-03-01-MS-5-1-2*	NA	258%			116%		
	CSL-1999030-03-01-MSD-5-1-2*	NA	202%	230%	24%	112%	114%	4%
	LWL-1999029-12-MS-5-1-2*	NA	351%			57%		
	LWL-1999029-12-MSD-5-1-2*	NA	317%	334%	10%	68%	63%	18%
	BTL-1999040-01-MS-5-1-2*	NA	138%			81%		
	BTL-1999040-01-MSD-5-1-2*	NA	132%	135%	5%	84%	83%	4%
	TNL-TUS4-MS-5-1-2	NA	68%			55%		
	TNL-TUS4-MSD-5-1-2	NA	E	68%	NA	E	55%	NA
	FSL-F295-MS-5-1	NA	111%			97%		
	FSL-F295-MSD-5-1	NA	106%	108%	5%	84%	90%	15%
	FSL-980390LB-MS-5-1	NA	77%			70%		
	FSL-980390LB-MSD-5-2*	NA	238%	158%	102%	76%	73%	9%
	GFL-R2CKDM-D1-MS-5-1-2	NA	101%			82%		
	GFL-R2CKDM-D1-MSD-5-1-	NA	112%	107%	11%	89%	86%	9%
	TTL-LCFTR99503C-MS-5-1-2*	NA	92%			66%		
	TTL-LCFTR99503C-MSD-5-1-2	NA	102%	97%	10%	72%	69%	8%
	MTL-10Vmnkrw98-MS-5-1-2	NA	108%			74%		
	MTL-10Vmnkrw98-MSD-5-1-2	NA	118%	113%	8%	84%	79%	13%
Liver Northern Fur Seal	P283	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	P283**	X	0.122			<LOQ (0.00683)		
	P295	NA	0.0547			<LOQ (0.00683)		
	97 CU 02	NA	NE			NE		
	98 CU KB 02	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	98 CU KB 03	NA	<LOQ (0.0347)			0.0587		
	98 CU KB 07	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	98 CU KB 09	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	98 CU KB 10	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	98 CU KB 11	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	98 CU KB 12	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	98 CU KB 13	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	98 CU KB 14	NA	<LOQ (0.0347)		NA	<LOQ (0.00683)		NA
	98 CU KB 15	NA	<LOQ (0.0347)	<LOQ - 2 outliers	NA	0.0858	<LOQ- 2 outliers	NA
Liver Polar Bear	970012	X	0.456			<LOQ (0.00683)		
	970201	X	0.301			<LOQ (0.00683)		
	980341	X	0.678			<LOQ (0.00683)		
	692-PLBR-0013	X	0.471			<LOQ (0.00683)		
	980127LB	X	0.539			<LOQ (0.00683)		
	980387LB	X	0.221			<LOQ (0.00683)		
	980390LB**	X	0.209			<LOQ (0.00683)		
	980343LA	X	0.438			<LOQ (0.00683)		
	980565LB	X	0.328			<LOQ (0.00683)		
	990117LB	X	0.175			<LOQ (0.00683)		
	980592LA	X	0.313			<LOQ (0.00683)		
	980594LB	X	0.356			<LOQ (0.00683)		
	980598LB	X	0.436			<LOQ (0.00683)		
	990600LB	X	0.224			<LOQ (0.00683)		
	990610LB	X	0.295			<LOQ (0.00683)		
	990632LC	X	0.235		38.6	<LOQ (0.00683)		NA
	990654LB	X	0.282	0.350	0.135	<LOQ (0.00683)	<LOQ	NA
Liver Mink	D0530	X	0.974			<LOQ (0.0171)		
	D0566*	X	2.68			<LOQ (0.0171)		
	D0590	X	2.38			<LOQ (0.0171)		
	D0618	X	0.974			<LOQ (0.0171)		
	D0630*	X	2.75			<LOQ (0.0171)		
	D0684*	X	3.50			<LOQ (0.0171)		
	D1000	X	3.42			<LOQ (0.0171)		
	D1024	X	2.13			<LOQ (0.0171)		
	D1050	X	3.28			<LOQ (0.0171)		
	D1092	X	3.22			<LOQ (0.0171)		
	D1110*	X	3.35			<LOQ (0.0171)		
	D1134	X	3.67			<LOQ (0.0171)		
	D1150X*	X	1.96			<LOQ (0.0171)		
	D1194	X	1.88			<LOQ (0.0171)		
	D1198	X	2.82			<LOQ (0.0171)		
	D1244	X	3.42			<LOQ (0.0171)		
	D1248	X	1.31		35.0	<LOQ (0.0171)		NA
	D1660*	X	3.68	2.63	0.919	<LOQ (0.0171)	<LOQ	NA

* High (>50%) surrogate deviations confirmed.
** positive analyte confirmation was not achieved, used 499 --> 99 transition.
Date Entered/Analyst: 12/22/99, 12/28/99, 12/29/99, 13/30/99, 01/12/00, 01/17/00, 01/18/00, 01/19/00, 01/20/00 LAC
Date Verified/Analyst: a

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation
X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
Method/Revision:	ETS-8-6.0 & ETS-8-7.0
Analytical Equipment System Number:	Sciex 020199, Amelia 062498
Instrument Software/Version:	Masslynx 3.3
Date of Extraction/Analyst:	12/13/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	12/17/99, 12/20/99, 12/28/99, 12/29/99, 01/03/00, 01/05/00, 01/06/00, 01/08/00 IAS/MMH
Date of Data Reduction/Analyst:	12/20/99, 12/21/99, 12/22/99, 12/30/99, 01/03/00, 01/05/00, 01/06/00, 01/07/00, 01/11/00 MMH/IAS

Sample Data

Miscellaneous Liver

Group Desc	Sample #	Concentration of PFOAA ng/g or % Rec.	Mean PFOAA ng/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ng/g or % Rec.	Mean PFOSA ng/g	RSD Std. Dev. MS/MSD RPD
Method Blk Unfiltered water	MSU12129-H2OBlk-unfiltered-5-1	NA			NA		
	MSU12129-H2OBlk-unfiltered-5-2	NA			NA		
	MSU12129-H2OBlk-unfiltered-5-3	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-5	E			E		
	MSU12129-H2OBlk-unfiltered-5-6	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-7	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	MSU12129-H2OBlk-unfiltered-5-8	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Method Blk Filtered water	MSU12129-H2OBlk-filtered-5-1	NA			NA		
	MSU12129-H2OBlk-filtered-5-2	NA			NA		
	MSU12129-H2OBlk-filtered-5-3	E			E		
	MSU12129-H2OBlk-filtered-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-5	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-6	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-7	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	MSU12129-H2OBlk-filtered-5-8	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Matrix Blk Fish Liver	FSH12129-LvrBlk-5-1	<LOQ (0.0719)			<LOQ (0.0188)		
	FSH12129-LvrBlk-5-2	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	FSH12129-LvrBlk-5-3	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Matrix Blk Rabbit Liver	RBL12129-LvrBlk-5-1	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-2	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-3	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-5*	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	RBL12129-LvrBlk-5-6*	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
QC 250 ng/g	Mink Liver, D530, MS-5-1-1	86%			43%		
	Mink Liver, D530, MSD-5-1-2*	84%	85%	3%	81%	62%	60%
	CSL-1999030-03-01-MS-5-1-2*	131%			53%		
	CSL-1999030-03-01-MSD-5-1-2*	111%	121%	17%	41%	47%	26%
	LWL-1999029-12-MS-5-1-2*	133%			67%		
	LWL-1999029-12-MSD-5-1-2*	145%	139%	9%	73%	70%	8%
	BTL-1999040-01-MS-5-1-2*	127%			83%		
	BTL-1999040-01-MSD-5-1-2*	120%	124%	6%	81%	82%	3%
	TNL-TU54-MS-5-1-2	84%			59%		
	TNL-TU54-MSD-5-1-2	E	84%	NA	E	59%	NA
	FSL-P295-MS-5-1	72%			76%		
	FSL-P295-MSD-5-1	80%	76%	10%	71%	73%	8%
	PBL-980390LB-MS-5-1	85%			60%		
	PBL-980390LB-MSD-5-2*	129%	107%	41%	54%	57%	11%
	GFL-KZCKDM-D1-MS-5-1-2	82%			71%		
	GFL-KZCKDM-D1-MSD-5-1-	103%	92%	23%	82%	77%	14%
	TTL-LCPTR99103C-MS-5-1-2*	73%			67%		
	TTL-LCPTR99103C-MSD-5-1-2	64%	69%	13%	59%	63%	12%
	MTL-10Vmuskrat98-MS-5-1-2	100%			63%		
	MTL-10Vmuskrat98-MSD-5-1-2	75%	88%	29%	63%	63%	1%
Liver Northern Fur Seal	P283	<LOQ (0.0719)			<LOQ (0.0188)		
	P285**	<LOQ (0.0719)			<LOQ (0.0188)		
	P295	<LOQ (0.0719)			<LOQ (0.0188)		
	97 CU 02	NE			NE		
	98 CU KB 02	<LOQ (0.0719)			<LOQ (0.0188)		
	98 CU KB 03	<LOQ (0.0719)			<LOQ (0.0188)		
	98 CU KB 07	<LOQ (0.0719)			<LOQ (0.0188)		
	98 CU KB 09	<LOQ (0.0719)			<LOQ (0.0188)		
	98 CU KB 10	<LOQ (0.0719)			<LOQ (0.0188)		
	98 CU KB 11	<LOQ (0.0719)			<LOQ (0.0188)		
	98 CU KB 12	<LOQ (0.0719)			<LOQ (0.0188)		
	98 CU KB 13	<LOQ (0.0719)			<LOQ (0.0188)		
	98 CU KB 14	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	98 CU KB 15	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Liver Polar Bear	970012	<LOQ (0.0719)			<LOQ (0.0188)		
	970201	<LOQ (0.0719)			<LOQ (0.0188)		
	980341	<LOQ (0.0719)			<LOQ (0.0188)		
	692-PLBR-0033	<LOQ (0.0719)			<LOQ (0.0188)		
	980127LB	<LOQ (0.0719)			<LOQ (0.0188)		
	980387LB	<LOQ (0.0719)			<LOQ (0.0188)		
	980390LB**	<LOQ (0.0719)			<LOQ (0.0188)		
	980563LA	<LOQ (0.0719)			<LOQ (0.0188)		
	980565LB	<LOQ (0.0719)			<LOQ (0.0188)		
	990112LB	<LOQ (0.0719)			<LOQ (0.0188)		
	990597LA	<LOQ (0.0719)			<LOQ (0.0188)		
	990594LB	<LOQ (0.0719)			<LOQ (0.0188)		
	990598LB	<LOQ (0.0719)			<LOQ (0.0188)		
	990600LB	<LOQ (0.0719)			<LOQ (0.0188)		
	990610LB	<LOQ (0.0719)			<LOQ (0.0188)		
	990652LC	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	990658LB	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Liver Mink	D0530	<LOQ (0.0719)			0.0334		
	D0566*	<LOQ (0.0719)			0.0266		
	D0590	<LOQ (0.0719)			0.0209		
	D0618	<LOQ (0.0719)			0.0581		
	D0630*	<LOQ (0.0719)			0.0703		
	D0684*	<LOQ (0.0719)			0.0612		
	D1000	<LOQ (0.0719)			0.0294		
	D1024	<LOQ (0.0719)			<LOQ (0.0188)		
	D1050	<LOQ (0.0719)			<LOQ (0.0188)		
	D1092	<LOQ (0.0719)			<LOQ (0.0188)		
	D1110*	<LOQ (0.0719)			0.0367		
	D1134	<LOQ (0.0719)			<LOQ (0.0188)		
	D1150X*	<LOQ (0.0719)			0.0340		
	D1194	<LOQ (0.0719)			0.0833		
	D1198	<LOQ (0.0719)			0.0242		
	D1244	<LOQ (0.0719)			<LOQ (0.0188)		
	D1248	<LOQ (0.0719)		NA	0.0345		0.5
	D1660*	<LOQ (0.0719)	<LOQ	NA	0.0621	0.0442 - 5 outliers	0.0201

* High (>50%) surrogate deviations confirmed.
** positive analyte confirmation was not achieved, used 499 --> 99 transition.
Date Entered/Analyst: 12/22/99, 12/28/99, 12/29/99, 12/30/99, 01/12/00, 01/17/00, 01/18/00, 01/19/00, 01/20/00 LAC
Date Verified/Analyst: 0

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation
X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
PFOAA = Perfluorooctanoate

FACT-GEN-030
MSU Environmental Samples

Study: GEN030 MSU Environmental Samples
Product Number(Test Substance): Various
Matrix: Various
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: 12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst: 12/17/99, 12/20/99, 12/28/99, 12/29/99, 01/03/00, 01/06/00, 02/01/00 IAS/MMH/MEE
Date of Data Reduction/Analyst: 12/20/99, 12/21/99, 12/22/99, 12/30/99, 01/03/00, 01/05/00, 01/07/00, 02/03/00 MMH/IAS/MEE

Sample Data

Miscellaneous Liver

Group Dose	Sample #	PFOS Verified	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/g or % Rec.	Mean PFHS ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk Unfiltered water	MSU12129-H2OBlk-unfiltered-5-1	NA	NA			NA		
	MSU12129-H2OBlk-unfiltered-5-2	NA	NA			NA		
	MSU12129-H2OBlk-unfiltered-5-3	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-H2OBlk-unfiltered-5-4	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-H2OBlk-unfiltered-5-5	NA	E			E		
	MSU12129-H2OBlk-unfiltered-5-6	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	MSU12129-H2OBlk-unfiltered-5-7	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	MSU12129-H2OBlk-unfiltered-5-8	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
Method Blk Filtered water	MSU12129-H2OBlk-filtered-5-1	NA	NA			NA		
	MSU12129-H2OBlk-filtered-5-2	NA	NA			NA		
	MSU12129-H2OBlk-filtered-5-3	NA	E			E		
	MSU12129-H2OBlk-filtered-5-4	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-H2OBlk-filtered-5-5	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	MSU12129-H2OBlk-filtered-5-6	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	MSU12129-H2OBlk-filtered-5-7	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	MSU12129-H2OBlk-filtered-5-8	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
Matrix Blk Fish Liver	FSH12129-LvrBlk-5-1	NA	0.0305			<LOQ (0.0171)		
	FSH12129-LvrBlk-5-2	NA	0.0331		32.1	<LOQ (0.0171)		NA
	FSH12129-LvrBlk-5-3	NA	0.0170	0.0269	0.00862	<LOQ (0.0171)	<LOQ	NA
Matrix Blk Rabbit Liver	RBL12129-LvrBlk-5-1	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	RBL12129-LvrBlk-5-2	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	RBL12129-LvrBlk-5-3	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	RBL12129-LvrBlk-5-4	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	RBL12129-LvrBlk-5-5*	NA	<LOQ (0.0696)		NA	<LOQ (0.0171)		NA
	RBL12129-LvrBlk-5-6*	NA	<LOQ (0.0696)	<LOQ	NA	<LOQ (0.0171)	<LOQ	NA
QC 250 ng/g	Mink Liver, D530, MS-5-1-1	NA	145%			61%		
	Mink Liver, D530, MSD-5-1-2*	NA	538%	342%	115%	50%	55%	21%
	CSL-1999030-03-01-MS-5-1-2*	NA	196%			116%		
	CSL-1999030-03-01-MSD-5-1-2*	NA	140%	168%	33%	112%	114%	4%
	LWL-1999029-12-MS-5-1-2*	NA	321%			57%		
	LWL-1999029-12-MSD-5-1-2*	NA	287%	304%	11%	68%	63%	8%
	BTL-1999040-01-MS-5-1-2*	NA	138%			81%		
	BTL-1999040-01-MSD-5-1-2*	NA	132%	135%	5%	84%	83%	4%
	TNL-TU54-MS-5-1-2	NA	68%			55%		
	TNL-TU54-MSD-5-1-2	NA	E	68%	NA	E	55%	NA
	FSL-P295-MS-5-1	NA	91%			97%		
	FSL-P295-MSD-5-1	NA	86%	88%	6%	84%	90%	15%
	PBL-980390LB-MS-5-1	NA	18%			70%		
	PBL-980390LB-MSD-5-2*	NA	179%	99%	163%	76%	73%	9%
	GFL-KZCKDM-D1-MS-5-1-2	NA	93%			82%		
	GFL-KZCKDM-D1-MSD-5-1-	NA	105%	99%	12%	89%	86%	9%
	TTL-LCPTR99503C-MS-5-1-2*	NA	90%			66%		
	TTL-LCPTR99503C-MSD-5-1-2	NA	100%	95%	11%	72%	69%	8%
	MTL-10Vancleave98-MS-5-1-2	NA	79%			74%		
	MTL-10Vancleave98-MSD-5-1-2	NA	89%	84%	11%	84%	79%	13%
Liver Map Turtle	F, #10, Vancleave 98	NA	0.0801			<LOQ (0.00683)		
	F, #09, Vancleave 98	NA	0.0514			<LOQ (0.00683)		
	F, #02, Leeksville 98	NA	0.0739			<LOQ (0.00683)		
	F, #06, Leeksville 99	NA	0.0394			<LOQ (0.00683)		
	M, (-1)	NA	0.703			<LOQ (0.00683)		
	F, (89, 8912)	NA	0.179			<LOQ (0.00683)		
Liver Terrapin	LCPTR 9503C	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	LCPTR 9504C	NA	<LOQ (0.0347)		0.730	<LOQ (0.00683)		NA
	LCPTR 9505C	NA	<LOQ (0.0347)	0.188 - 3 outliers	0.257	<LOQ (0.00683)	<LOQ	NA
Liver Tuna	TU25*	X	<LOQ (0.0696)			<LOQ (0.0171)		
	TU34*	X	<LOQ (0.0696)			<LOQ (0.0171)		
	TU41*	X	<LOQ (0.0696)			<LOQ (0.0171)		
	TU48*	X	<LOQ (0.0696)			<LOQ (0.0171)		
	TU49	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	TU54*	X	<LOQ (0.0696)			<LOQ (0.0171)		
	TU58	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	TU63*	X	<LOQ (0.0696)			<LOQ (0.0171)		
	TU66	X	0.00698			<LOQ (0.0171)		
	TU84	NA	<LOQ (0.0696)			<LOQ (0.0171)		
	TU88	X	<LOQ (0.00696)		NA	<LOQ (0.0171)		NA
	TU90	NA	<LOQ (0.0696)	<LOQ - 1 outlier	NA	<LOQ (0.0171)	<LOQ	NA
Liver Green Frog	KZCKDM-D1	NA	<LOQ (0.0347)			<LOQ (0.00683)		
	KZCKDM-D2	X	0.285			<LOQ (0.00683)		
	Pool of 4	NA	<LOQ (0.0347)		NA	<LOQ (0.00683)		NA
	SJ0001	X	<LOQ (0.0347)	<LOQ - 1 outlier	NA	<LOQ (0.00683)	<LOQ	NA

* High (>50%) surrogate deviations

NE = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation
X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Date Entered/Analyst: 12/22/99, 12/28/99, 12/29/99, 12/30/99, 01/12/00, 01/17/00, 01/18/00, 02/04/00 LAC
Date Verified/Analyst:

FACT-GEN-030
MSU Environmental Samples

Study:	GEN030 MSU Environmental Samples
Product Number(Test Substance):	Various
Matrix:	Various
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:	12/12/99 SAL/KK/SRP/CSH
Date of Analysis/Analyst:	12/17/99, 12/20/99, 12/28/99, 12/29/99, 01/03/00, 01/06/00, 02/01/00 IAS/MMH/MEE
Date of Data Reduction/Analyst:	12/20/99, 12/21/99, 12/22/99, 12/30/99, 01/03/00, 01/05/00, 01/07/00, 02/03/00 MMH/IAS/MEE

Sample Data

Miscellaneous Liver

Group Dose	Sample #	Concentration of POAA ng/g or % Rec.	Mean POAA ng/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFOSA ng/g or % Rec.	Mean PFOSA ng/g	RSD Std. Dev. MS/MSD RPD
Method Blk	MSU12129-H2OBlk-unfiltered-5-1	NA			NA		
Unfiltered water	MSU12129-H2OBlk-unfiltered-5-2	NA			NA		
	MSU12129-H2OBlk-unfiltered-5-3	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-5	E			E		
	MSU12129-H2OBlk-unfiltered-5-6	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-unfiltered-5-7	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	MSU12129-H2OBlk-unfiltered-5-8	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Method Blk	MSU12129-H2OBlk-filtered-5-1	NA			NA		
Filtered water	MSU12129-H2OBlk-filtered-5-2	NA			NA		
	MSU12129-H2OBlk-filtered-5-3	E			E		
	MSU12129-H2OBlk-filtered-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-5	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-6	<LOQ (0.0719)			<LOQ (0.0188)		
	MSU12129-H2OBlk-filtered-5-7	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	MSU12129-H2OBlk-filtered-5-8	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Matrix Blk	FSH12129-LvrBlk-5-1	<LOQ (0.0719)			<LOQ (0.0188)		
Fish Liver	FSH12129-LvrBlk-5-2	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	FSH12129-LvrBlk-5-3	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Matrix Blk	RBL12129-LvrBlk-5-1	<LOQ (0.0719)			<LOQ (0.0188)		
Rabbit Liver	RBL12129-LvrBlk-5-2	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-3	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-4	<LOQ (0.0719)			<LOQ (0.0188)		
	RBL12129-LvrBlk-5-5*	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	RBL12129-LvrBlk-5-6*	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
QC	Mink Liver, D530, MS-5-1-1	86%			43%		
250 ng/g	Mink Liver, D530, MSD-5-1-2*	84%	85%	3%	81%	62%	60%
	CSL-1999030-03-01-MS-5-1-2*	131%			64%		
	CSL-1999030-03-01-MSD-5-1-2*	111%	121%	17%	52%	58%	21%
	LWL-1999029-12-MS-5-1-2*	133%			77%		
	LWL-1999029-12-MSD-5-1-2*	145%	139%	9%	83%	80%	7%
	BTL-1999040-01-MS-5-1-2*	127%			94%		
	BTL-1999040-01-MSD-5-1-2*	120%	124%	6%	92%	93%	3%
	TNL-TU54-MS-5-1-2	84%			71%		
	TNL-TU54-MSD-5-1-2	E	84%	NA	E	71%	NA
	FSL-P295-MS-5-1	72%			87%		
	FSL-P295-MSD-5-1	80%	76%	10%	82%	85%	7%
	PBL-980390LB-MS-5-1	85%			71%		
	PBL-980390LB-MSD-5-2*	129%	107%	41%	65%	68%	9%
	GFL-KZCKDM-D1-MS-5-1-2	82%			82%		
	GFL-KZCKDM-D1-MSD-5-1-	103%	92%	23%	93%	88%	12%
	TTL-LCPTR99503C-MS-5-1-2*	73%			78%		
	TTL-LCPTR99503C-MSD-5-1-2	64%	69%	13%	71%	74%	10%
	MTL-10Vancleave98-MS-5-1-2	100%			74%		
	MTL-10Vancleave98-MSD-5-1-2	75%	88%	29%	74%	74%	0%
Liver	F, #10, Vancleave 98	<LOQ (0.0719)			<LOQ (0.0188)		
Map Turtle	F, #09, Vancleave 98	<LOQ (0.0719)			<LOQ (0.0188)		
	F, #02, Leeksville 98	<LOQ (0.0719)			<LOQ (0.0188)		
	F, #06, Leeksville 99	<LOQ (0.0719)			<LOQ (0.0188)		
	M, (-1)	<LOQ (0.0719)			<LOQ (0.0188)		
	F, (89, 8912)	<LOQ (0.0719)			<LOQ (0.0188)		
Liver	LCPTR 9503C	<LOQ (0.0719)			<LOQ (0.0188)		
Terrapin	LCPTR 9504C	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	LCPTR 9505C	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Liver	TU25*	<LOQ (0.0719)			<LOQ (0.0188)		
Tuna	TU34*	<LOQ (0.0719)			<LOQ (0.0188)		
	TU41*	<LOQ (0.0719)			<LOQ (0.0188)		
	TU48*	<LOQ (0.0719)			<LOQ (0.0188)		
	TU49	<LOQ (0.0719)			<LOQ (0.0188)		
	TU54*	<LOQ (0.0719)			<LOQ (0.0188)		
	TU58	<LOQ (0.0719)			<LOQ (0.0188)		
	TU63*	<LOQ (0.0719)			<LOQ (0.0188)		
	TU66	<LOQ (0.0719)			<LOQ (0.0188)		
	TU84	<LOQ (0.0719)			<LOQ (0.0188)		
	TU88	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	TU90	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA
Liver	KZCKDM-D1	<LOQ (0.0719)			<LOQ (0.0188)		
Green Frog	KZCKDM-D2	<LOQ (0.0719)			<LOQ (0.0188)		
	Pool of 4	<LOQ (0.0719)		NA	<LOQ (0.0188)		NA
	SJ0001	<LOQ (0.0719)	<LOQ	NA	<LOQ (0.0188)	<LOQ	NA

* High (>50%) surrogate deviations

NB = Not Extracted
E = Lost during extraction
NA = Not Applicable
LOQ = Limit of Quantitation
X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctane sulfonamide
PFHS = Perfluorohexanesulfonate
POAA = Perfluorooctanoate

Date Entered/Analyst: 12/22/99, 12/28/99, 12/29/99, 12/30/99, 01/12/00, 01/17/00, 01/18/00, 02/04/00 LAC

Date Verified/Analyst:

Study:	GEN033, MSU - Liver Samples		
Product Number(Test Substance):	NA		
Matrix:	Various livers - Unextracted Curves	Filename:	See Attachments
Method/Revision:	BTS-8-6.0 and BTS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Davey 070799, Amelia 062498	Slope:	See Attachments
Instrument Software/Version:	Masslynx 3.3	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	03/14/00 SAL/CSH/KKK		
Date of Analysis/Analyst:	03/16/00, 03/17/00, 3/19/00, 03/20/00, 03/21/00, 03/29/00, 04/07/00 IAS/MMH		
Date of Data Reduction/Analyst:	03/20/00, 03/22/00, 03/23/00, 03/24/00, 04/04/00, 04/11/00 IAS/MMH		

Sample Data

Various Livers

Group Dose	Sample #	Concentration of PFOS ug/g or % Rec	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of POAA ug/g or % Rec	Mean POAA ug/g	RSD Std. Dev. MS/MSD RPD
Mink Liver	C23 (45)	0.801			< LOQ (0.0359 ug/g)		
	C26 (44)	0.443			< LOQ (0.0359 ug/g)		
	C27 (49)	0.145			< LOQ (0.0359 ug/g)		
	C33 (46)	0.435			< LOQ (0.0359 ug/g)		
	C35 (47)	0.355			< LOQ (0.0359 ug/g)		
	C37 (48)	0.833			< LOQ (0.0359 ug/g)		
	C42 (43)	0.420			< LOQ (0.0359 ug/g)		
	C44 (54)	0.237			< LOQ (0.0359 ug/g)		
	D10 (50)	1.67			< LOQ (0.0359 ug/g)		
	F15 (55)	0.548			< LOQ (0.0359 ug/g)		
	F19 (56)	0.783			< LOQ (0.0359 ug/g)		
	F21 (57)	1.03			< LOQ (0.0359 ug/g)		
	F24 (58)	0.868			< LOQ (0.0359 ug/g)		
	P01 (60)	2.16			< LOQ (0.0359 ug/g)		
	P03 (64)	4.80			< LOQ (0.0359 ug/g)		
	P09 (61)	0.841			< LOQ (0.0359 ug/g)		
	S11 (37)	0.902			< LOQ (0.0359 ug/g)		
	S15 (41)	1.27			< LOQ (0.0359 ug/g)		
	S18 (40)	1.99			< LOQ (0.0359 ug/g)		
	S19 (59)	2.68			< LOQ (0.0359 ug/g)		
	S25 (39)	0.509			< LOQ (0.0359 ug/g)		
	S30 (36)	0.186			< LOQ (0.0359 ug/g)		
	S35 (42)	0.0933			< LOQ (0.0359 ug/g)		
	S39 (38)	0.317			< LOQ (0.0359 ug/g)		
	T01 (51)	0.633			< LOQ (0.0359 ug/g)		
	T04 (53)	1.35			< LOQ (0.0359 ug/g)		
	T03 (52)	0.565			< LOQ (0.0359 ug/g)		
	V12 (62)	4.87			< LOQ (0.0359 ug/g)		
	V03 (65)	1.52		103	< LOQ (0.0359 ug/g)		NA
	V08 (63)	3.65	1.23	1.27	< LOQ (0.0359 ug/g)	<LOQ	NA
Baikal Seal Liver	J08 (81)	0.0127			< LOQ (0.0359 ug/g)		
	J09 (87)	0.0228			< LOQ (0.0718 ug/g)		
	J10 (86)	<LOQ (0.0347 ug/g)			< LOQ (0.0718 ug/g)		
	J12 (89)	0.0141			< LOQ (0.0359 ug/g)		
	J19 (84)	0.00931			< LOQ (0.0359 ug/g)		
	J20 (88)	0.0154			< LOQ (0.0718 ug/g)		
	J24 (82)	<LOQ (0.0347 ug/g)			< LOQ (0.0718 ug/g)		
	J27 (83)	<LOQ (0.0347 ug/g)			< LOQ (0.0718 ug/g)		
	J36 (85)	0.0146			< LOQ (0.0359 ug/g)		
	J37 (80)	<LOQ (0.0347 ug/g)			< LOQ (0.0718 ug/g)		
	R04 (69)	<LOQ (0.0347 ug/g)			< LOQ (0.0718 ug/g)		
	R13 (78)	0.0100			< LOQ (0.0359 ug/g)		
	R14 (74)	<LOQ (0.0347 ug/g)			< LOQ (0.0718 ug/g)		
	R16 (71)	0.00808			< LOQ (0.0359 ug/g)		
	R29 (72)	0.00795			< LOQ (0.0359 ug/g)		
	R42 (66)	0.0138			< LOQ (0.0359 ug/g)		
	R43 (73)	0.0156			< LOQ (0.0359 ug/g)		
	R45 (79)	0.00848			< LOQ (0.0359 ug/g)		
	R46 (77)	0.00778			< LOQ (0.0359 ug/g)		
	R47 (70)	<LOQ (0.0347 ug/g)			< LOQ (0.0718 ug/g)		
	R54 (67)	<LOQ (0.0347 ug/g)			< LOQ (0.0718 ug/g)		
	R55 (75)	0.0133			< LOQ (0.0718 ug/g)		
	R57 (76)	0.0158		33.7	< LOQ (0.0359 ug/g)		NA
	R64 (68)	0.00786	0.0123	0.00416	< LOQ (0.0359 ug/g)	<LOQ	NA
Ganges Dolphin Liver	L04 (91)	<LOQ (0.0347 ug/g)		NA	<LOQ (0.0718 ug/g)		NA
	L05 (90)	0.0813	NA	NA	<LOQ (0.0718 ug/g)	<LOQ	NA

LOQ = Limit of Quantitation
NA = Not Applicable

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFHS = Perfluorohexane sulfonate
POAA = Perfluorooctanoate

Data Entered/Analyst: 03/28/00, 04/05/00, 04/07/00, 05/07/00 MMH/LAC
Data Verified/Analyst:

Study: GEN033, MSU - Liver Samples
Product Number(Test Substance): NA
Matrix: Various livers - Unextracted Curves
Method/Revision: BTS-8-6.0 and BTS-8-7.0
Analytical Equipment System Number: Davey 070799, Amelia 062498
Instrument Software/Version: Masslynx 3.3
Date of Extraction/Analyst: 03/14/00 SAL/CSH/KKK
Date of Analysis/Analyst: 03/16/00, 03/17/00, 3/19/00, 03/20/00, 03/21/00, 03/29/00, 04/07/00 IAS/MMH
Date of Data Reduction/Analyst: 03/20/00, 03/22/00, 03/23/00, 03/24/00, 04/04/00, 04/11/00 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

Various Livers

Group Dose	Sample #	Concentration of PFOSA ug/g or % Rec	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/g or % Rec	Mean PFHS ug/g	RSD Std. Dev. MS/MSD RPD
Mink Liver	C23 (45)	0.0383			< LOQ (0.00683 ug/g)		
	C26 (44)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	C27 (49)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	C33 (46)	< LOQ (0.0376 ug/g)			0.00833		
	C35 (47)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	C37 (48)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	C42 (43)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	C44 (54)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	D10 (50)	0.0828			< LOQ (0.00683 ug/g)		
	F15 (55)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	F19 (56)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	F21 (57)	0.0579			< LOQ (0.00683 ug/g)		
	F24 (58)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	P01 (60)	0.551			0.0315		
	P03 (64)	0.590			0.0852		
	P09 (61)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	S11 (37)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	S15 (41)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	S18 (40)	0.0414			< LOQ (0.00683 ug/g)		
	S19 (59)	0.132			0.0102		
	S25 (39)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	S30 (36)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	S35 (42)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	S39 (38)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	T01 (51)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	T04 (53)	< LOQ (0.0376 ug/g)			0.0104		
	T03 (52)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	V12 (62)	0.345			< LOQ (0.00683 ug/g)		
	V03 (65)	0.0594		NA	< LOQ (0.00683 ug/g)		NA
	V08 (63)	0.0586	<LOQ - 10 Outlier	NA	< LOQ (0.00683 ug/g)	<LOQ - 5 Outlier	NA
Baikal Seal Liver	J06 (81)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J09 (87)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J10 (86)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J12 (89)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J19 (84)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J20 (88)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J24 (82)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J27 (83)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J36 (85)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	J37 (80)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R04 (69)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R13 (78)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R14 (74)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R16 (71)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R29 (72)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R42 (66)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R43 (73)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R45 (79)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R46 (77)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R47 (70)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R54 (67)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R55 (75)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	R57 (76)	< LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	R64 (68)	< LOQ (0.0376 ug/g)	<LOQ	NA	< LOQ (0.00683 ug/g)	<LOQ	NA
Ganges Dolphin Liver	L04 (91)	< LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	L05 (90)	< LOQ (0.0376 ug/g)	<LOQ	NA	< LOQ (0.00683 ug/g)	<LOQ	NA

LOQ = Limit of Quantitation
NA = Not Applicable

PFOS = Perfluorooctanesulfonate
PFOSA = Perfluorooctanesulfonamide
PFHS = Perfluorohexane sulfonate
POAA = Perfluorooctanoate

Date Entered/Analyst: 03/28/00, 04/05/00, 04/07/00, 05/07/00 MMH/LAC
Date Verified/Analyst:

Study: GEN033, MSU - Liver Samples
Product Number(Test Substance): NA
Matrix: Various livers - unextracted curves
Method/Revision: ETS-8-6.0 and ETS-8-7.0
Analytical Equipment System Number: Davey 070799, Ansilla 062498
Instrument Software/Version: Masslynx 3.3
Date of Extraction/Analyst: 03/14/00 SAL/CSH/KKK
Date of Analysis/Analyst: 03/16/00, 03/17/00, 3/19/00, 03/20/00, 03/21/00, 03/29/00, 04/07/00 IAS/MMH
Date of Data Reduction/Analyst: 03/20/00, 03/22/00, 03/23/00, 03/24/00, 04/04/00, 04/11/00 IAS/MMH

Filename: See Attachment
R-Squared Value: See Attachment
Slope: See Attachment
Y-Intercept: See Attachment

Sample Data

Various Livers

Group Dose	Sample #	Concentration of PFOS ng/g or % Rec	Mean PFOS ng/g	RSD Std. Dev. MS/MSD RPD	Concentration of POAA ng/g or % Rec	Mean POAA ng/g	RSD Std. Dev. MS/MSD RPD
Cormorant Liver Female, Adult	4F (1),F,A	0.0432			0.0414		
	29F (5),F,A**	0.0565			0.102		
	30F (6),F,A	0.0485			0.0506		
	32F (7),F,A	0.0977			0.0897		
	34F (9),F,A**	0.0355		61.9	0.0297		56.9
	42F (11),F,A	0.150	0.0715	0.0442	0.143	0.0761	0.0432
Cormorant Liver Female, Juvenile	7F (2),F,J	0.0913			0.0841		
	9F (3),F,J	0.468		113	0.444		115
	36F (10),F,J	0.0499	0.203	0.230	0.0467	0.192	0.219
Cormorant Liver Male, Juvenile	22F (4),M,J	0.0433			0.0454		
	33F (8),M,J	0.0516		17.2	0.0303		25.7
	44F (12),M,J	0.0337	0.0362	0.00623	0.0293	0.0350	0.00899
Bottlenose Dolphin Liver	TtS/91 (13)	0.181			<LOQ (0.0359 ng/g)		
	TtS/91 (17)	0.296			<LOQ (0.0718 ng/g)		
	TtS1 (14)	0.169			<LOQ (0.0718 ng/g)		
	TtA11 (16)	0.425		44.5	<LOQ (0.0718 ng/g)		NA
	TtKT2 (15)	<LOQ (0.09696 ng/g)	0.268 - One Outlier	0.119	<LOQ (0.0718 ng/g)	<LOQ	NA
Striped Dolphin Liver	SCP02 (10)	0.161			<LOQ (0.0718 ng/g)		
	SCV1 (19)	0.0891			<LOQ (0.0718 ng/g)		
	SCP03 (20)	0.0944		40.2	<LOQ (0.0718 ng/g)		NA
	SCP04 (21)	0.0647	0.102	0.0410	<LOQ (0.0718 ng/g)	<LOQ	NA
Weddell Seal Liver	W31 (22)	<LOQ (0.0347 ng/g)	<LOQ	NA	<LOQ (0.0718 ng/g)	<LOQ	NA
Swordfish Liver	S23 (23)	< LOQ (0.09696 ng/g)			<LOQ (0.0359 ng/g)		
	S24 (24)	< LOQ (0.09696 ng/g)			<LOQ (0.0359 ng/g)		
	S25 (25)	0.00774			<LOQ (0.0359 ng/g)		
	S32 (27)	< LOQ (0.09696 ng/g)		NA	<LOQ (0.0359 ng/g)		NA
	S48 (26)	0.0135	<LOQ - Two Outliers	NA	<LOQ (0.0359 ng/g)	<LOQ	NA
Tenufish Liver	T1 (33)	< LOQ (0.0347 ng/g)			<LOQ (0.0359 ng/g)		
	T1LP156 (35)	0.0433			<LOQ (0.0718 ng/g)		
	T2 (29)	0.0874			<LOQ (0.0359 ng/g)		
	T15 (28)	0.0568			<LOQ (0.0718 ng/g)		
	T17 (31)	0.0491			<LOQ (0.0359 ng/g)		
	T20 (32)	0.0207			<LOQ (0.0359 ng/g)		
	T23 (30)	0.0560		46.2	<LOQ (0.0359 ng/g)		NA
	T25 (34)	0.0250	0.0483 - One Outlier	0.0223	<LOQ (0.0718 ng/g)	<LOQ	NA
Blacktailed Gull Liver	BHG01 (100)	0.292			<LOQ (0.0359 ng/g)		
	BHG02 (101)	0.280			<LOQ (0.0359 ng/g)		
	BHG03 (102)	0.168			<LOQ (0.0359 ng/g)		
	BHG04 (103)	0.503			<LOQ (0.0359 ng/g)		
	BHG05 (104)	0.271			<LOQ (0.0359 ng/g)		
	BTG9305 (92)	0.0881			<LOQ (0.0359 ng/g)		
	BTG9306 (99)	0.107			<LOQ (0.0359 ng/g)		
	BTG9310 (96)	0.215			<LOQ (0.0359 ng/g)		
	BTG9311 (98)	0.143			<LOQ (0.0359 ng/g)		
	BTG9312 (97)	0.0705			<LOQ (0.0359 ng/g)		
	BTG9401 (93)	0.126			<LOQ (0.0359 ng/g)		
	BTGhongdo1 (94)	0.0737			<LOQ (0.0359 ng/g)		
	BTOmundo1 (95)	0.0707			<LOQ (0.0359 ng/g)		
	HRG04 (106)	0.116		69.2	<LOQ (0.0359 ng/g)		NA
	HRG09 (105)	0.0939	0.172	0.119	<LOQ (0.0359 ng/g)	<LOQ	NA

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PFOS = Perfluorooctanesulfonate
PFOAA = Perfluorooctanoate
PFOSA = Perfluorooctanesulfonamide
PFHS = Perfluorohexane sulfonate

Date Entered/Analyst: 03/28/00, 04/05/00, 04/07/00, 05/07/00 MMH/LAC
Date Verified/Analyst:

Study:	GEN033, MSU - Liver Samples		
Product Number(Test Substance):	NA		
Matrix:	Various livers - unextracted curves	Filename:	See Below
Method/Revision:	ETS-8-6.0 and ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Davey 070799, Amelia 042498	Slope:	See Attachments
Instrument Software/Version:	Masslynx 3.3	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	03/14/00 SAL/CSH/KJK		
Date of Analysis/Analyst:	03/16/00, 03/17/00, 3/19/00, 03/20/00, 03/21/00, 03/29/00, 04/07/00 IAS/MMH		
Date of Data Reduction/Analyst:	03/20/00, 03/22/00, 03/23/00, 03/24/00, 04/04/00, 04/11/00 IAS/MMH		

Sample Data

Various Livers

Group Dose	Sample #	Concentration of PFOSA ug/g or % Rec	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/g or % Rec	Mean PFHS ug/g	RSD Std. Dev. MS/MSD RPD
Cemernat Liver Female, Adult	6F (1),F,A	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	29F (5),F,A**	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	30F (6),F,A	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	32F (7),F,A	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	34F (9),F,A**	<LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	42F (11),F,A	<LOQ (0.0376 ug/g)	<LOQ	NA	< LOQ (0.00683 ug/g)	<LOQ	NA
Cemernat Liver Female, Juvenile	7F (2),F,J	0.0888			< LOQ (0.00683 ug/g)		
	9F (3),F,J	<LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	36F (10),F,J	<LOQ (0.0376 ug/g)	<LOQ - One Outlier	NA	< LOQ (0.00683 ug/g)	<LOQ	NA
Cemernat Liver Male, Juvenile	22F (4),M,J	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	33F (8),M,J	<LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	44F (12),M,J	<LOQ (0.0376 ug/g)	<LOQ	NA	< LOQ (0.00683 ug/g)	<LOQ	NA
Bottlenose Dolphin Liver	TS/91 (13)	0.224			< LOQ (0.00683 ug/g)		
	TB/91 (17)	0.358			< LOQ (0.00683 ug/g)		
	Tt61 (14)	0.129			< LOQ (0.00683 ug/g)		
	TtA11 (16)	0.129		54.1	< LOQ (0.00683 ug/g)		NA
	TtXT2 (15)	0.115	0.191	0.103	< LOQ (0.00683 ug/g)	<LOQ	NA
Striped Dolphin Liver	SCP02 (18)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	SCV1 (19)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	SCP03 (20)	<LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	SCP04 (21)	<LOQ (0.0376 ug/g)	<LOQ - One Outlier	NA	0.0270	<LOQ - One Outlier	NA
Weddell Seal Liver	WS1 (22)	<LOQ (0.0376 ug/g)	<LOQ	NA	< LOQ (0.00683 ug/g)	<LOQ	NA
Swordfish Liver	S23 (23)	<LOQ (0.0376 ug/g)			0.00954		
	S24 (24)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	S25 (25)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	S32 (27)	<LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	S48 (26)	<LOQ (0.0376 ug/g)	<LOQ	NA	< LOQ (0.00683 ug/g)	<LOQ - One Outlier	NA
Tunafish Liver	T1 (33)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	T1LF156 (35)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	T2 (29)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	T15 (28)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	T17 (31)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	T20 (32)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	T23 (30)	<LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	T25 (34)	<LOQ (0.0376 ug/g)	<LOQ	NA	< LOQ (0.00683 ug/g)	<LOQ	NA
Blackstolled Gull Liver	BHG01 (100)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BHG02 (101)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BHG03 (102)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BHG04 (103)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BHG05 (104)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BTG9305 (92)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BTG9306 (99)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BTG9310 (96)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BTG9311 (98)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BTG9312 (97)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BTG9401 (93)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BTGhongdo1 (94)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	BTGmendo1 (95)	<LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	HRG04 (106)	<LOQ (0.0376 ug/g)		NA	< LOQ (0.00683 ug/g)		NA
	HRG09 (105)	<LOQ (0.0376 ug/g)	<LOQ	NA	< LOQ (0.00683 ug/g)	<LOQ	NA

** NO PFOS confirmation performed.
LOQ = Limit of Quantitation
NA = Not Applicable

PFOS = Perfluorooctanesulfonate
POAA = Perfluorooctanoate
PFOSA = Perfluorooctanesulfonamide
PFHS = Perfluorohexane sulfonate

Date Entered/Analyst: 03/28/00, 04/05/00, 04/07/00, 05/07/00 MMH/LAC
Date Verified/Analyst:

Study:	GEN033, MSU - Liver Samples		
Product Number(Test Substance):	NA		
Matrix:	Various livers - unextracted curves	Filename:	See Attachments
Method/Revision:	ETS-8-6.0 and ETS-8-7.0	R-Squared Value:	See Attachments
Analytical Equipment System Number:	Davey 070799, Amelia 062498	Slope:	See Attachments
Instrument Software/Version:	Masslynx 3.3	Y-Intercept:	See Attachments
Date of Extraction/Analyst:	03/14/00 SAL/CSH/KKK		
Date of Analysis/Analyst:	03/16/00, 03/17/00, 3/19/00, 03/20/00, 03/21/00, 03/29/00, 04/07/00 LAS/MMH		
Date of Data Reduction/Analyst:	03/20/00, 03/22/00, 03/23/00, 03/24/00, 04/04/00, 04/11/00 LAS/MMH		

Sample Data

Various Livers

Group Dose	Sample #	Concentration of PFOS ug/g or % Rec	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of POAA ug/g or % Rec	Mean POAA ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	RBL03140-H2O Blk-5-1	< LOQ (0.00696 ug/g)			<LOQ (0.0359 ug/g)		
	RBL03140-H2O Blk-5-2	< LOQ (0.00696 ug/g)			<LOQ (0.0359 ug/g)		
	RBL03140-H2O Blk-5-3	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-H2O Blk-5-4	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-H2O Blk-5-5	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-H2O Blk-5-6	< LOQ (0.0347 ug/g)			<LOQ (0.00719 ug/g)		
	RBL03140-H2O Blk-5-7	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-H2O Blk-5-8	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-H2O Blk-5-9	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-H2O Blk-5-10	< LOQ (0.0347 ug/g)			<LOQ (0.00719 ug/g)		
	RBL03140-H2O Blk-5-11	< LOQ (0.0347 ug/g)			<LOQ (0.0359 ug/g)		
	RBL03140-H2O Blk-5-12	< LOQ (0.0347 ug/g)		NA	<LOQ (0.0359 ug/g)		NA
	RBL03140-H2O Blk-5-13	< LOQ (0.0347 ug/g)	<LOQ	NA	<LOQ (0.0359 ug/g)	<LOQ	NA
Matrix Blk	RBL03140-Liver Blk-5-1	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-2	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-3	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-4	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-5	0.00883			<LOQ (0.0359 ug/g)		
	RBL03140-Liver Blk-5-6	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-7	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-8	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-9	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-10	< LOQ (0.0694 ug/g)			<LOQ (0.0718 ug/g)		
	RBL03140-Liver Blk-5-11	< LOQ (0.0347 ug/g)			<LOQ (0.0359 ug/g)		
	RBL03140-Liver Blk-5-12	< LOQ (0.0347 ug/g)		NA	<LOQ (0.0359 ug/g)		NA
	RBL03140-Liver Blk-5-13	< LOQ (0.0347 ug/g)	<LOQ - One Outlier	NA	<LOQ (0.0359 ug/g)	<LOQ	NA
QC 250 ppb	RBL03140-MS-1 ppb-5-1*	104%			112%		
	RBL03140-MSD-1 ppb-5-2*	114%	109%	9%	106%	109%	6%
	RBL03140-MS-250 ppb-5-1	120%			128%		
	RBL03140-MSD-250 ppb-5-2	133%	127%	10%	128%	128%	0%
	44F (12)-MS	98%			108%		
	44F (12)-MSD	107%	103%	5%	109%	107%	2%
	T6/91 (17)-MS	131%			59%		
	T6/91 (17)-MSD	140%	135%	6%	64%	61%	9%
	SCV1 (19)-MS	66%			58%		
	SCV1 (19)-MSD	93%	79%	35%	70%	64%	30%
	WS1 (22)-MS	81%			63%		
	WS1 (22)-MSD	73%	77%	10%	62%	62%	2%
	324 (24)-MSD	92%	NA	NA	101%	NA	NA
	T15 (28)-MS	84%			93%		
	T15 (28)-MSD	103%	94%	21%	105%	99%	12%
	T1LF156 (35)-MS	93%			92%		
	T1LF156 (35)-MSD	72%	82%	25%	80%	86%	15%
	S19 (39)-MS	326%			126%		
	S19 (39)-MSD	234%	280%	33%	154%	140%	20%
	P9 (61)-MS	231%			102%		
	P9 (61)-MSD	142%	187%	48%	112%	107%	9%
	R47 (70)-MS	68%			70%		
	R47 (70)-MSD	69%	69%	2%	68%	69%	3%
	L4 (91)-MS	69%			73%		
	L4 (91)-MSD	187%	128%	92%	165%	119%	78%
	BTG9305 (92)-MS	143%	NA	NA	153%	NA	NA
	BTGhengde1 (94)-MS	122%			137%		
	BTGhengde1 (94)-MSD	120%	121%	2%	154%	146%	13%

LOQ = Limit of Quantitation
NA = Not Applicable

PFOS = Perfluorooctanesulfonate
POAA = Perfluorooctanoate
PFOSA = Perfluorooctanesulfonamide
PFHS = Perfluorohexane sulfonate

Date Entered/Analyst: 03/23/00, 04/05/00, 04/07/00, 05/07/00 MMH/LAC
Date Verified/Analyst:

Study: GEN033, MSU - Liver Samples
Product Number(Test Substance): NA
Matrix: Various livers - uncontracted curves
Method/Revision: ETS-8-6.0 and ETS-8-7.0
Analytical Equipment System Number: Davey 070799, Amelia 062498
Instrument Software/Version: Masslynx 3.3
Date of Extraction/Analyst: 03/14/00 SAL/CSH/KKK
Date of Analysis/Analyst: 03/16/00, 03/17/00, 3/19/00, 03/20/00, 03/21/00, 03/29/00, 04/07/00 IAS/MMH
Date of Data Reduction/Analyst: 03/20/00, 03/22/00, 03/23/00, 03/24/00, 04/04/00, 04/11/00 IAS/MMH

Filename: See Below
R-Squared Value: See Attachments
Slope: See Attachments
Y-Intercept: See Attachments

Sample Data

Various Livers

Group Dose	Sample #	Concentration of PFOSA ug/g or % Rec	Mean PFOSA ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/g or % Rec	Mean PFHS ug/g	RSD Std. Dev. MS/MSD RPD
Method Blk	RBL03140-H2O Blk-5-1	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-2	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-3	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-4	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-5	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-6	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-7	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-8	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-9	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-10	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-11	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-H2O Blk-5-12	< LOQ (0.0376 ug/g)		NA	<LOQ (0.00683 ug/g)		NA
	RBL03140-H2O Blk-5-13	< LOQ (0.0376 ug/g)	<LOQ	NA	<LOQ (0.00683 ug/g)	<LOQ	NA
Matrix Blk	RBL03140-Liver Blk-5-1	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-2	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-3	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-4	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-5	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-6	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-7	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-8	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-9	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-10	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-11	< LOQ (0.0376 ug/g)			<LOQ (0.00683 ug/g)		
	RBL03140-Liver Blk-5-12	< LOQ (0.0376 ug/g)		NA	<LOQ (0.00683 ug/g)		NA
	RBL03140-Liver Blk-5-13	< LOQ (0.0376 ug/g)	<LOQ	NA	<LOQ (0.00683 ug/g)	<LOQ	NA
QC 250 ppb	RBL03140-MS-1 ppb-5-1*	66%			87%		
	RBL03140-MSD-1 ppb-5-2*	67%	67%	2%	57%	72%	42%
	RBL03140-MS-250 ppb-5-1	114%			61%		
	RBL03140-MSD-250 ppb-5-2	84%	99%	30%	48%	54%	25%
	44F (12)-MS	94%			84%		
	44F (12)-MSD	89%	92%	5%	87%	86%	3%
	Td/91 (17)-MS	148%			65%		
	Td/91 (17)-MSD	191%	168%	26%	65%	65%	1%
	SCV1 (19)-MS	53%			54%		
	SCV1 (19)-MSD	87%	70%	48%	64%	59%	17%
	WS1 (22)-MS	69%			59%		
	WS1 (22)-MSD	69%	69%	0%	60%	60%	1%
	S24 (24)-MSD	98%	NA	NA	78%	NA	NA
	T15 (28)-MS	66%			67%		
	T15 (28)-MSD	95%	81%	30%	102%	84%	40%
	T1LF156 (35)-MS	86%			88%		
	T1LF156 (35)-MSD	66%	76%	26%	73%	80%	19%
	S19 (39)-MS	140%			97%		
	S19 (39)-MSD	2%	71%	195%	-1%	48%	203%
	P9 (61)-MS	72%			52%		
	P9 (61)-MSD	68%	70%	7%	48%	50%	6%
	R47 (70)-MS	68%			59%		
	R47 (70)-MSD	78%	73%	14%	55%	57%	5%
	L4 (91)-MS	50%			58%		
	L4 (91)-MSD	119%	85%	82%	145%	102%	86%
	BTG9365 (92)-MS	70%	NA	NA	31%	NA	NA
	BTGhongdu1 (94)-MS	62%			18%		
	BTGhongdu1 (94)-MSD	63%	63%	2%	22%	20%	20%

LOQ = Limit of Quantitation
NA = Not Applicable

PFOS = Perfluorooctanesulfonate
POAA = Perfluorooctanoate
PFOSA = Perfluorooctanesulfonamide
PFHS = Perfluorohexane sulfonate

Data Entered/Analyst: 03/28/00, 04/05/00, 04/07/00, 05/07/00 MMH/LAC
Data Verified/Analyst:

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:

Author: Lisa Clemen, Glenn Langenburg

Approved By:

Laboratory Manager Date

Group Leader Date

Technical Reviewer 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 method.

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:

$$\frac{\text{mL of standard} \times \text{concentration of standard (µg /mL)}}{\text{mL of standard} + \text{mL of surrogate standard} + \text{initial matrix volume (mL)}} =$$

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

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):

$$\frac{\dfrac{\text{(ng of PFOS calc. from std. Curve x Dilution Factor)}}{\text{(Initial Weight of Liver (g)}}}{\text{Final Volume (mL)}} \times \frac{1\ \mu g}{1000\ ng}$$

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

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:**

Revision Date:

Author: Lisa Clemen, Robert Wynne

Approved By:

Laboratory Manager Date

Group Leader Date

Technical Reviewer Date

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 sample 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:

$$\frac{\text{g of Liver} \times \text{Average density* of homogenate (mg/mL)}}{(\text{g of Liver} + \text{g of Water})}$$

* 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:

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

*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 Potassium Perfluorooctanesulfonate or other Fluorochemicals in Liver Extracts 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 ng/mL	
120	120	120	120	120	120		0.500	
299	299	299	299	299	299			
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 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:

Author: Lisa Clemen, Robert Wynne

Approved By:

Laboratory Manager Date

Group Leader Date

Technical Reviewer Date

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):

$$\frac{\dfrac{(\text{ng of PFOS calc. from std. Curve} \times \text{Dilution Factor})}{(\text{Initial Volume of matrix (mL)} + \text{mL of Surrogate Standard})}}{\text{Final Volume (mL)}} \times \frac{1\ \mu g}{1000\ ng}$$

14.0 METHOD PERFORMANCE

14.1 Method Detection Limit (MDL) and Limit of Quantitation (LOQ) are method, analyte, and matrix specific. 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:**

Revision Date:

Author: Lisa Clemen, Glenn Langenburg

Approved By:

Laboratory Manager Date

Group Leader Date

Technical Reviewer Date

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