COMPOSITE ANALYTICAL LABORATORY REPORT

ON THE

Quantitative Analysis of Fluorochemicals in Environmental Samples

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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

> Exhibit 2814 State of Minnesota v. 3M Co., Court File No. 27-CV-10-28862

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TABLE OF CONTENTS

Analytical Study Personnel and Contributors	1
Introduction Purpose Test and Control Article Sample Collection and Analysis.	2 2
Sample Receipt and Maintenance	
Chemical Characterization Procurement	3 3
Method Summaries Preparatory and Analytical Methods Analytical Equipment	3 4 5
Data Summary, Analyses, and Results Summary of Quality Control Analyses Results Summary of Sample Results	6
Data Quality Objectives	8
Statement of Conclusion	9
References	9
Attachments	10
Report Signature	10

LIST OF TABLES

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4

Table 1. Description of Samples, by Study	2
Table 2. Procurement Information for Reference Materials in the Analysis of Environmenta Samples	
Table 3. Ions Monitored in the Analyses of Extracts of Groundwater	6
Table 4. Range of LOQs for Sera, by Study	8
Table 5. Range of LOQs for Liver and Other Tissues, by Study	8

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Proprietary and Confidential

ANALYTICAL STUDY PERSONNEL AND CONTRIBUTORS

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INTRODUCTION

Purpose

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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.

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, Henring Guli Plasma, Bald Eagle plasma, Cormorant blood, Henring Guli blood	Loon, Brown Pelican, Albatross	Albatross kidney, Cormorant yolk, Guli 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

Table 1. Description of Samples, by Study

Page 2 of 10

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

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Reference Material	LOT NUMBER	SOURCE
PFOS (potassium sait)	· 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

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

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

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 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 μ g/g of PFOS were subject to an additional PFOS verification process. Each sample was analyzed separately with respect to the 499 \rightarrow 99 transition and the 499 \rightarrow 80 transitions. The quantitative results

Page 4 of 10

3M_MN01666002

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 \rightarrow 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:

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

Injection volume: 10 µL Solvent Gradient:

Time (min)	%В
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

Page 5 of 10

3M_MN01666003

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

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

* 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

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- Standard Curves: The coefficient of determination (r²) for all 1/X weighted curves bracketing useable data was ≥ 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.

Page 6 of 10

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:

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- 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 Baid 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.

Page 7 of 10

- 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 (r²) 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

Page 8 of 10

3M_MN01666006

- 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

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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

Page 9 of 10

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-Q30 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

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

Dale L. Bacon, Spensor Representative

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Page 10 of 10

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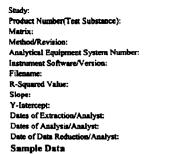
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BLOOD

ETS-8-4.1 & ETS-8-5.1 using unextracted curves Amelia 062498 Massiyax 3.2 See list to right See Attachments See Attachments 08/25/99 MCH/KK/SAL/SEE 08/26/99, 08/27/99, 08/28/99, 12/09/99 MEE/TAS 08/27/99, 08/30/99, 08/31/99, 12/10/99, 01/20/00 MEE/TAS

Sample #	Concentration of PFOS ug/mL	Mean PFOS ag/mL	RSD Std. Dev.	Concentration of PFOSA ug/mL	Mean PFOSA ug/mL	RSD Std. Dev.
H2O Blk-1 8/25/99	<loq (0.0116)<="" td=""><td></td><td></td><td><loq (0.00625)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00625)<="" td=""><td></td><td></td></loq>		
H2O BB-2 8/25/99	<loq (="" 0.0116)<="" td=""><td></td><td></td><td><loq (="" 0.00625)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.00625)<="" td=""><td></td><td></td></loq>		
H2O B&-3 8/25/99	<loq (0.0116)<="" td=""><td></td><td>NA</td><td><loq (="" 0.00625)<="" td=""><td>·</td><td>NA</td></loq></td></loq>		NA	<loq (="" 0.00625)<="" td=""><td>·</td><td>NA</td></loq>	·	NA
H2O Blk-4 8/25/99	<loq (="" 0.0116)<="" td=""><td>⊲LOQ</td><td>NA</td><td><loq (="" 0.00625)<="" td=""><td><1.0Q</td><td>NA</td></loq></td></loq>	⊲LOQ	NA	<loq (="" 0.00625)<="" td=""><td><1.0Q</td><td>NA</td></loq>	<1.0Q	NA
W2491-40,J 53	0.0180			<loq (="" 0.00625)<="" td=""><td></td><td></td></loq>		
W2491-41, J 11	<loq (0.0116)<="" td=""><td></td><td>•</td><td><loq (="" 0.00625)<="" td=""><td></td><td></td></loq></td></loq>		•	<loq (="" 0.00625)<="" td=""><td></td><td></td></loq>		
W2491-42,J 46	<loq (="" 0.0116)<="" td=""><td></td><td></td><td><loq (="" 0.00625)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.00625)<="" td=""><td></td><td></td></loq>		
W2491-43,J Ì7	0.0166			<loq (="" 0.00625)<="" td=""><td></td><td></td></loq>		
W2491-44,J 13	<loq (="" 0.0116)<="" td=""><td></td><td></td><td><loq (="" 0.00625)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.00625)<="" td=""><td></td><td></td></loq>		
W2491-45,J 8	0.0131		1	<loq (="" 0.00625)<="" td=""><td></td><td></td></loq>		
W2491-46,J 12	<loq (0.0116)<="" td=""><td>1</td><td></td><td><loq (0.00625)<="" td=""><td>1</td><td></td></loq></td></loq>	1		<loq (0.00625)<="" td=""><td>1</td><td></td></loq>	1	
W2491-47J 9	<loq (0.0116)<="" td=""><td></td><td></td><td><loq (0.00625)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00625)<="" td=""><td></td><td></td></loq>		
W2491-48,J 14	<loq (0.0116)<="" td=""><td></td><td></td><td><loq (0.00625)<="" td=""><td>1</td><td></td></loq></td></loq>			<loq (0.00625)<="" td=""><td>1</td><td></td></loq>	1	
W2491-49J 18	<loq (0.0116)<="" td=""><td></td><td></td><td><loq (="" 0.00625)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.00625)<="" td=""><td></td><td></td></loq>		
W2491-50 J 52	<loq (0.0116)<="" td=""><td></td><td></td><td><loq (0.00625)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00625)<="" td=""><td></td><td></td></loq>		
W2491-51J 55	<loq (0.0116)<="" td=""><td></td><td></td><td><loq (0.00625)<="" td=""><td>ļ</td><td>1</td></loq></td></loq>			<loq (0.00625)<="" td=""><td>ļ</td><td>1</td></loq>	ļ	1
W2491-52_J 10	<loq(0.0116)< td=""><td></td><td>NA</td><td><loq (0.00625)<="" td=""><td>I</td><td>NA</td></loq></td></loq(0.0116)<>		NA	<loq (0.00625)<="" td=""><td>I</td><td>NA</td></loq>	I	NA
W2491-53 J 15	<loq (="" 0.0116)<="" td=""><td><loq -="" 3="" outliers<="" td=""><td>NA</td><td><.00 (0.00625)</td><td><.00</td><td>NA</td></loq></td></loq>	<loq -="" 3="" outliers<="" td=""><td>NA</td><td><.00 (0.00625)</td><td><.00</td><td>NA</td></loq>	NA	<.00 (0.00625)	<.00	NA
	H2O Blk-1 8/25/99 H2O Blk-2 8/25/99 H2O Blk-3 8/25/99 H2O Blk-3 8/25/99 H2O Blk-4 8/25/99 W2491-40,J 53 W2491-41,J 11 W2491-42,J 46 W2491-43,J 17 W2491-43,J 17 W2491-43,J 8 W2491-45,J 8 W2491-45,J 8 W2491-45,J 8 W2491-45,J 18 W2491-45,J 18 W2491-50,J 52 W2491-51,J 55 W2491-52,J 10	of PFOS ug/mL H2O Bib-1 8/25/99 <loq (0.0116)<="" td=""> H2O Bib-2 8/25/99 <loq (0.0116)<="" td=""> H2O Bib-3 8/25/99 <loq (0.0116)<="" td=""> H2O Bib-4 8/25/99 <loq (0.0116)<="" td=""> H2O Bib-4 8/25/99 <loq (0.0116)<="" td=""> W2491-40J 53 0.0180 W2491-41 J 11 <loq (0.0116)<="" td=""> W2491-42 J 46 <loq (0.0116)<="" td=""> W2491-43 J 17 0.0166 W2491-43 J 17 0.0166 W2491-44 J 13 <loq (0.0116)<="" td=""> W2491-45 J 8 0.0131 W2491-45 J 12 <loq (0.0116)<="" td=""> W2491-45 J 14 <loq (0.0116)<="" td=""> W2491-45 J 15 <loq (0.0116)<="" td=""> W2491-45 J 18 <loq (0.0116)<="" td=""> W2491-45 J 15 <loq (0.0116)<="" td=""> W2491-51 J 55 <loq (0.0116)<="" td=""> W2491-51 J 55 <loq (0.0116)<="" td=""> W2491-52 J 10 <loq (0.0116)<="" td=""></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq>	of PFOS PFOS ug/mL ug/mL ug/mL H2O Bib-1 \$25/99 <loq (0.0116)<="" td=""> H2O Bib-2 \$25/99 <loq (0.0116)<="" td=""> H2O Bib-3 \$25/99 <loq (0.0116)<="" td=""> H2O Bib-4 \$25/99 <loq (0.0116)<="" td=""> H2O Bib-4 \$25/99 <loq (0.0116)<="" td=""> H2O Bib-4 \$25/99 <loq (0.0116)<="" td=""> W2491-40,J 33 0.0180 W2491-41,J 11 <loq (0.0116)<="" td=""> W2491-42,J 46 <loq (0.0116)<="" td=""> W2491-43,J 17 0.0166 W2491-44,J 13 <loq (0.0116)<="" td=""> W2491-45,J 8 0.0131 W2491-45,J 14 <loq (0.0116)<="" td=""> W2491-44,J 13 <loq (0.0116)<="" td=""> W2491-45,J 14 <loq (0.0116)<="" td=""> W2491-45,J 15 <loq (0.0116)<="" td=""> W2491-43,J 14 <loq (0.0116)<="" td=""> W2491-43,J 15 <loq (0.0116)<="" td=""> W2491-43,J 15 <loq (0.0116)<="" td=""> W2491-50,J 52 <loq (0.0116)<="" td=""> W2491-51,J 55 <loq (0.0116)<="" td=""> W2491-52,J 10 <loq (0.0116)<="" td=""></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq></loq>	of PPOS PFOS Std. Dev. H2O Bib-1 \$25599 <loq (0.0116)<="" td=""> sig/mL sig/mL</loq>	of PFOS ug/mL PFOS ug/mL Std. Dev. ug/mL of PFOSA ug/mL H2O Bib-1 \$225/99 <loq (0.0116)<="" td=""> ug/mL ug/mL</loq>	of PFOS ugmL PFOS ugmL Std. Dev. of PFOSA ugmL UpmL UpmL </td

FACT-GEN-021

None

Blood

GEN021 Various Matrices from MSU

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:
 08/27/59, 09/01/59, 12/30/99, 01/20/00, 02/14/00
 LAC

 Date Verified/ By:
 02/22/00
 MEE
 02/22/00
 MEE

LOQ = Limit of Quantita NA = Not Applicable

RSD = Relative Standard Deviation

ND = Not Detected

D = Detected

PFOS = Perfluorooctanesulfonanie PFOSA = Perfluorooctanesulfonanie POAA = Perfluorooctanesulfonate PFHS = Perfluorohexanesulfonate

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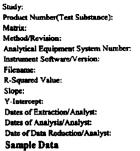
ETS-8-5.1 Excel Version 5/95

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5/10/00

2814.0014

GEN-021-sem_xis



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Group Dose	Sample #	Concentration of POAA	Mean POAA	RSD Std. Dev.	Concentration of PFHS	Menn PFHS	RSD Std. Dev.
1		ug/mL	eg/mL		eg/mL	ug/mL	
Method Blk	H2O Blk-1 \$/25/99	0.00629			סא		
	H2O Blk-2 \$/25/99	<loq (="" 0.00599)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loq>			ND		
	H2O Blk-3 \$/25/99	<loq (="" 0.00599)<="" td=""><td></td><td>NA</td><td>ND</td><td></td><td>NA</td></loq>		NA	ND		NA
	H20 Blk-4 \$/25/99	<loq (0.00599)<="" td=""><td><loq-1 outlier<="" td=""><td>NA</td><td>ND</td><td>ND</td><td>NA</td></loq-1></td></loq>	<loq-1 outlier<="" td=""><td>NA</td><td>ND</td><td>ND</td><td>NA</td></loq-1>	NA	ND	ND	NA
appian Seal Blood	W2491-40,J 53	<loq (="" 0.00599)<="" td=""><td></td><td></td><td>D</td><td></td><td></td></loq>			D		
	W2491-41,J 11	0.00759			D		
	W2491-42,3.46	<loq (0.00599)<="" td=""><td>· ·</td><td></td><td>ND</td><td></td><td></td></loq>	· ·		ND		
	W2491-43 J 17	<loq (0.00599)<="" td=""><td></td><td></td><td>D</td><td></td><td></td></loq>			D		
	W2491-44_J 13	<loq (0.00599)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loq>			ND		
1	W2491-45_J B	<loq (0.00599)<="" td=""><td></td><td>1</td><td>ND</td><td></td><td>:</td></loq>		1	ND		:
	W2491-46,J 12	<loq (0.00599)<="" td=""><td></td><td>i</td><td>ND</td><td></td><td>· ·</td></loq>		i	ND		· ·
	W2491-47 J 9	<loq (0.00599)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loq>			ND		
	W2491-48 J 14	0.00728			ND		
·	W2491-49J 18	<loq (="" 0.00599)<="" td=""><td></td><td>Į .</td><td>ND</td><td></td><td></td></loq>		Į .	ND		
· · · · · ·		0.0108		1	ND .		
	• W2491-51,J 55	0.0234			ND		
	W2491-52J 10	<loq (="" 0.00599)<="" td=""><td></td><td>NA</td><td>ND</td><td>· .</td><td>NA</td></loq>		NA	ND	· .	NA
	W2491-53J 15	<loq (0.00599)<="" td=""><td><.00 - 4 Outliers</td><td>NA</td><td>ND</td><td>ND - 3 Outliers</td><td>NA</td></loq>	<.00 - 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: Date Verified/ By:

08/27/99, 09/01/99, 12/30/99, 01/20/00, 02/14/00 LAC 02/22/00 MEE

FACT-GEN-021

GEN021 Various Matrices from MSU None Blood ETS-8-4.1 & ETS-8-5.1 using unextracted curves Amelia 062498 Massiynx 3.2 See list to right See Attachments See Attachments See Attachments 08/25/99 MCH/KK/SAL/SEE 08/26/99, 08/27/99, 08/28/99, 12/09/99 MEE/IAS 08/27/99, 08/30/99, 08/31/99, 12/10/99, 01/20/00 MEE/LAS

> NA - Not Applicable RSD - Relative Standard Deviation POAA - Perfluorooctanoate ND - Not Detected D - Detected

PFOS = Perfluorooct emito PFOSA - Perfluorooctanesulfonat

PFHS - Perfluorobexanesulfonate

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ETS-8-5.1 Excel Version 5/95

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5/10/00

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Study Product Number(Ten Substance): Matrix: Method/Revision Analytical Equipment System Number: Instrument Software/Version: Filename: R-Squared Value: Slope: Y-Intercept: Dates of Extraction/Analyst: Dates of Analysis/Analyst: Date of Data Reduction/Analyst:

Sample Data

BLOOD

GEN021	Various Matrices from MSU
None	
Blood	
BTS-8-4.1	& ETS-8-5.1 using unextracted curves
Amelia 06	2498
Manilynx	3.2, 3.3
08/28/99,	12/09/99 LAS
08/28/99	12/09/99 IAS
08/30/99,	12/10/99 IAS
See Attac	hments
08/25/99	MCH/KK/SAL/SEE
08/28/99.	12/09/99 LAS
08/30/99,	12/10/99 LAS

D Q	ic						
Group Dese	Sample #	Concentration of PFOS ug/mL or % Rec	Menn 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 Bik	H2O Bik-1 \$/25/99	NA			NA		
	H2O Bik-2 \$/25/99	NA			NA		1 •
	H2O Bik-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%			105%		
-	W2491-41,J 11-MS	11596			98%		1
1	W2491-42,J 46-MS	101%			73%		1
1	W2491-43,J 17-MS	2431%	-		1991%	• .	1:
	W2491-44J 13-MS	67%	I		50%		
	W2491-45, J.B. MS	103%			79%		
	W2491-46,J 12-MS	150%			116%		
	W2491-47, J 9-MS	103%			85%		1
	W2491-48J 14-MS	103%			90%		
	W2491-49J 18-MS	73%			47%		1
1	W2491-50,J 52-MS	65%			49%		
I	W2491-51 J 55-MS	17%	1		10%	Į	1.1
	W2491-52,J 10-MS	12%	• outlier excluded	58%	10%	• outlier excluded	58%
· '	W2491-53,J 15-MS	17%	84%	48%	12%	63%	37%
	PFHS based on PPOS response.	·····	LOQ = Limit of Quanti	tation	PPOS = Perfluorooctan	eralfonate	•

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

Date Verified/ By:

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02/16/00, 02/17/00 LAC 02/22/00 MEE

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NA = Not Applicable RSD = Relative Standard Deviation

ND = Not Detected D = Detected NS = Not Spiked

PFOSA = Perfluoroccanerulfonamide

<u>.</u>

POAA = Perfluorooctanoate PFHS = Perfluorohexanesulfonate

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Study: Product Number(Test Substance): Matrix: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Filename: R-Squared Value: Slope: Y-Intercept: Dates of Extraction/Analyst: Dates of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

BLOOD

GEN021	Various Matrices from MSU
None	
Blood	
ETS-8-4	1 & ETS-8-5.1 using unextracted curves
Amelia (62498
Meestyna	c 3.2, 3.3
08/28/99	12/09/99 IAS
08/28/99	12/09/99 IAS
08/30/99	12/10/99 IAS
See Atta	chments
08/25/99	MCH/KK/SAL/SEE
08/28/99	12/09/99 IAS
08/30/99	12/10/99 IAS

Greep	Sample #	Concentration	Mean	RSD	Concentration	Mean	RSD
Dese		of POAA	POAA	Std. Dev.	of PTHS	PTHS	Sel. Dev.
		ug/mL or % Roc	ug/mL or % Rec		ug/mL or % Roc	ug/mL er % Rec	
Method Bik	H2O Bik-1 8/25/99	NA			N\$		•
	H2O Blk-2 8/25/99	NA		1	NS	1	•
	H2O Blk-3 \$/25/99	NA		NA	NS		NS
•	H2O Blt-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		
	W2491-42,1 46-MS	100%		1	NS		
	W2491-43,J 17-MS	2056%	•		MS		
	W2491-44,J 13-MS	29%	· · ·	1	NS		
	W2491-45,J 8-MS	108%			NS		
	W2491-46,J 12-MS	174%			NS		
	W2491-47,J 9-MS	111%		1	NS		
	W2491-48,J 14-MS	112%		1	NS ·		· ·
	W2491-49,J 18-M5	91%		1	NS		
	W2491-50,J 52-MS	78%			NS	1	
	W2491-51, J 55-MS	14%			NS		
	W2491-52,J 10-M5	19%	• outlier excluded	54%	NS		NS
	W2491-53,J 15-MS	19%	91%	50%	NS	NS	NS

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

QC

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

D = Detected NS = Not Spiked PFOS - Perfluoroccanesulfonate PFOSA - Perfluoroccanesulfonate POAA - Perfluoroccanesulfonate PFHS - Perfluoroccanesulfonate

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ETS-8-5.1 Excel Version 5/95

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Study: Product Number(Test Substance): Matrix: Method/Ravision: Analytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

LIVER/WHOLE BLOOD

GEN021 Various Matrices from MSU None Various Matrices . ETS-4-6.0 & ETS-8-7.0 using unextracted ourves Amelia 062498 Mashyox 3-2 08/25/99 MCH/KE/SAL/SEE 08/25/99 MCH/KE/SAL/SEE 08/25/99, 08/27/99, 08/21/99, 12/10/99, 01/20/00 MEE/LAS

FACT-GEN-021

Group	Sample #	Concentration	Mean	RSD	Concentration	Mean	RSD
Dose		of PTOS	PFOS	Std. Dev.	of PFOSA	PFOSA	Std. Dev.
		wg/g or % Rec.	•2/2		ug/g or % Rec.	*#/g	
Method Bik	H2O Blb-1 \$/25/99	<loq (="" 0.0348)<="" td=""><td></td><td></td><td><loq (="" 0.0375)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0375)<="" td=""><td></td><td></td></loq>		
	H2O Blk-2 1/25/99	<loq (="" 0.0348)<="" td=""><td></td><td>1</td><td><loq (="" 0.0375)<="" td=""><td></td><td></td></loq></td></loq>		1	<loq (="" 0.0375)<="" td=""><td></td><td></td></loq>		
	H2O Blk-3 #/25/99	<loq (0.0348)<="" td=""><td></td><td><l0q< td=""><td><loq (="" 0.0375)<="" td=""><td></td><td>NA</td></loq></td></l0q<></td></loq>		<l0q< td=""><td><loq (="" 0.0375)<="" td=""><td></td><td>NA</td></loq></td></l0q<>	<loq (="" 0.0375)<="" td=""><td></td><td>NA</td></loq>		NA
	H2O Bth-4 \$25/99	<loq (="" 0.0348)<="" td=""><td><.0Q</td><td><L0Q</td><td><loq (="" 0.0375)<="" td=""><td><.00</td><td>NA</td></loq></td></loq>	<.0Q	< L 0Q	<loq (="" 0.0375)<="" td=""><td><.00</td><td>NA</td></loq>	<.00	NA
California Sea Lion Liver	W2491-3,CSL 3448	0.0384			0.0443		•
1	₩2491-4,CSL 3395	0.0494			<loq (="" 0.0375)<="" td=""><td>1</td><td></td></loq>	1	
	W2491-4,CSL 3020	<loq (0.0348)<="" td=""><td></td><td></td><td><loq (="" 0.0375)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-7,CSL 2169	<loq (0.0548)<="" td=""><td></td><td></td><td><loq (="" 0.0375)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-9,CSL 2839	<loq (="" 0.0348)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0375)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (="" 0.0375)<="" td=""><td></td><td>NA</td></loq>		NA
	W2491-10,CSL 2367	<loq (="" 0.0348)<="" td=""><td><loq -="" 2="" outliers<="" td=""><td>0.00773</td><td><loq (="" 0.0375)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>NA</td></loq></td></loq></td></loq></td></loq>	<loq -="" 2="" outliers<="" td=""><td>0.00773</td><td><loq (="" 0.0375)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>NA</td></loq></td></loq></td></loq>	0.00773	<loq (="" 0.0375)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>NA</td></loq></td></loq>	<loq -="" 1="" outlier<="" td=""><td>NA</td></loq>	NA
Elephant Seal Liver	W2491-2,ES 1500	<loq (0.0348)<="" td=""><td></td><td></td><td><loq (="" 0.0375)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-5,ES 1552	<loq (0.0548)<="" td=""><td></td><td></td><td><loq (="" 0.0375)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-11,ES 808	<.0Q(0.0345)			<loq (="" 0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-12,ES 772	<loq (0.0540)<="" td=""><td>1</td><td>NA</td><td><loq (0.0375)<="" td=""><td></td><td>NA</td></loq></td></loq>	1	NA	<loq (0.0375)<="" td=""><td></td><td>NA</td></loq>		NA
	W2491-14,ES 782	<loq (="" 0.0548)<="" td=""><td>4.00</td><td>NA</td><td><loq (0.0375)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	4.00	NA	<loq (0.0375)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
Harbor Seal Liver	W2491-8,HS	<loq (="" 0.0548)<="" td=""><td></td><td></td><td><loq (0.0375)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-13,ES 1191	<loq (0.0548)<="" td=""><td></td><td>NA</td><td><loq (0.0375)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0375)<="" td=""><td></td><td>NA</td></loq>		NA
	W2491-15,FIS 1199	0.0571	<loq -="" 1="" outlier<="" td=""><td>NA</td><td><loq (0.0375)<="" td=""><td>₹00</td><td>NA</td></loq></td></loq>	NA	<loq (0.0375)<="" td=""><td>₹00</td><td>NA</td></loq>	₹00	NA
Cessi Liver	W2491-1,NFS 100	0.133	0,133	NA	<loq (="" 0.0375)<="" td=""><td>₹.00</td><td>NA</td></loq>	₹.00	NA
Miak Liver	W2491-34,D1030 USFWS	4.85			<loq (0.0375)<="" td=""><td></td><td></td></loq>		
• .	W2491-35,D1146 USFWS	2.41		81.7	<loq (0.0375)<="" td=""><td></td><td>NA</td></loq>		NA
•	W2491-36,D1158 USFWS	0.587	2.62	2.14	<loq (0.0375)<="" td=""><td><100</td><td>NA</td></loq>	<100	NA
River Otter Liver	W2491-29,RAG 066	0.279			0.0371		
River Other Liver	W2491-JQ,RAG 028	0.994			0.0448		
ξ. 	W2491-31,RAG 148	0.189			0.0716		1
	W2491-J2,RAG 230	0.0336		116	<loq (="" 0.0375)<="" td=""><td></td><td>33.1</td></loq>		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)<="" td=""><td>I</td><td></td><td>0.0806</td><td></td><td>1</td></loq>	I		0.0806		1
	W2491-19,SO 11494-001	<loq (="" 8.6345)<="" td=""><td>1</td><td>1</td><td><loq (0.0375)<="" td=""><td></td><td></td></loq></td></loq>	1	1	<loq (0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-22,SO 11940-001	<loq (="" 0.0348)<="" td=""><td></td><td></td><td><loq (="" 0.0375)<="" td=""><td>1</td><td></td></loq></td></loq>			<loq (="" 0.0375)<="" td=""><td>1</td><td></td></loq>	1	
	W2491-24,SO 11309-001	<loq (="" 0.0343)<="" td=""><td></td><td></td><td><loq (="" 0.0375)<="" td=""><td></td><td>ł</td></loq></td></loq>			<loq (="" 0.0375)<="" td=""><td></td><td>ł</td></loq>		ł
	W2491-25,SO 12797-001	<loq (="" 0.0848)<="" td=""><td></td><td></td><td><loq (0.0375)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-26,SO 13110-001	<loq (="" 0.0848)<="" td=""><td></td><td></td><td><loq (0.0375)<="" td=""><td></td><td>1</td></loq></td></loq>			<loq (0.0375)<="" td=""><td></td><td>1</td></loq>		1
	W2491-27,SO 12679-001	<loq(0.0348)< td=""><td></td><td>NA</td><td><loq (="" 0.0375)<="" td=""><td></td><td>NA</td></loq></td></loq(0.0348)<>		NA	<loq (="" 0.0375)<="" td=""><td></td><td>NA</td></loq>		NA
	W2491-21_SO 12707-001	<loq (="" 0.0548)<="" td=""><td><.00</td><td>NA</td><td><loq (="" 0.0375)<="" td=""><td><loq -="" 1="" outlier.<="" td=""><td>. NA</td></loq></td></loq></td></loq>	<.00	NA	<loq (="" 0.0375)<="" td=""><td><loq -="" 1="" outlier.<="" td=""><td>. NA</td></loq></td></loq>	<loq -="" 1="" outlier.<="" td=""><td>. NA</td></loq>	. NA
Turtle Liver	W2491-37, Male Turtle (-2,8), Turtle Liver	0.239		İ.	<loq (0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-38, Male Turtle (2, 12), Turtle Liver	0.358		56.0	<loq (="" 0.0375)<="" td=""><td></td><td>I NA</td></loq>		I NA
	W2491-39, Female Turtle (-3,9), Turtle Liver	0.099	0.232	0.130	<1.00 (0.0375)	4.00	NA
San Otter Brain	W2491-18.SO 12593-001.Sea Otter Brain	<loq (="" 0.0548)<="" td=""><td>1</td><td>NA</td><td>0.0664</td><td></td><td>NA</td></loq>	1	NA	0.0664		NA
	W2491-21,SO 11494-001,Sea Otter Brain	<loq (0.0348)<="" td=""><td>₹.00</td><td>NA</td><td><loq (0.0375)<="" td=""><td>NA</td><td>NA</td></loq></td></loq>	₹.00	NA	<loq (0.0375)<="" td=""><td>NA</td><td>NA</td></loq>	NA	NA
Sea Otter Kidney	W2491-17,SO 12593-001,Sea Otter Kidney	4.00 (0.0545)	1	1 .	<loq (0.0375)<="" td=""><td>-</td><td>†</td></loq>	-	†
•	W2491-20,SO 11494-001,Sea Other Kidney	<loq (="" 0.0348)<="" td=""><td></td><td>NA</td><td><loo (="" 0.0375)<="" td=""><td></td><td>NA</td></loo></td></loq>		NA	<loo (="" 0.0375)<="" td=""><td></td><td>NA</td></loo>		NA
	W2491-23,SO 11940-001,Sea Other Kidney	<loq (="" 0.0348)<="" td=""><td><00⊳</td><td>NA</td><td><loq (0.0375)<="" td=""><td><.00</td><td>NA</td></loq></td></loq>	<00⊳	NA	<loq (0.0375)<="" td=""><td><.00</td><td>NA</td></loq>	<.00	NA
Whele Blood	W2491-54 Consonant DCCO L Charity	0.190	1	1	<loq (="" 0.0375)<="" td=""><td></td><td></td></loq>		
	W2491-55,Cormorant DCCO Hym Island, Lake Sup	0.0472	1	95.2	<loq (0.0375)<="" td=""><td></td><td>NA</td></loq>		NA
	W2491-56.Otter DCCO Great Lakes	0.0392	0.0904	0.0461	0.112	<loq-1 outlier<="" td=""><td></td></loq-1>	
FDS/FFHS - no curve analyzed PFHS ba		LOQ - Limit of Quantitat			PFOS = Perfuorooctanenal		1

Producers - and curve analysis a Prior South on Proc response. No PPOS qualitative confirmation performed. Identifications are preliminary. Devices Surrogate levels are not noted and ware not confirmed. LOQ = Limit or Quantization RSD = Relative Standard Deviation NA = Not Applicable ND = Not Detected PFOSA = Perfluorooctanesalitinasi PFOSA = Perfluorooctanesalitinasi POAA = Perfluorooctanesali PFHS = Perfluoroiceumeralitinase

Date Entered/Analyst: Date Verified/Analyst:

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08/27/99, 09/01/99, 12/30/99, 01/20/00, 02/14/00 LAC 02/22/00 MEE

ND = Not Detected D = Detected

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Study: Product Number(Test Substance): Matrix: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

GEN021 Various Matrices from MSU None None Various Matrices ETS-8-6.0 & ETS-8-7.0 using uncutracted curves Amelia 062498 Masslynx 3.2 04/25/99 MCH/KK/SAL/SEE 04/26/99 08/27/99 08/24/99, 12/09/99 MEE/IAS 04/27/99, 08/30/99, 08/31/99, 12/10/99, 01/20/00 MEE/IAS

LIVER/WHOLE BLOOD

Group	Sample #	Concentration	Mean	RSD	Concentration	Mean	RSD
Dase		AAO9 to	POAA	Std. Dev.	of PFHS	PFHS	Std. Dev.
		ug/g or % Rat.	*9/1		way or % Rec.	45/5	
Method Blk	H2O Blk-1 8/25/99	0.00602			ND		
	H2O Bit-2 \$/25/99	<loq (="" 0.0359)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loq>			ND		
	H2O Bik-3 \$/25/99	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td>ND</td><td></td><td>NA</td></loq>		NA	ND		NA
	H2O Blk-4 \$/25/99	<loq (="" 0.0359)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>NA</td><td>ND</td><td>סא</td><td>NA .</td></loq></td></loq>	<loq -="" 1="" outlier<="" td=""><td>NA</td><td>ND</td><td>סא</td><td>NA .</td></loq>	NA	ND	סא	NA .
California Sea Lion Liver	W2491-3,CSL 3448	<loq (="" 0.0359)<="" td=""><td></td><td></td><td>ND</td><td>,</td><td></td></loq>			ND	,	
	W2491-4,CSL 3395	0.0409	ļ		DM	,	·
	W2491-6,CSL 3020	<loq (="" 0.0359)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loq>			ND		
	W2491-7,CSL 2169	<loq (="" 0.0359)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loq>			ND		
	W2491-9.CSL 2139	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td>D</td><td></td><td>NA</td></loq>		NA	D		NA
	W2491-10,CSL 2367	<loq (0.0359)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>NA</td><td>ND</td><td>ND - 1 Outlier</td><td>NA</td></loq></td></loq>	<loq -="" 1="" outlier<="" td=""><td>NA</td><td>ND</td><td>ND - 1 Outlier</td><td>NA</td></loq>	NA	ND	ND - 1 Outlier	NA
Elephant Seal Liver	W2491-2,83 1500	<loq (0.0359)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loq>			ND		
	W2491-5,25 1552	<loq (0.0359)<="" td=""><td></td><td></td><td>סא</td><td>1 1</td><td></td></loq>			סא	1 1	
	W2491-11,85 806	<loq (0.0359)<="" td=""><td></td><td></td><td>ND</td><td>1 1</td><td>1</td></loq>			ND	1 1	1
	W2491-12ES 772	<loq (0.0359)<="" td=""><td></td><td>NA</td><td>ND D</td><td></td><td>NA</td></loq>		NA	ND D		NA
	W2491-14,25 782	<loq (0.0359)<="" td=""><td>4.00</td><td>NA</td><td>DM</td><td>ND</td><td>NA</td></loq>	4.00	NA	DM	ND	NA
Harber Seel Liver	W2491-6,HS	<loq (0.0359)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loq>			ND		
	W2491-13,85 1191	<loq (0.0359)<="" td=""><td></td><td>NA</td><td>ND</td><td></td><td>• NA</td></loq>		NA	ND		• NA
	W2491-15,85 1199	<loq (0.0359)<="" td=""><td>4.00</td><td>NA</td><td>ND</td><td>ND</td><td>NA</td></loq>	4.00	NA	ND	ND	NA
Gent Liver	W2491-1,10PS 100	<loq (="" 0.0359)<="" td=""><td>4.0Q</td><td>NA</td><td>D</td><td>D</td><td>NA</td></loq>	4.0Q	NA	D	D	NA
Mink Liver	W2491-34 D1050 USFWS	<loq (="" 0.0359)<="" td=""><td></td><td></td><td>D</td><td></td><td></td></loq>			D		
	W2491-35_D1146 USFWS	<loq (="" 0.0159)<="" td=""><td>1</td><td>NA</td><td>DND</td><td></td><td>NA</td></loq>	1	NA	DND		NA
	W2491-36,D1158 USFWS	<loq (="" 0.0359)<="" td=""><td>400</td><td>NA</td><td>ND</td><td>ND - 1 Outlier</td><td>NA</td></loq>	400	NA	ND	ND - 1 Outlier	NA
River Otter Liver	W2491-29,RAG 066	<loq (="" 0.0559)<="" td=""><td></td><td></td><td>Q</td><td></td><td></td></loq>			Q		
River Otter Liver	W2491-30 RAG 028	4.0Q (0.0359)			D		
	W2491-3LRAG 148	<loq (="" 0.0359)<="" td=""><td></td><td>l</td><td>D ·</td><td>1</td><td></td></loq>		l	D ·	1	
A. A	W2491-32,BAG 230	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td>ND</td><td></td><td>NA</td></loq>		NA	ND		NA
· .	W2491-33,RAG 237	<loq (="" 0.0359)<="" td=""><td><00⊅</td><td>NA</td><td>D</td><td>D - I Outlier</td><td>NA</td></loq>	<00⊅	NA	D	D - I Outlier	NA
Sea Otter Liver	W2491-16,S0 12593-001	<loq (="" 0.0359)<="" td=""><td></td><td></td><td>ND</td><td></td><td>÷</td></loq>			ND		÷
	W2491-19,SO 11494-001 ·	<1.00 (0.0359)			D		۰,
1	W2491-22.50 11940-001	<loo (="" 0.0359)<="" td=""><td></td><td></td><td>ND</td><td></td><td></td></loo>			ND		
	W2491-24,50 11309-001	<loq (="" 0.0359)<="" td=""><td></td><td>1</td><td>ND</td><td>1. 1</td><td></td></loq>		1	ND	1. 1	
	W2491-25.SO 12797-001	<loq (="" 0.0359)<="" td=""><td></td><td></td><td>D</td><td></td><td></td></loq>			D		
	W2491-26.SO 131 10-001	<loq (="" 0.0359)<="" td=""><td></td><td>1</td><td>ND</td><td></td><td></td></loq>		1	ND		
	W2491-27,50 12679-001	<0Q(0.0359)		NA	ND		NA
	W2491-28,SO 12707-001	<loq (="" 0.0359)<="" td=""><td><0Q.></td><td>NA</td><td>ND</td><td>ND - 2 Outliers</td><td>. NA</td></loq>	<0Q.>	NA	ND	ND - 2 Outliers	. NA
Turtle Liver	W2491-37 Male Turte (-2,8), Turtle Liver	<loq(0.0359)< td=""><td></td><td></td><td>DN</td><td></td><td></td></loq(0.0359)<>			DN		
	W2491-38, Male Turde (2,12), Turtle Liver	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td>D ·</td><td>1</td><td>NA</td></loq>		NA	D ·	1	NA
	W2491-39,Female Twtle (-3,9),Twtle Liver	<loq(0.0359)< td=""><td><.0Q</td><td>NA</td><td>D</td><td>D · 1 Outlier</td><td>NA</td></loq(0.0359)<>	<.0Q	NA	D	D · 1 Outlier	NA
Sea Otter Brain	W2491-18.SO (2593-00), See Otter Brain	<loq(0.0359)< td=""><td></td><td>NA</td><td>ND</td><td></td><td>NA</td></loq(0.0359)<>		NA	ND		NA
	W2491-21,SQ 11494-001,Sea Otter Brain	<loq (="" 0.0359)<="" td=""><td>⊲0Q</td><td>NA</td><td>ND</td><td>ND</td><td>NA</td></loq>	⊲0 Q	NA	ND	ND	NA
Sea Otter Kidney	W2491-17.SO 12593-001 Sea Otter Kidney	<.00 (0.0359)			ND		2.4
See one many	W2491-20.SO 11494-001.Sea Otter Kidney	<loq (="" 0.0359)<="" td=""><td>l l</td><td>NA</td><td>ND</td><td></td><td>NA</td></loq>	l l	NA	ND		NA
	W2491-23,SO 11946-001,Sea Other Kidney	<loq (="" 0.0359)<="" td=""><td> ⊲.00</td><td>NA</td><td>ND</td><td>ND</td><td>NA</td></loq>	⊲.00	NA	ND	ND	NA
Whele Blood	W2491-54 Consorant DCCO L Charity	<loo (="" 0.0359)<="" td=""><td></td><td>1</td><td>ND</td><td></td><td></td></loo>		1	ND		
	W2491-55,Connormat DOCO Hym Island, Lake Sup	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td>D</td><td></td><td>NA</td></loq>		NA	D		NA
					D D	D-1 Outlier	NA

PFDS/PFHS - no carve analyzed PFHS based on PFOS response. No PFOS qualitative confirmation performed. Identifications are preliminary. Devient Serregate levels are not noted and were not confirmal.

Date Entered/Analyst: Date Verified/Analyst:

06/27/99, 09/01/99, 12/30/99, 01/20/00, 02/14/00 LAC 02/22/00 MEE

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

PFOS - Perfluor PFOSA - Perfluo POAA - Perfluo eul fran

PTHS - Perfe .

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Study: Product Number(Test Substance): Matrix: Method/Revision: Analytical Equipment System Number: Instrument Software/Version:

Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data GEN021 Various Matrices from MSU None Various Matrices ETS-8-60 & ETS-8-7.0 using unextracted curves Amelia 062498 Masslynx 3.2, 3.3 08/25/99 MCH/KK/SAL/SEE 08/25/99 12/00/99 LAS 08/30/99, 12/10/99 LAS

Groep	Sample #	Concentration	Mean	RSD	Concentration	Mean	RSD
Dase		of PFOS ug/g or % Rec.	PFOS Recovery	Std. Dev.	of PFOSA	PFOSA	Std. De
Method Blk	H2O Bik-1 \$/25/99	NA	1		NA	198/8	
}	H2O Blk-2 8/25/99	NA			NA		1
1	H2O BIk-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%	1		66%		
	W2491-4,CSL 3395-MS	78%			52%		
	W2491-6,CSL 3020-MS	69%			48%		1
	W2491-7,CSL 2169-MS	45%			30%		
	W2491-9,CSL 2839-MS	31%	1	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 508-MS	51%			35%		
	W2491-12,ES 772-MS	44%		47%	18%		56%
ļ	W2491-14,ES 782-MS	33%	62%	29%	29%	45%	25%
Harbor Seal Liver	W2491-4.HS-MS	35%			42%		
	W2491-13,HS 1191-MS	\$1%		42%	79%		39%
	W2491-15.HS 1199-MS	50%	55%	23%	42%	54%	21%
Gozzi Liver	W2491-1 NFS 100-MS	57%	NA	NA	63%	NA	NA
Mink Liver	W2491-34.D1030 USFWS-MS	NR		<u> </u>	NR		144
·	W2491-35.D1 146 USFW3-MS	NR		NA	NR		
*	W2491-36.D1158 USFWS-MS	NR	NR	NA	NR	NR	NA
River Otter Liver	W2491-29.RAG 066-MS	NR			NR		1 100
	W2491-30,RAG 028-MS	NR	1		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%		- 374
	W2491-19.SO 11494-001-MS	61%		1	50%		
1	W2491-22.SO 11940-001-MS	44%			35%		1
	W2491-24_SO 11309-001-MS	27%			16%	1	
1	W2491-25.SO 12797-001-MS	34%			22%		
	W2491-26,SO 13110-001-MS	69%		ł	44%	1 1	
	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%	417	1074	42%	- 30%	137
	W2491-38, Male Turtle (2,12)-MS	35%		20%	51%		159
	W2491-39 Female Turtle (-3.9)-MS	51%	41%	207	39%	44%	6%
Sea Otter Brain	W2491-18.SO 12593-001-MS	42%	170	7%		4476	
	W2491-21_SO-11494-001-MS	46%	44%	3%	-3% 27%	12%	171
Sea Otter Kidney	W2491-17.SO 12593-001-MS	61%	4470	376		12%	219
Jek Otter KAusey	W2491-17,30 12393-001-MS	75%			66%		1
		24%	53%	49%	57%	1	619
Whole Blood	W2491-23,SO 11940-001-MS W2491-54.Comporant DCOD L Charity-MS	63%	3376	26%	14%	46%	28
D0040 9008 V				1	65%		1
	W2491-55,Cormorant DCCO Hym Island, Lake Sup-MS	69%		34%	64%		29
	W2491-56,Otter DCCO Great Lakes-MS	115%	\$2%	28%	104%	77%	23
THS - no curve analyzed, PTHS based		LOQ = Limit of Quanti			PFOS - Perfluorooctanesu		
XS qualitative confirmation performed. In lot reported, appears the spike wasn't dete		RSD = Relative Standar NA = Not Applicable	d Deviation		PFOSA = Perfluorooctane	PERCENT AND C	- i -
					POAA - Perfluorooctance		

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

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Study: Product Number(Test Substance): Matrix: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

GEN021 Various Matrices from MSU None Various Matrices ETS-8-6.0 & ETS-8-7.0 using unextracted curves E15-8-8.0 at 515-8-7.0 uning u Amelia 062498 Mamlynx 3.2, 3.3 08/25/99 MCH/KK/SAL/SEE 08/28/99, 12/09/99 LAS 08/30/99, 12/10/99 LAS

Groep		Sample #	Concentration	Mean	RSD	Concentration	Mean	RSD
Dose			of POAA	POAA	Std. Dev.	of PFHS	PTHS	Std. De
			ug/g or % Rec.	*9'E		WE/E OF % Rec.		2107 176
Method Bik		820 Bik-1 \$/25/99	NA			NS	·**/*	
		H2O Blk-2 \$/25/99	NA			NS		1
		H2O Bik-3 \$/25/99	NA		NA	NS	1	NA
		H2O Blk-4 \$/25/99	NA	NA	NA	NS ·	NS	- NA
California Sea Lio	Liver	W2491-3.CSL 3448-MS	109%			NS		
		W2491-4.CSL 3395-MS	85%			NS	1	1
		W7491-6.CSL 3020-MS	68%			NS		
		W2491-7,CSL 2169-MS	45%			NS		
		W2491-9,CSL 2839-MS	20%		47%	NS		NA NA
		W2491-10.CSL 2367-MS	64%	65%	31%	NS	NS	NA NA
Elephant Seal L	Iver	W2491-Z.ES 1500-MS	100%			NS		
		JV2491-5,ES 1552-MS	80%			NS		1.
		-W2491-11_ES 808-MS	44%			NS		
,		W2491-12.ES 772-MS	31%		52%	NS	1	. NA
		W2491-14.ES 782-MS	36%	58%	30%	NS	NS	NA NA
Harbor Seal L		W2491-4.HS-MS	48%			NS		1 84
		W2491-13,HS 1191-MS	94%		37%	NS	· •	l
	i	W2491-15,HS 1199-MS	58%	67%	25%	NS	NS	NA NA
Gozzi Live		W2491-1.5453 100-MS	91%	NA	NA NA	NS	NS	NA
- Mink Live		W2491-1,CU'S TOCHES W2491-34,D1030 USFWS-MS		NA .	<u> </u>		- NS	<u> </u>
- Minik Live		W2491-34,D1050 USF WS-MS W2491-35,D1146 USF WS-MS	NR NR			NS	1	
	3		NR	NR	NA NA	NS		NA
N		W2491-34,D1158 USFWS-MS	21%	MK	NA	NS	NS	NA
River Otter L	wer i	W2491-29,RAG 066-MS , W2491-30,RAG 028-MS	46%			NS		
•					1	NS		1.
		W2491-31,RAG 148-MS	69%			NS		ł
		W2491-32,RAG 230-MS	52%		- 39%	NS		NA
		W2491-33,RAG 237-MS	70%	52%	20%	NS	NS -	<u>N/</u>
Sea Otter Li	MET	W2491-16,SO 12593-001-MS	47%			NS	· ·	1 ·
		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%	t		NS		1 :
	1	W2491-26,SO 13110-001-MS	83%			NS		12
		W2491-27,SO 12679-001-MS	44%		39%	NS		- NA
		W2491-28,SO 12707-001-MS	64%	50%	20%	NS	• NS	NA
Turtle Liv	*	W2491-37, Male Turtle (-2,8)-MS	48%			NS		1
		W2491-38, Male Turde (2,12)-MS	71%		29%	NS	1	NA
		W2491-39,Female Twtle (-3,9)-MS	42%	54%	15%	NS	NS	N/
Sea Otter B	ala l	W2491-18,SO 12593-001-MS	41%		13%	NS		N/
		W2491-21,SO 11494-001-MS	50%	46%	6%	NS	NS	N/
Sea Otter Ki	lawy	W2491-17,SO 12593-001-MS	94%			NS		T
		W2491-20,SO 11494-001-MS	95%		75%	NS		N/
	1.	W2491-23,SO 11940-001-MS	9%	66%	49%	NS	NS	N.
Whole Blo	M I	W2491-54,Cormorant DCCO L Charity-MS	112%	1	· ·	NS		1
		W2491-55, Cormorant DCCO Hyrn Island, Lake Sup-MS	122%	1	2%	NS	· ·	N
		W2491-56,Otter DCCO Great Lakes-MS	123%	121%	3%	NS	NS	N
PFDS/PFHS = no curve and				LOO - Limit of		PFOS = Perfluomoctan		_

No PFOS qualitative confirmation performed. Identifications are preliminary. NR = Not reported, appears the spike wan't detectable from endogenous levels.

Date Entered/Analyst: Date Verified/Analyst:

02/16/00, 02/17/00 LAC 20/22/00 MEE

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 RSD = Relative Standard Deviation
 POCA - Perfluorocctaneouslo

 NA = Not Applicable
 POAA = Perfluorocctaneouslo

 NS = Not spilced
 PFHS = Perfluorocctaneouslo

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C 1991 Wilson Jones Company

Study: Product Number(Test Substance): Matrix: Method/Revision: Analytical Equipment System Number: instrument Software/Version: Filename R-Squared Value: R-Squared Value: Slope: Y-Intercept: Dates of Extraction/Analyst: Dates of Data Reduction/Analyst: Dates of Data Reduction/Analyst:

GEN024 MSU Environmental Samples GEN024 MSU Environmental Samples Various Various ETS-8-4.1 & ETS-8-5.1 using unextracted curves Ametia 062498 Massiyau. 3.3 See Attachments See Attachments See Attachments See Attachments 10/1299 S.A./KK 10/15/99, 10/19/99 IAS/MMH, 12/13/99 IAS 10/18/99, 10/20/99 HOJ, 12/14/99 MMH

Sample Data

Group Dose	Sample #	Concentration of PFOS	Menn PFOS	RSD Std. Dev.	Concentration of PFHS	Mean PTHS	RSD Std. Dev.
		ug/mi, or % Rec	eg/mi.	RPD-MS/MSD	ug/mL or % Rec	eg/est.	RPD-MS/MS
Method Blk	Bird10129-WBlk-5-1	<loq (0.00116="" ml)<="" th="" ug=""><th>QQ</th><th>40Q</th><th><loq (0.00114="" ml)<="" th="" ug=""><th>4.00</th><th><0Q</th></loq></th></loq>	QQ	40Q	<loq (0.00114="" ml)<="" th="" ug=""><th>4.00</th><th><0Q</th></loq>	4.00	<0Q
Matrix Blk	NE	NB			NE		
	NE	NE	NE	NE	NE	NE ·	NB
MS/MSD	Bird 040-Alb sera-MS-250 ppb-5-1	81%			68%		
250 ppb	Bird 040-Alb sem-MSD-250 ppb-5-1	96%	89%	17%	81%	75%	17%
	Bird 060-E plasma-MS-250 pph-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%
Albairos	Bird 034-Albetross Chick sera	0.0393			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 035-Albetross pleams	0.0256			<loq (0.00114="" td="" waymal.)<=""><td></td><td></td></loq>		
	Bird 036-Albatross sen	0.00755			<loq (0.00114="" ml)<="" td="" w=""><td></td><td></td></loq>		
	Bird 037-Albetros plasme	0.0196			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 438-Albetrom pers	0.00346			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>•</td></loq>		•
	Bird 039-Albatrons sera	0.00677			<loq (0.00114="" mg="" ml)<="" td=""><td></td><td></td></loq>		
	Bird 040-Albetross sera	0.00621			<loq (0.00114="" mg="" ml)<="" td=""><td></td><td>• • •</td></loq>		• • •
	Bird 041-Albetross sura	0.00430	Planna	47.0	<loq (0.00114="" ml)<="" td="" ug=""><td>Plasma</td><td>NÁ</td></loq>	Plasma	NÁ
	Bird 042-Albetross sera	0.00402	0.0180	0.00647	<loq (0.00114="" ml)<="" td="" ug=""><td>4.00</td><td>NA -</td></loq>	4.00	NA -
	Bird 043-Alberross sera	0.00546			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 044-Albatross sera	0.00897		1	<loq (0.00114="" ml)<="" td="" up=""><td></td><td></td></loq>		
	Bird 045-Albetross sera	0.00657	Sera	115	<loq (0.00114="" ml)<="" td="" ug=""><td>Sen</td><td>NA</td></loq>	Sen	NA
•	Bird 046-Albetross plasms	0.00089	0.00926	0.0107	<loq (0.00114="" ml.)<="" td="" wg=""><td>₹.00</td><td>NA</td></loq>	₹.00	NA
Comerant	Bird 047-Comorant plasma	0.431			<loq (0.00114="" ml)<="" mm="" td=""><td></td><td></td></loq>		
Comorant	Bird 048-Comorant plasma	0.242		61.8	<loq (0.00114="" m="" ml)<="" td=""><td></td><td>NA</td></loq>		NA
	Bird 049-Comorant plasma	0.110	0.261	0.161	<loq (0.00114="" mg="" ml)<="" td=""><td><l00< td=""><td>NA NA</td></l00<></td></loq>	<l00< td=""><td>NA NA</td></l00<>	NA NA
	Bird 059-Comprant blood	0.0725	0.201		<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 051-Comprant blood	0.218					ł
					<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>1 -</td></loq>		1 -
	Bird 052-Comorant blood	0.153			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 053-Comorant blood	<loq (0.00116="" mg="" ml)<="" td=""><td></td><td></td><td><loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 054-Comprent blood	0.144	1	44.5	<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>NA -</td></loq>		NA -
· · ·	Bird 055-Comorant blood	0.273	0.172 - 1 Outlier	0.0764	<loq (0.00114="" mll)<="" td="" ug=""><td><.0Q</td><td>NA</td></loq>	<.0Q	NA
Herring	Bird 056-Herring Gull plasma	0.453		34.1	0.00243		NA NA
	Bird 057-Herring Gull plasma	0.277	0.365	0.125	<loq (0.00114="" ml)<="" td="" up=""><td>NA</td><td>NA</td></loq>	NA	NA
	Bird 058-Herring Gull blood	0.0790		12.2	<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>NA .</td></loq>		NA .
	Bird 059-Herring Gull blood	0.0664	0.0727	0.00888	<loq (0.00114="" ml)<="" td="" up=""><td>. <loq< td=""><td>NA</td></loq<></td></loq>	. <loq< td=""><td>NA</td></loq<>	NA
Baid Eagle	Bird 060-Bald Eagle planma	0.449			<loq (0.00114="" ml)<="" td="" wg=""><td></td><td></td></loq>		
	Bird 061-Bald Eagle plasma	0.316	l .'		<loq (0.00114="" ml)<="" td="" ug=""><td>• *</td><td></td></loq>	• *	
	Bird 062-Baid Eagle plasma	0.185		1	0.00148		
	Bird 063-Bald Eagle plasma	0,167	. ·		<loq (0.001="" [4="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 064-Bald Bagle plasma	0.0402	ł		<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 065-Baid Engle plasma	0.0147			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 056-Bald Eagle plasma	0.122	1		<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 067-Bald Eagle plasma	0.0665			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>1</td></loq>		1
	Bird 068-Bald Eagle plasma	2.57			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 069-Bald Eagle planne	2.35			<loq (0.00114="" mg="" ml)<="" td=""><td></td><td></td></loq>		
	Bird 070-Bald Eagle plasma	0.0324			<loq (0.00114="" ml)<="" mm="" td=""><td></td><td>· ·</td></loq>		· ·
	Bird 071-Bald Eagle planna	0.0908			<loq (0.00114="" m="" ml)<="" td=""><td>[</td><td></td></loq>	[
	Bird 072-Bald Eagle plasma	0.217	1		<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 073-Baid Eagle please	0.168	ļ		0.00131	•	
	Bird 074-Bald Ragie pingan	0.163	1	· ·	<loq (0.00114="" ml)<="" td="" up=""><td></td><td></td></loq>		
	Bird 075-Baid Engle playan	0.0622			<loq (0.00114="" ml.)<="" td="" up=""><td></td><td>1</td></loq>		1
	Bird 076-Baid Eagle planne	0.0236			<loo (0.00114="" m="" ml)<="" td=""><td></td><td>1 .</td></loo>		1 .
	Bird 077-Baid Eagle plasma	0.0573	1	1	<loq (0.00114="" ml)<="" mm="" td=""><td></td><td>1</td></loq>		1
	Bird 077-Baid Eagle plasma	<loq (0.00116="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.00114="" ml)<="" td="" ug=""><td></td><td>· ·</td></loq></td></loq>			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>· ·</td></loq>		· ·
		0.216	1	1		1	1
	Bird 079-Bald Eagle plasma		1		<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 000-Bald Eagle plasma	0.281	l		<loq (0.00114="" ml)<="" td="" wg=""><td></td><td></td></loq>		
	Bird 061-Bald Eagle plasma	0.649	1		0.00617		
	Bird 082-Bald Eagle plasma	0.335	1	1	<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>1 7</td></loq>		1 7
	Bird 063-Bald Eagle plasma	0.0718	1	1	<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>1</td></loq>		1
	Bird 064-Bald Eagle plasma	0.0381	1	143	<loq (0.00114="" ml)<="" td="" ug=""><td></td><td>NA'</td></loq>		NA'
	Bird 065-Bald Eagle plasms	0.118	0.356 - 1 Outlie	ar 0.651	<loq (0.00114="" ml)<="" td="" ug=""><td><loo -="" 1="" outliers<="" td=""><td>NA NA</td></loo></td></loq>	<loo -="" 1="" outliers<="" td=""><td>NA NA</td></loo>	NA NA

No FFOS qualitative confirmation performed. Ident * Appears to not have been spiked. LAC 10/19/99

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

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

PFOS = Perfluorobecanoralfonate PFHS = Perfluorobecanorsalfonate POAA = Perfluorobecanorsalfonate PFOSA = Perfluoroocunesulfo .

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ETS-4-5.1 Excel Version 5/95

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GEN-024-sera.xis

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Study: Product Number(Test Substance): Matrix: Metta: Metbod/Revision: Analytical Equipment System Number: Instrument Software/Version: Filename: riterana: R-Squared Value: Slope: Y-Intercept Dates of Analysis/Analyst: Dates of Analysis/Analyst: Dates of Data Reduction/Analyst:

Sample Data

SERA

GEN024	MSU Environmental Samples
Various	-
Various	
BTS-8-4.	1 & ETS-8-5.1 using unextracted curves
Amelia 0	62498
Massiym	(3.3
See Atta	churrente
See Atta	chments
See Atta	chrocots
See Atta	chments
10/12/99	SAL/KK
10/15/99	, 10/19/99 JAS/MMH, 12/13/99 JAS
10/18/99	10/20/99 HOJ, 12/14/99 MMH

Dose Method Blk Matrix Blk MS/MSD 250 ppb	Bird10129-WBDc-5-1 NE	of POAA ug/mL or % Rec <loq (0.0299="" ml)<="" th="" ug=""><th>POAA ug/mL</th><th>Std. Dev. RPD-MS/MSD</th><th>of PFOSA ug/mL or % Rec</th><th>PFOSA ug/mL</th><th>Std. Dev.</th></loq>	POAA ug/mL	Std. Dev. RPD-MS/MSD	of PFOSA ug/mL or % Rec	PFOSA ug/mL	Std. Dev.
Matrix Blk MS/MSD							
Matrix Blk MS/MSD			<0Q	4.00	<loq (0.00625="" ml)<="" th="" ug=""><th><.00</th><th>RPD-MS/MS</th></loq>	<.00	RPD-MS/MS
MS/MSD		NE			NE		400
	NE	NE	NE	NE	NE	· NB	NE
250 ppb	Bird 040-Alb sem-MS-250 ppb-S-1	19%			70%		
	Bird 040-Alb sera-MSD-250 ppb-5-1	105%	97%	17%	80%	75%	14%
	Bird 060-E plasma-MS-250 ppb-5-2	77%			59%		
	Bird 060-E plasma-MSD-250 ppb-5-2	14%	80%		65%	62%	9%
	Bird 054-C blood-MS-250 ppb-5 Bird 054-C blood-MSD-250 ppb-5	90%, 23%	56%	119%	77%		
Albatron	Bird 034-Alberross Chick sen	<loq (0.0299="" ml)<="" td="" ug=""><td>2070</td><td>11776</td><td>14% <loq (0.00625="" ml)<="" td="" ug=""><td>45%</td><td>140%</td></loq></td></loq>	2070	11776	14% <loq (0.00625="" ml)<="" td="" ug=""><td>45%</td><td>140%</td></loq>	45%	140%
	Bird 035-Albetron planma	<loq (0.0299="" ml)<="" td="" w=""><td> </td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 036-Alberron sure	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.00625="" ml.)<="" td="" ug=""><td></td><td>•</td></loq></td></loq>			<loq (0.00625="" ml.)<="" td="" ug=""><td></td><td>•</td></loq>		•
	Bird 037-Albetross plasma	<loq (0.0299="" mg="" ml)<="" td=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 038-Alberros sera	<loq (0.0299="" ml)<="" td="" w=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 039-Albetrons sera	<loq (0.0299="" ml)<="" td="" up=""><td>1</td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>	1		<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 040-Albutrons sera	<loq (0.0299="" ml)<="" td="" w=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 041-Albatross sura	<loq (0.0299="" ml)<="" td="" up=""><td>Plasma</td><td>NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td>Plasma</td><td>NA</td></loq></td></loq>	Plasma	NA	<loq (0.00625="" ml)<="" td="" ug=""><td>Plasma</td><td>NA</td></loq>	Plasma	NA
	Bird 042-Albetroes sure	<loq (0.0299="" ml)<="" td="" ug=""><td>₹00</td><td>NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td><0Q </td><td>NA</td></loq></td></loq>	₹00	NA	<loq (0.00625="" ml)<="" td="" ug=""><td><0Q </td><td>NA</td></loq>	<0Q	NA
-	Bird 043-Albetross sera	<loq (0.0299="" tal.)<="" td="" we=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
•	Bird 044-Albetross sers	<loq (0.0299="" ml)<="" td="" w=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 045-Alberross sera	<loq (0.0299="" mg="" ml)<="" td=""><td>Sena ⊲_000</td><td>NA NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td>Sera</td><td>NA</td></loq></td></loq>	Sena ⊲_000	NA NA	<loq (0.00625="" ml)<="" td="" ug=""><td>Sera</td><td>NA</td></loq>	Sera	NA
	Bird 046-Albetrost plasma Bird 047-Comorant plasma	<loq (0.0299="" mg="" ml)<="" td=""><td>and</td><td>NA</td><td><1.0Q (0.00625 mg/mL)</td><td><.0Q</td><td>NA</td></loq>	and	NA	<1.0Q (0.00625 mg/mL)	<.0Q	NA
Comorant	Bird 048-Comorant plasma	<loq (0.0299="" ml)<br="" ug=""><loq (0.0299="" ml)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.00625="" ag="" ml)<="" td=""><td></td><td></td></loq></td></loq></loq>		NA	<loq (0.00625="" ag="" ml)<="" td=""><td></td><td></td></loq>		
	Bird 049-Comorant plasma	<loq (0.0299="" ml)<="" td="" w=""><td>4.00</td><td>NA</td><td><loq (0.00625="" ml)<br="" wg=""><loq (0.00625="" ml)<="" td="" wg=""><td>4.00</td><td>NA NA</td></loq></loq></td></loq>	4.00	NA	<loq (0.00625="" ml)<br="" wg=""><loq (0.00625="" ml)<="" td="" wg=""><td>4.00</td><td>NA NA</td></loq></loq>	4.00	NA NA
	Bird 050-Comorant blood	<loq (0.0299="" ml)<="" td="" w=""><td></td><td>144</td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>nia.</td></loq></td></loq>		144	<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>nia.</td></loq>		nia.
	Bird 05 L-Compress blood	<loq (0.0299="" ml)<="" td="" w=""><td></td><td></td><td>0.0145</td><td>1 1</td><td></td></loq>			0.0145	1 1	
	Bird 052-Comorant blood	<loo (0.0299="" ag="" ml.)<="" td=""><td></td><td></td><td>0.00871</td><td></td><td></td></loo>			0.00871		
	Bird 053-Comorant blood	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td>1 1</td><td>. –</td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td>1 1</td><td>. –</td></loq>	1 1	. –
	Bird 054-Comorant blood	<loq (0.0299="" mg="" ml)<="" td=""><td></td><td>NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>82.7</td></loq></td></loq>		NA	<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>82.7</td></loq>		82.7
	Bird 055-Comorant blood	0.0689	<loq-1 outlier<="" td=""><td>NA</td><td>0.0426</td><td>0.0219 - 3 Outliers</td><td>° 0.0131</td></loq-1>	NA	0.0426	0.0219 - 3 Outliers	° 0.0131
Herring	Bird 056-Herring Gull plasma	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>NA</td></loq>		NA
	Bird 057-Herring Gull plasma	<loq (0.0299="" ml)<="" td="" ug=""><td>4.00</td><td>NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td>⊲0Q</td><td>NA</td></loq></td></loq>	4.00	NA	<loq (0.00625="" ml)<="" td="" ug=""><td>⊲0Q</td><td>NA</td></loq>	⊲ 0Q	NA
	Bird 058-Herring Gull blood	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>, NA</td></loq></td></loq>		NA	<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>, NA</td></loq>		, NA
	Bird 059-Herring Gull blood	<loq (0.0299="" ml)<="" td="" ug=""><td>4.00</td><td>NA</td><td><loq (0.00625="" mg="" ml)<="" td=""><td>_4.0Q</td><td>NA</td></loq></td></loq>	4.00	NA	<loq (0.00625="" mg="" ml)<="" td=""><td>_4.0Q</td><td>NA</td></loq>	_4.0Q	NA
Baid Lagie	Bird 060-Bald Eagle plasma	<loq (0.0299="" ml)<="" td="" ug=""><td>· .</td><td>1</td><td><loq (0.00625="" mg="" ml)<="" td=""><td>· ·</td><td>- ·</td></loq></td></loq>	· .	1	<loq (0.00625="" mg="" ml)<="" td=""><td>· ·</td><td>- ·</td></loq>	· ·	- ·
	Bird 061-Bald Eagle plasma Bird 062-Bald Eagle plasma	<loq (0.0299="" ml)<br="" ug=""><loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.00625="" ml)<br="" ug=""><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></loq></td></loq></loq>			<loq (0.00625="" ml)<br="" ug=""><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></loq>		
	Bird 063-Bald Eagle plasma	<1.0Q (0.0299 ag/mL)	· ·		<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>•••</td></loq>		•••
	Bird 064-Bald Eagle planna	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 065-Bald Eagle plasma	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>• :</td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>• :</td></loq>		• :
	Bird 066-Bald Engle ploame	<loq (0.0299="" mg="" ml.)<="" td=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td>1</td><td>ł</td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td>1</td><td>ł</td></loq>	1	ł
	Bird 067-Bald Eagle plasma	<loq (0.0299="" td="" winl.)<=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>1</td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>1</td></loq>		1
1	Bird 068-Bald Eagle plasma	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td>0.0751</td><td></td><td></td></loq>			0.0751		
	Bird 069-Baid Eagle plasma	<loq (0.0299="" mi.)<="" ng="" td=""><td></td><td></td><td>0.0996</td><td></td><td></td></loq>			0.0996		
	Bird 070-Bald Eagle plasma.	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.00625="" ml.)<="" td="" ug=""><td></td><td>1</td></loq></td></loq>			<loq (0.00625="" ml.)<="" td="" ug=""><td></td><td>1</td></loq>		1
	Bird 071-Buid Engle planam	<loq (0.0299="" mg="" ml)<="" td=""><td></td><td>1</td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>		1	<loq (0.00625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	Bird 072-Bald Eagle plasma	<.00 (0.0299 up/mL)	i i		<loq (0.00625="" ml)<="" td="" wg=""><td></td><td></td></loq>		
	Bird 073-Bald Engle plasma	<loq (0.0299="" ml)<="" td="" up=""><td></td><td></td><td><loq (0.00625="" mg="" ml)<="" td=""><td></td><td>1</td></loq></td></loq>			<loq (0.00625="" mg="" ml)<="" td=""><td></td><td>1</td></loq>		1
	Bird 074-Baid Eagle plasma Bird 075-Baid Eagle plasma	<loq (0.0299="" ml)<br="" vg=""><loq (0.0299="" ml)<="" td="" vg=""><td></td><td></td><td><loq (0.00625="" ml)<br="" wg=""><loq (0.00625="" ml)<="" td="" wg=""><td></td><td>1</td></loq></loq></td></loq></loq>			<loq (0.00625="" ml)<br="" wg=""><loq (0.00625="" ml)<="" td="" wg=""><td></td><td>1</td></loq></loq>		1
	Bird 075-Baid Eagle plasme	<loq (0.0299="" ml)<="" td="" w=""><td>1</td><td></td><td><loq (0.00625="" ml)<="" td="" up=""><td>1</td><td></td></loq></td></loq>	1		<loq (0.00625="" ml)<="" td="" up=""><td>1</td><td></td></loq>	1	
	Bird 077-Bald Eagle planna	<loq (0.0299="" ml)<="" td="" w=""><td></td><td>ł</td><td><loq (0.00625="" mg="" ml)<="" td=""><td></td><td></td></loq></td></loq>		ł	<loq (0.00625="" mg="" ml)<="" td=""><td></td><td></td></loq>		
	Bird 078-Bald Eagle planns	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td><.00 (0.00625 ur/mL)</td><td>1</td><td>57</td></loq>			<.00 (0.00625 ur/mL)	1	57
	Bird 079-Baid Engle plasma	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.00625="" ml)<="" td="" w=""><td>ł</td><td>1</td></loq></td></loq>			<loq (0.00625="" ml)<="" td="" w=""><td>ł</td><td>1</td></loq>	ł	1
	Bird 080-Bald Eagle planne	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td>1</td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>1</td></loq></td></loq>		1	<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>1</td></loq>		1
	Bird 081-Bald Eagle plasma	<loq (0.0299="" ml)<="" td="" w=""><td>1</td><td>1</td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>· ·</td></loq></td></loq>	1	1	<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>· ·</td></loq>		· ·
	Bird 082-Bald Eagle plasma	<loq (0.0299="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>1</td></loq></td></loq>	1		<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>1</td></loq>		1
	Bird 083-Baid Eagle plasma	<loq (0.0299="" ml)<="" td="" up=""><td></td><td>1</td><td><1.0Q (0.00625 ug/mL)</td><td></td><td></td></loq>		1	<1.0Q (0.00625 ug/mL)		
	Bird 064-Bald Eagle plasma	<loq (0.0299="" ml)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.00625="" ml)<="" td="" ug=""><td></td><td>NA</td></loq>		NA
	Bird 085-Bald Eagle planns a performed. Identifications are preliminary.	<loq (0.0299="" ml)<="" td="" ug=""><td><loq NE = Not estracted</loq </td><td>NA</td><td><loq (0.00625="" ml)<="" td="" ug=""><td><loq -="" 2="" outliers<="" td=""><td>NA</td></loq></td></loq></td></loq>	<loq NE = Not estracted</loq 	NA	<loq (0.00625="" ml)<="" td="" ug=""><td><loq -="" 2="" outliers<="" td=""><td>NA</td></loq></td></loq>	<loq -="" 2="" outliers<="" td=""><td>NA</td></loq>	NA

No PFOS qualitative confirmation performed. Identifications are preliminary. • Appears to not have been spiked. LAC 10/19/99

Dess Entered/By: Data Verified/ By:

10/18/99, 10/20/99 LAC 12/30/99 MMH 2/17/00 mmk

NE = Not estract NE = Not extracted NA = Not Applicable NS = Not Spikad LOQ = Lizzh of Quantitation RSD = Relative Standard Deviation RPD = Relative Percent Difference

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PFHS = Perfluorohexamenal POAA = Perfluorooctanosis PFOSA = Perfluorooctanosis

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Study: Product Number(Test Substance): Matrix; Mains: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

GEN024 MSU Environmental Samples Various Various ETS-8-6.0 & ETS-8-7.0 using unextracted curves Ametia 062498 Massiyox 3.3 10/12/99 SAL/KK 10/14/99, 12/13/99 HOJ/LAS 10/15/99, 11/15/99, 12/14/99 HOJ/MMH

LIVER, KIDNEY, YOLK

Group Dose	Sampie #	Concentration of PFOS	Menn PFOS	RSD Std. Dev.	Concentration of PFHS	Mean PFHS	RSD Std. Dev.
		we'z or % Rec.	45/2	MS/MSD RPD	ug/g or % Rec.	-1/1	MS/MSD RP
Method Blk	Bird10129-wblk-5-1	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td></td><td>MONTOD RE</td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td></td><td>MONTOD RE</td></loq>		MONTOD RE
	Bird10129-wblk-6-1	<loq (0.0348="" g)<="" td="" ug=""><td><.00</td><td>NA</td><td><loq (0.0683="" g)<="" td="" ug=""><td> ⊲.00</td><td>NA</td></loq></td></loq>	<.00	NA	<loq (0.0683="" g)<="" td="" ug=""><td> ⊲.00</td><td>NA</td></loq>	⊲.00	NA
QC	Bird001-Loon Lvr-MS	28%			54%	1 .ms	<u></u>
250 ng/g	Bird001-Loon Lvr-MSD	28%	28%	3%	65%	60%	18%
	Bird023-Albetross Kday-MS	71%			63%		1070
	Bird023-Albetross Kday-MSD	87%	79%	20%	77%	70%	20%
	Bird030-Connorant Yelk-MS	121%			71%	- ///	207
	Bird030-Connorant Yolk-MSD	134%	128%	10%	75%	73%	5%
Liver	Bird001-Loom	0.345			<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird002-Loom	0.689			<loo (0.0683="" g)<="" td="" ug=""><td></td><td></td></loo>		
	Bird003-Loon	0.185	ł		<loq (0.0683="" g)<="" td="" ug=""><td></td><td>-</td></loq>		-
	Bird004-Loon	0.199	- · ·		<loq (0.0613="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird005-Loop	0.202			<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird006-Loom	0,105			<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
1	Bird007-Loom	<loq (0.0348="" g)<="" td="" ug=""><td></td><td>73.6</td><td></td><td>1 1</td><td></td></loq>		73.6		1 1	
1	Bird008-Loos	<loq (0.0348="" g)<="" td="" ug=""><td>0.287 - 2 Outliers</td><td>0.212</td><td></td><td>ممه</td><td></td></loq>	0.287 - 2 Outliers	0.212		ممه	
Liver	Bird009-Brown Polican	0.0460	where a contrary	103	<loq (0.0683="" g)<="" td="" ug=""><td></td><td>NA</td></loq>		NA
	Bird010-Brown Pelican	0.294	0.170	0.175	<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
Liver	Bird011-Albairons	0.617	0.170	u.1/5	<loq (0.0683="" g)<="" td="" ug=""><td>₫00</td><td>NA</td></loq>	₫00	NA
Lave	Bird012-Alberton				<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird012-Alberrow	<loq (0.0341="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td>1 1</td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td>1 1</td><td></td></loq>	1 1	
		<loq (0.0341="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq>	1	
	Bird014-Albertons	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
· · · [Bird015-Albeiross	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird016-Albetrons	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird020-Albatrons	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq>	1	
	Bird021-Albaiross	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird022-Albetrons	<loq (0.0348="" g)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.0683="" g)<="" td="" ug=""><td> ∢.00</td><td>NA</td></loq></td></loq>		NA	<loq (0.0683="" g)<="" td="" ug=""><td> ∢.00</td><td>NA</td></loq>	∢ .00	NA
Kidney	Bird017-Albaross	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0613="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0613="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird018-Albeiross	<loq (0.0348="" g)<="" td="" ug=""><td></td><td>· ·</td><td><loq (0.0683="" g)<="" td="" ug=""><td>1.</td><td></td></loq></td></loq>		· ·	<loq (0.0683="" g)<="" td="" ug=""><td>1.</td><td></td></loq>	1.	
	Bird019-Albertons	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird023-Alberros	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq>	1	
	Bird024-Albeiross	<loq (0.0348="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq></td></loq>			<loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq>	1	
	Bird025-Alberros	<loq (0.0348="" g)<="" td="" ug=""><td></td><td>1</td><td><loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>		1	<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird026-Albatreas	<loq (0.0348="" g)<="" td="" ug=""><td><00</td><td>NA</td><td><loq (0.0683="" g)<="" td="" ug=""><td> ⊲.00</td><td>NA</td></loq></td></loq>	<00	NA	<loq (0.0683="" g)<="" td="" ug=""><td> ⊲.00</td><td>NA</td></loq>	⊲.00	NA
Yolk	Bird027-Comparent	<loq (0.0348="" g)<="" td="" ug=""><td>1</td><td></td><td><loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq></td></loq>	1		<loq (0.0683="" g)<="" td="" ug=""><td>1</td><td></td></loq>	1	
	Bird028-Comorant	0.134	1.		<loq (0.0683="" g)<="" td="" ug=""><td></td><td>•</td></loq>		•
	Bird029-Comorant	0.317	1 ·	39.5	<loq (0.0683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird030-Comorant	0.254	0.235 - 1 Outlier	0.0930	<loq (0.0683="" g)<="" td="" ug=""><td>₹.00</td><td>NA ·</td></loq>	₹.00	NA ·
Yolk	Bird031-Gull	0.146			<loq (0.0683="" g)<="" td="" ug=""><td>+</td><td></td></loq>	+	
	Bird032-Guli	<loq (0.0348="" g)<="" td="" ug=""><td></td><td>64.8</td><td><loq (0.0683="" g)<="" td="" ug=""><td>1</td><td>· ·</td></loq></td></loq>		64.8	<loq (0.0683="" g)<="" td="" ug=""><td>1</td><td>· ·</td></loq>	1	· ·
	Bird033-Gull	0.0541	0.0999 - 1 Outlier	0.0648	<loq (0.0683="" g)<="" td="" ug=""><td><.00</td><td>NA</td></loq>	<.00	NA
FOS qualitative confirmation	a performed. Identifications are preliminary.		1	PFOS - Perfluorooct		Lind	
- Limit of Quantitation	· · · · · · · · · · · · · · · · · · ·	NA = Not Applicable		PFHS = Perfluerohez			
- Relative Standard Deviat	ion			POAA = Perfluoroec			
= Relative Percent Differen				PFOSA = Pethorocci			
	723/99 LAC, 12/1/99 GML, 12/30/99 MMH			TTUSA = PELNOTOCC	une sullonamide		
	17/00 MMH						

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Study: Product Number(Test Substance): Matrix: Mariu: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

LIVER, KIDNEY, YOLK

GEN024 MSU Environmental Samples Various Various ETS-8-6.0 & ETS-8-7.0 using unextracted curves ETS-6-0.0 & 215-6-7.0 Using unextinuous (Ametia 052498 Massiyux 3.3 10/12/99 SAL/KK 10/14/99, 12/13/99 HOJ/AS 10/15/99, 11/15/99, 12/14/99 HOJ/AMH

Group Dose	Sample #	Concentration of POAA	Mean POAA	RSD Std. Dev.	Concentration	Mean	RSD
		vy/g or % Rec.			of PFOSA	PFOSA	Std. Dev.
Method Bilk	Bird10129-wblk-5-1			MS/MSD RPD	wg/g or % Rec.	ug/g	MS/MSD RPI
Mediod Dat	Bird10129-wblk-6-1	<loq (0.110="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
QC	Bird001-Loos Lvr-MS	<loq (0.190="" g)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.00750="" g)<="" td="" ug=""><td>00</td><td>NA</td></loq></td></loq>		NA	<loq (0.00750="" g)<="" td="" ug=""><td>00</td><td>NA</td></loq>	00	NA
250 ng/g	Bird001-Loon Lvr-MSD	101%			53%		••
200 100		108%	104%	6%	51%	• 52%	4%
	Bird023-Albetross Kday-MS	113%			56%		
	Bird023-Albetross Kday-MSD	121%	E17%	7%	58%	57%	5%
	Bird030-Cormorant Yolk-MS	105%			62%		
	Bird030-Consorant Yolk-MSD	130%	117%	22%	72%	67%	15%
Liver	Bird001-Loon	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td>0.0153</td><td></td><td></td></loq>			0.0153		
	Bird002-Loon	<loq (0.180="" g)<="" td="" ug=""><td>1 1</td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>	1 1		<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird003-Loon	<loq (0.110="" g)<="" td="" wg=""><td>1</td><td></td><td>0.0147</td><td></td><td></td></loq>	1		0.0147		
	Bird004-Loos	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td>0.0262</td><td>1 1</td><td></td></loq>			0.0262	1 1	
	Bird005-Loon	<loq (0.180="" g)<="" td="" ug=""><td>-</td><td></td><td>0.0213</td><td>[</td><td></td></loq>	-		0.0213	[
	Bird006-Loon	<loq (0.110="" g)<="" m="" td=""><td></td><td></td><td>0.0242</td><td>1 1</td><td><u>t</u></td></loq>			0.0242	1 1	<u>t</u>
	Bird007-Loon	<loq (0.180="" g)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.00750="" g)<="" td="" ug=""><td>1</td><td>25.5</td></loq></td></loq>		NA	<loq (0.00750="" g)<="" td="" ug=""><td>1</td><td>25.5</td></loq>	1	25.5
	Bird008-Loon	<loq (0.100="" g)<="" td="" ug=""><td><100</td><td>NA</td><td><loq (0.00750="" g)<="" td="" ug=""><td>0.0204 - 3 Outliers</td><td>0.00520</td></loq></td></loq>	<100	NA	<loq (0.00750="" g)<="" td="" ug=""><td>0.0204 - 3 Outliers</td><td>0.00520</td></loq>	0.0204 - 3 Outliers	0.00520
Liver	Bard009-Brown Pelicas	4.0Q (0.180 wp/g)		NA	<loq (0.00750="" g)<="" td="" µg=""><td></td><td>0.0020</td></loq>		0.0020
	Bird010-Brown Pelican	<loq (0.180="" g)<="" td="" up=""><td><100</td><td>NA</td><td>0.178</td><td></td><td></td></loq>	<100	NA	0.178		
Liver	Bird011-Albetross	<loq (0.180="" g)<="" td="" ug=""><td></td><td>114</td><td></td><td>0.178 - 1 Outlier</td><td>NA</td></loq>		114		0.178 - 1 Outlier	NA
:	Bird012-Albetrose	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td>0.527</td><td></td><td></td></loq>			0.527		
	Bird0i 3-Albutrons				<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird014-Alberton				<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird014-Albetroes Bird015-Albetroes	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
•	Birdot 5-Albeiros				<loq (0.00750="" g)<="" td="" ug=""><td></td><td>5</td></loq>		5
•	Bird020-Albetrom	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
		<loq (0.180="" g)<="" td="" up=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird021-Albetroes	<loq (0.180="" g)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>		NA	<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird022-Alberross	0.182	< LOQ - 1 Outlier	NA	<loq (0.00750="" g)<="" td="" ug=""><td><0Q </td><td>NA</td></loq>	<0Q	NA
Kidney	Bird017-Albetross	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird018-Albetrons	<loq (0.180="" g)<="" mg="" td=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td>ыÇ</td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td></td><td>ыÇ</td></loq>		ыÇ
	Bird019-Albetross	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td>· ·</td><td>N 1.2</td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td>· ·</td><td>N 1.2</td></loq>	· ·	N 1.2
	Bird023-Alberross	<loq (0.150="" g)<="" td="" ug=""><td>1 1</td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>	1 1		<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird024-Albetross	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird025-Albatross	<loq (0.130="" g)<="" td="" ug=""><td>1</td><td>NA</td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>	1	NA	<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird026-Albetrose	<loq (0.180="" g)<="" td="" ug=""><td><l00< td=""><td>NA</td><td><loq (0.00750="" s)<="" td="" ug=""><td>₹.00</td><td>·····NA</td></loq></td></l00<></td></loq>	<l00< td=""><td>NA</td><td><loq (0.00750="" s)<="" td="" ug=""><td>₹.00</td><td>·····NA</td></loq></td></l00<>	NA	<loq (0.00750="" s)<="" td="" ug=""><td>₹.00</td><td>·····NA</td></loq>	₹.00	·····NA
Yolk	Bird027-Comorant	<loq (0.180="" g)<="" td="" ug=""><td></td><td></td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird028-Comorant	0.245			<loq (0.00750="" g)<="" td="" ug=""><td>1 .</td><td></td></loq>	1 .	
	Bird029-Comorant	<loq (0.180="" g)<="" td="" ug=""><td></td><td>17.2</td><td><loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq></td></loq>		17.2	<loq (0.00750="" g)<="" td="" ug=""><td></td><td></td></loq>		
	Bird030-Comorant	0.192	0.218-2 outliers	0.0374	<loq (0.00750="" g)<="" td="" ug=""><td><100</td><td>NA</td></loq>	<100	NA
Yolk	Bird031-Guil	0,197			<loq (0.00750="" g)<="" td="" ug=""><td></td><td>N/A</td></loq>		N/A
	Bird032-Gull	<loq (0.180="" g)<="" td="" ug=""><td></td><td>0.528</td><td></td><td></td><td></td></loq>		0.528			
	Bird033-Gull	0.196	0.196 - 1 Outlier	0.328	<loq (0.00750="" g)<="" td="" ug=""><td>1</td><td></td></loq>	1	
PROS andianing and	rmation performed. Identifications are preliminary.		V120-1 (Valler	0.00104	<loq (0.00750="" g)<="" td="" ug=""><td><1.0Q</td><td>NA</td></loq>	<1.0Q	NA
0 = Limit of Ouantitati					PFOS = Perfluorooctanesul	baste	

RSD ~ Relative Standard Deviation RSD ~ Relative Standard Deviation RPD = Relative Percent Difference

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

ETS-8-7.0 Excel Version 5/95 .

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POAA = Perfluorocctanoste PFOSA = Pethorooctane sulfor مققه

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WilsonJones - Quick Reference Index System

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

BLOOD

Study:

Greup Dusc	Sample #	Concentration of PFOS ug/mL or % Rec.	Mean PFOS ug/m1,	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS ug/mL or % Rec.	Mean PFHS ng/mL	RSD Std. Dev. MS/MSD RP
Method Bik	MSU12129-H2Obik unfiltered 5-3	<loq (0.00290="" ml)<="" th="" ug=""><th></th><th>NA</th><th><loq (0.00114="" ml)<="" th="" ug=""><th></th><th>NA NA</th></loq></th></loq>		NA	<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>NA NA</th></loq>		NA NA
	MSU12129-H2Oblk filtered 5-4	<loq (0.00290="" ml)<="" td="" ug=""><td>₹.0Q</td><td>NA</td><td><loq (0.00114="" ml)<="" td="" ug=""><td><r00< td=""><td>NA</td></r00<></td></loq></td></loq>	₹.0Q	NA	<loq (0.00114="" ml)<="" td="" ug=""><td><r00< td=""><td>NA</td></r00<></td></loq>	<r00< td=""><td>NA</td></r00<>	NA
Matrix Blk	HMB12129-blood blk-5-1	0.0253			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	HMB12129-blood bik-5-2 HMB12129-blood bik-5-3	0.0248 0.0262			<loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	EMB12129-blood bit-5-4	0.0243			<loq (0.00114="" ml)<br="" ug=""><loq (0.00114="" ml)<="" td="" ug=""><td></td><td></td></loq></loq>		
	ED/B12129-blood bik-5-5	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	HMB12129-blood bit-5-6*	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	HMB12129-blood blk-5-7	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	HMB12129 blood blk-5-8*	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>NA</td></loq>		NA
	HMB12129-blood bit-5-9	<loq (0.00579="" ml)<="" td="" ug=""><td><loq -="" 4="" outliers<="" td=""><td>0.000790</td><td><loq (0.0114="" ml)<="" td="" ug=""><td> ∢.00</td><td>NA</td></loq></td></loq></td></loq>	<loq -="" 4="" outliers<="" td=""><td>0.000790</td><td><loq (0.0114="" ml)<="" td="" ug=""><td> ∢.00</td><td>NA</td></loq></td></loq>	0.000790	<loq (0.0114="" ml)<="" td="" ug=""><td> ∢.00</td><td>NA</td></loq>	∢.00	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 HMB-FE52189-250 MS-5-1-2	-1%	71%	1756	77%	80%	9%
		-176	38%	208%	74%	37%	196%
S. Blood	P205	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>r ,</td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>r ,</td></loq>	1	r ,
Northern Fur Seal	P206	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
Pups	P207	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
•	P208	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td>1</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>		1	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P209	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P210	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	F211	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P212	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P215	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>4.5</td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>4.5</td></loq>		4.5
	P217 P219	<loq (0.00579="" ml)<br="" ug=""><loq (0.00579="" ml)<="" td="" ug=""><td></td><td>ļ</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq></loq>		ļ	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P219	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td>-</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>		-	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P221	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<br="" ug=""><loq (0.0114="" ml)<="" td="" ug=""><td>1 :</td><td></td></loq></loq></td></loq>			<loq (0.0114="" ml)<br="" ug=""><loq (0.0114="" ml)<="" td="" ug=""><td>1 :</td><td></td></loq></loq>	1 :	
	P222	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P223	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>	1		<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P224	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1</td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1</td></loq>	1	1
	P226	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P229	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>NA NA</td></loq></td></loq>		NA	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>NA NA</td></loq>		NA NA
	1230	<loq (0.00579="" ml)<="" td="" ug=""><td><.0Q</td><td>NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td>₫00</td><td>NA</td></loq></td></loq>	<.0Q	NA	<loq (0.0114="" ml)<="" td="" ug=""><td>₫00</td><td>NA</td></loq>	₫00	NA
Blood	M104	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
Northern Fur Seal	M105	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>- 1</td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>- 1</td></loq>		- 1
Adult Females	M106	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1.1.1</td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1.1.1</td></loq>	1	1.1.1
	M107 M112	<loq (0.00579="" ml)<br="" ug=""><loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml.)<="" td="" ug=""><td></td><td>1</td></loq></td></loq></loq>			<loq (0.0114="" ml.)<="" td="" ug=""><td></td><td>1</td></loq>		1
	Milts	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<br="" ug=""><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></loq></td></loq>			<loq (0.0114="" ml)<br="" ug=""><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></loq>		
	M116	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>Į</td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>Į</td></loq>	1	Į
	Mills	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1.</td></loq></td></loq>	1		<loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1.</td></loq>	1	1.
	M119	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>" NA</td></loq></td></loq>		NA	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>" NA</td></loq>		" NA
	M122	<loq (0.00579="" ml)<="" td="" ug=""><td><00⊅</td><td>NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td><00⊳</td><td>- NA</td></loq></td></loq>	<00⊅	NA	<loq (0.0114="" ml)<="" td="" ug=""><td><00⊳</td><td>- NA</td></loq>	<00⊳	- NA
Bleed	S001	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>· ·</td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>· ·</td></loq>		· ·
Northern Fur Seal	\$002	<loq (0.00579="" ml)<="" td="" ug=""><td>ļ</td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>· ·</td></loq></td></loq>	ļ		<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>· ·</td></loq>		· ·
Subadult Males	\$003	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1</td></loq></td></loq>	1		<loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1</td></loq>	1	1
	\$006	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1 .</td></loq></td></loq>	1		<loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>1 .</td></loq>	1	1 .
	\$007	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td></td></loq></td></loq>			<loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td></td></loq>	1	
	5008 5009	<loq (0.00579="" ml)<="" td="" ug=""><td><100</td><td>NA NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td>4.00</td><td>NA NA</td></loq></td></loq>	<100	NA NA	<loq (0.0114="" ml)<="" td="" ug=""><td>4.00</td><td>NA NA</td></loq>	4.00	NA NA
Bleed	P298B	<pre><loq (0.00579="" <="" ml)="" pre="" ug=""><loq (0.00579="" ml)<="" pre="" ug=""></loq></loq></pre>	+ <u> </u>		<loq (0.0114="" ml)<br="" ug=""><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td></td></loq></loq>	1	
Northern Fur Seal	P236A	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.0114="" ml)<br="" ug=""><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>N 2</td></loq></loq></td></loq>	1		<loq (0.0114="" ml)<br="" ug=""><loq (0.0114="" ml)<="" td="" ug=""><td>1</td><td>N 2</td></loq></loq>	1	N 2
	368C	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>	1		<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	P406	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>1</td></loq></td></loq>	1		<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>1</td></loq>		1
1	P411	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td>1</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>1</td></loq></td></loq>	1	1	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>1</td></loq>		1
	94 CUIKB 3*	<loq (0.00579="" ml)<="" td="" ug=""><td></td><td>1 ·</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>		1 ·	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td></td></loq>		
	98 CUICB 7	<loq (0.00579="" ml)<="" td="" ug=""><td>1</td><td>NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td></td><td>NA</td></loq></td></loq>	1	NA	<loq (0.0114="" ml)<="" td="" ug=""><td></td><td>NA</td></loq>		NA
NA2 EDTA	98 CUKB 9	<loq (0.00579="" ml)<="" td="" ug=""><td><10Q</td><td>NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td>4.00</td><td>NA</td></loq></td></loq>	<10Q	NA	<loq (0.0114="" ml)<="" td="" ug=""><td>4.00</td><td>NA</td></loq>	4.00	NA
	ntion, not confirmed			NE - Not Extracted			

FACT-GEN-030

** PFOS NOT confirmed; NS transitions variations >39% Date Entered/By: 01/21/00 LAC

ETS-8-5.1 Excel Version 5/95

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.' B = Lost during extraction PFOSA = Pefluorooctane sulfonamide NA = Not Applicable PFHS = Perfluorobexanesulfonate LOQ = Limit of Quantitation POAA = Perfluorobexanesul

Study: Product Number(Test Substance): Matrix: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Filename: R-Squared Value: R-Squared Value: Slope: Y-Intercept: Dates of Extraction/Analyst: Dates of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

GEN030 MSU Environmental Samples Various Various Blood Various Blood ETS-8-4.1 & ETS-8-5.1 using unextracted curves Soup020199 Massiynx 3.3 See Attachments See Attachments See Attachments See Attachments 12/14/99 SAL/SRP/KK 01/06/00, 01/07/00 MMH/IAS 01/07/00, 01/10/00 LAS/MMH

Г	Group	Sample #	Concentration	Mean	RSD	Cencentration	Menn	RSD
	Dose	•	of POAA	POAA	Std. Dev.	of PFOSA	PFOSA	
			ug/mL or % Rec.	ng/mL	MS/MSD RPD	ug/mL or % Rec.		Std. Dev.
r	Method Blk	MSU12129-H2Oblk unfiltered 5-3	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td>NA</td><td><loq (0.000625="" ml)<="" td="" ug=""><td>eg/mL</td><td>MS/MSD R</td></loq></td></loq>		NA	<loq (0.000625="" ml)<="" td="" ug=""><td>eg/mL</td><td>MS/MSD R</td></loq>	eg/mL	MS/MSD R
		MSU12129-H2Obik filtered 5-4	<loq (0.00240="" ml)<="" td="" ug=""><td><00></td><td>NA</td><td><loq (0.000625="" ml)<="" td="" ug=""><td>100</td><td>NA</td></loq></td></loq>	<00>	NA	<loq (0.000625="" ml)<="" td="" ug=""><td>100</td><td>NA</td></loq>	100	NA
	Matrix Blk	HMB12129-blood bits-5-1	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td>0.00195</td><td>1000</td><td>NA</td></loq>			0.00195	1000	NA
		HMB12129-blood bilk-5-2	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td>0.00190</td><td></td><td>]</td></loq>			0.00190]
Į		HMB12129-blood blk-5-3	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td>0.00225</td><td>1</td><td></td></loq>			0.00225	1	
		HMB12129-blood blk-5-4	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td>0.00189</td><td>1</td><td>i</td></loq>			0.00189	1	i
		HMB12129-blood blk-5-5	<loq (0.00958="" mg="" ml)<="" td=""><td></td><td></td><td>1</td><td></td><td>1</td></loq>			1		1
		HMB12129-blood bik-5-6*	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>ł. i</td><td></td><td></td></loq>			ł. i		
		HMB12129-blogd blk-5-7	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>i i</td><td></td><td></td></loq>			i i		
		HMB12129-blood bik-5-8	<loq (0.00958="" ml)<="" td="" us=""><td></td><td>NA</td><td>l i</td><td></td><td>NA</td></loq>		NA	l i		NA
		HMB12129-blood bits-5-9	<loq (0.00958="" ml)<="" td="" ug=""><td><00⊳</td><td>NA</td><td>l i</td><td>1</td><td>NA</td></loq>	<00⊳	NA	l i	1	NA
- F	<u>QC</u>	PBB-6255-250MS-5-1-2	76%			50%	<u> </u>	<u> </u>
		PBB-6255-250MSD-5-1-2	75%	76%	1%	47%	49%	6%
	Г	FSB-S009-250ppb MS-5-1-2	0%			1		
		FSB-S009-250ppb MSID-5-1-2	1%	1%	36%	i	1	NA
	F	SSB-SSL49-250ppb MS-5-1-2	76%	1		i	<u>+</u>	
1		SSB-SSL49-250ppb MSD-5-1-2	71%	74%	7%	i	I	NA
	· · · · · ·	HMB-FE52189-250 MS-5-1-2	1%	1		<u>i</u>	+	NA.
L		HD4B-FE52189-250 MSD-5-1-2	70%	35%	195%		I I	
F	Bleed	1205	<loq (0.00958="" ml)<="" td="" ug=""><td>3376</td><td>13374</td><td><u> </u></td><td><u> </u></td><td>NA</td></loq>	3376	13374	<u> </u>	<u> </u>	NA
	Northern Fur Seal	P206	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>I I</td><td></td><td></td></loq>			I I		
1	Pups	207	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td></td><td>1 t</td><td></td><td></td></loq>	1		1 t		
		P206	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td></td><td></td><td></td></loq>					
		P209	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>1 :</td><td></td><td>1</td></loq>			1 :		1
		P210		1			1	
		P210	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td></td><td></td><td></td><td></td></loq>	1				
1			<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td></td><td>1</td><td></td></loq>				1	
		P212	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>1</td><td></td><td></td></loq>			1		
		P215	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td></td><td></td><td></td></loq>					
		P217	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td>1</td><td></td><td></td><td></td></loq>		1			
		P219	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>1 1</td><td></td><td>1 .</td></loq>			1 1		1 .
		P220	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td></td><td>I I.</td><td></td><td></td></loq>	1		I I.		
		P221	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>1 I.</td><td></td><td></td></loq>			1 I.		
		P222	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>1</td><td></td><td></td></loq>			1		
1		P223	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td></td><td></td><td>1</td></loq>					1
		P224	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td></td><td>1 I</td><td></td><td></td></loq>	1		1 I		
1		P226	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>T</td><td></td><td>1</td></loq>			T		1
		P229	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td>NA</td><td>1</td><td></td><td>NA</td></loq>		NA	1		NA
L		P230	<loq (0.00958="" ml)<="" td="" ug=""><td>₹00</td><td>NA</td><td>I</td><td>I</td><td>NA NA</td></loq>	₹00	NA	I	I	NA NA
	Bleed	M104	<loq (0.00958="" ml)<="" td="" ug=""><td>Υ</td><td></td><td>I</td><td></td><td></td></loq>	Υ		I		
	Northern Fur Seal	M105	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>I</td><td></td><td></td></loq>			I		
	Adult Females	M106	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>I</td><td></td><td>ł</td></loq>			I		ł
		M107	<loq (0.00958="" ml)<="" ng="" td=""><td></td><td></td><td>1</td><td></td><td>1</td></loq>			1		1
	1	M112	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td>1</td><td>1</td><td>1</td><td>1</td></loq>	1	1	1	1	1
I		M115	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td>1</td><td>1</td><td>1</td><td>1</td></loq>	1	1	1	1	1
1	1	M116	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>i i</td><td>1</td><td></td></loq>			i i	1	
1		M118	<loq (0.00958="" mg="" ml)<="" td=""><td>1</td><td></td><td>i i</td><td>1</td><td></td></loq>	1		i i	1	
	1	MI 19	<loq (0.00958="" ml)<="" td="" up=""><td>1</td><td>NA</td><td>i i</td><td>1</td><td>NA</td></loq>	1	NA	i i	1	NA
1		M122	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td>NA</td><td>i</td><td>1 1</td><td>NA</td></loq>		NA	i	1 1	NA
- 1	Bleed	\$001	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td>1</td><td>1</td><td>+</td><td></td></loq>		1	1	+	
	Northern Pur Seal	S002	<loq (0.00958="" ml)<="" td="" us=""><td>1</td><td></td><td>i i</td><td>1</td><td>1</td></loq>	1		i i	1	1
	Subscuit Males	S003	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td>1</td><td>1 i</td><td>1</td><td>1</td></loq>	1	1	1 i	1	1
	1	S006	<loq (0.00958="" ml)<="" td="" up=""><td>1</td><td></td><td></td><td></td><td></td></loq>	1				
	1	\$007	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td></td><td></td><td>1</td><td>1</td></loq>	1			1	1
-		5008	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td>NA</td><td></td><td></td><td>NA</td></loq>	1	NA			NA
		5009	<loq (0.00958="" ml)<="" td="" ug=""><td>1 4.00</td><td>NA</td><td>1 1</td><td>1</td><td>NA</td></loq>	1 4.00	NA	1 1	1	NA
t	Blood	P2918	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td></td><td>+ ••••</td><td></td></loq>				+ ••••	
	Northern Fur Seal	P236A	<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td>1</td><td></td><td></td><td></td></loq>	1	1			
		3680	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td>1</td><td>1</td><td>1</td><td></td></loq>		1	1	1	
		P406						1
1		P411	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td></td><td></td><td></td></loq>					
			<loq (0.00958="" ml)<="" td="" ug=""><td>1</td><td></td><td>I</td><td>1</td><td>1</td></loq>	1		I	1	1
		98 CUKB 3*	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td>· · …</td><td></td><td>1</td><td>1</td></loq>		· · …		1	1
ļ		98 CUKB 7	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td>NA</td><td>1</td><td>1</td><td>NA</td></loq>		NA	1	1	NA
	NA2 EDTA	98 CUKB 9	<loq (0.00958="" ml)<="" td="" ug=""><td>QQ</td><td>NA</td><td><u> </u></td><td>I</td><td>NA</td></loq>	QQ	NA	<u> </u>	I	NA
	* Surrogate >50% deviation, no			NE = Not		PFOS - Perfluoroectanceul		
		ss for PFOSA, interferent present in both analyses i transitions variation > 30%	L.		luring extraction	PFOSA = Pefluorooctane si		•
					Applicable	PFHS = Perfluorohexaneru		

BTS-8-5.1 Excel Version 5/95

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Study:
Product Number(Test Substance):
Matrix
Method/Revision:
Analytical Equipment System Number:
Lastrument Software/Version:
Fileneme:
R-Squared Value:
Slope:
Y-intercent:
Dates of Extraction/Analyst:
Dates of Analysis/Analyst:
Date of Data Reduction/Analyst:
•
Sample Data

GEN030 MSU Environmental Samples Various Various Blood BTS-8-4.1 & BTS-8-5.1 using unextracted curves Soup020199 Masslynx 3.3 See Attachments See Attachments See Attachments See Attachments See Attachments 12/14/99 SAL/SRP/KKK 01/06/00, 01/07/00 MMH/IAS 01/07/00, 01/10/00 IAS/MMH

BLOOD

ſ	Group	Sample #	PFOS	Concentration	Mean	RSD	Concentration	Mean	RSD
1	Deer		Verified	of PFOS	PFOS	Std. Dev.	of PFHS	PFHS	Std. Dev.
				ng/mL or % Rec.	mg/msL	MS/MSD RPD	ug/mL or % Rec.	eg/mĽ	MS/MSD RPD
	Method Bik	MSU12129-H2Obik unfiltered 5-3	NA	<loq (0.00290="" ml)<="" th="" ug=""><th></th><th>NA</th><th><loq (0.00114="" ml)<="" th="" ug=""><th></th><th> NA</th></loq></th></loq>		NA	<loq (0.00114="" ml)<="" th="" ug=""><th></th><th> NA</th></loq>		NA
		MSU12129-H2Oblk filtered 5-4	NA	<loq (0.00290="" ml)<="" th="" ug=""><th>⊲.0Q</th><th>NA</th><th><loq (0.00114="" ml)<="" th="" ug=""><th><1.0Q</th><th>NA</th></loq></th></loq>	⊲.0Q	NA	<loq (0.00114="" ml)<="" th="" ug=""><th><1.0Q</th><th>NA</th></loq>	<1.0Q	NA
	Matrix Blk	HMB12129-blood blk-5-1	NA	0.0253			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
	l l l l l l l l l l l l l l l l l l l	HMB12129-blood blk-5-2	NA	0.0248			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
- 1		HIMB12129-blood bik-5-3	NA	0.0262			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
- 1		HMB12129-blood blk-5-4	NA	0.0243			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
		HMB12129-blood bik-5-5	NA	<loq (0.0579="" ml)<="" th="" ug=""><th></th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq></th></loq>			<loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
		HMB12129-blood blk-5-6*	NA	<loq (0.0579="" ml)<="" th="" ug=""><th></th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq></th></loq>			<loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
		HMB12129-blood bik-5-7	NA	<loq (0.0579="" ml)<="" th="" ug=""><th></th><th></th><th><loq (0.0114="" mi.)<="" th="" ug=""><th></th><th></th></loq></th></loq>			<loq (0.0114="" mi.)<="" th="" ug=""><th></th><th></th></loq>		
		HMB12129-blood bik-S-E	NA	<loq (0.0579="" ml)<="" th="" ug=""><th></th><th>NA</th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th>NA</th></loq></th></loq>		NA	<loq (0.0114="" ml)<="" th="" ug=""><th></th><th>NA</th></loq>		NA
		HMB12129-blood blk-5-9	NA	<loq (0.0579="" ml)<="" th="" ug=""><th><loq -="" 4="" outliers<="" th=""><th>0.000790</th><th><loq (0.0114="" ml)<="" th="" ug=""><th><00 ·</th><th>NA NA</th></loq></th></loq></th></loq>	<loq -="" 4="" outliers<="" th=""><th>0.000790</th><th><loq (0.0114="" ml)<="" th="" ug=""><th><00 ·</th><th>NA NA</th></loq></th></loq>	0.000790	<loq (0.0114="" ml)<="" th="" ug=""><th><00 ·</th><th>NA NA</th></loq>	<00 ·	NA NA
	ÓC.	PBB-6255-250MS-5-1-2	NA	74%			76%		
		PBB-6255-250MSD-5-1-2	NA	91%	\$2%	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%		
	4	HMB-FE52189-250 MSD-5-1-2	NA	77%	38%	208%	74%	37%	196%
	Bieod	6255 (Heparin)	x	0.0518			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>1</th></loq>		1
	Polar Bear	20436 (NA2 EDTA)	X	0.0001			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
		20467 (NA2 EDTA)	· x	0.0358			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>· ·</th></loq>		· ·
		20468 (EDTA)	x	0.0315			<loq (0.00114="" ml)<="" th="" wg=""><th></th><th></th></loq>		
		20470	,`X	0.0281			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>17</th></loq>		17
		20472	X	0.0327			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
		20473*	×	0.0309			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>· ·</th></loq>		· ·
		20474 (NA2 EDTA)	x	0.0256			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
		20475 (NA2 EDTA)	X	<loq (0.00290="" ml)<="" th="" ug=""><th></th><th></th><th><loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq></th></loq>			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
	1	20475 (NA2 EDTA)	x	0.0345			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>-</th></loq>		-
		20477	x	0.0272			<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>-</th></loq>		-
		20485 (Heparin)	x	0.0429	1		<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>-</th></loq>		-
		20486 (Heperia)	x	0.0316		0.214	<loq (0.00114="" ml)<="" th="" ug=""><th></th><th>NA</th></loq>		NA
	·	20487 (Heparia)	X	<loq (0.00290="" .<="" ml)="" th="" ug=""><th>0.0342 - 2 outliers</th><th>0.00731</th><th><loq (0.00114="" ml)<="" th="" ug=""><th><10Q</th><th>NA</th></loq></th></loq>	0.0342 - 2 outliers	0.00731	<loq (0.00114="" ml)<="" th="" ug=""><th><10Q</th><th>NA</th></loq>	<10Q	NA
	Bloed	SSL49 (7.2 mg K2 EDTA)	NA	<loq (0.00579="" ml)<="" th="" ug=""><th></th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq></th></loq>			<loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
	Stellar Sealion	SSL50 (7.2 mg K2 EDTA)	NA	<loq (0.00579="" ml)<="" th="" vg=""><th></th><th>i</th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th>1</th></loq></th></loq>		i	<loq (0.0114="" ml)<="" th="" ug=""><th></th><th>1</th></loq>		1
	1	SSL51 (7.2 mg K2 EDTA)	NA	<loq (0.00579="" ml)<="" th="" ug=""><th></th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq></th></loq>			<loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
		SSL52 (7.2 mg K2 EDTA)	, NA	<loq (0.00579="" ml.)<="" th="" ug=""><th></th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th>1</th></loq></th></loq>			<loq (0.0114="" ml)<="" th="" ug=""><th></th><th>1</th></loq>		1
		SSL53 (7.2 mg K2 EDTA)	NA	<loq (0.00579="" ml)<="" th="" ug=""><th></th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq></th></loq>			<loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
		SSL54 (7.2 mg K2 EDTA)	NA	<loq (0.00579="" ml)<="" th="" ug=""><th>ł</th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th>1 :</th></loq></th></loq>	ł		<loq (0.0114="" ml)<="" th="" ug=""><th></th><th>1 :</th></loq>		1 :
	1	SSL55 (5.4 mg K2 EDTA)	NA	<loq (0.00579="" ml)<="" th="" ug=""><th>1</th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th>1.</th></loq></th></loq>	1		<loq (0.0114="" ml)<="" th="" ug=""><th></th><th>1.</th></loq>		1.
	1	SSL56 (5.4 mg K2 EDTA)**	NA	<loq (0.00579="" ml)<="" th="" ug=""><th>1</th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq></th></loq>	1		<loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
	1	SSL57 (5.4 mg K2 EDTA)	NA	<loq (0.00579="" ml)<="" th="" ug=""><th>1</th><th>1</th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th>·]</th></loq></th></loq>	1	1	<loq (0.0114="" ml)<="" th="" ug=""><th></th><th>·]</th></loq>		·]
		SSLSE (5.4 mg K2 EDTA)*	NA	<loq (0.00579="" ml)<="" th="" ug=""><th>1</th><th></th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq></th></loq>	1		<loq (0.0114="" ml)<="" th="" ug=""><th></th><th></th></loq>		
	1	SSL59 (5.4 mg K2 EDTA)	NA	<loq (0.00579="" ml)<="" th="" ug=""><th></th><th>NA</th><th><loq (0.0114="" ml)<="" th="" ug=""><th></th><th>NA</th></loq></th></loq>		NA	<loq (0.0114="" ml)<="" th="" ug=""><th></th><th>NA</th></loq>		NA
		SSL60 (5.4 mg K2 EDTA)*	NA	<loq (0.00579="" ml)<="" td="" ug=""><td>4.00</td><td>NA</td><td><loq (0.0114="" ml)<="" td="" ug=""><td>QQ</td><td>NA</td></loq></td></loq>	4.00	NA	<loq (0.0114="" ml)<="" td="" ug=""><td>QQ</td><td>NA</td></loq>	QQ	NA

 Image: SSL60 (5.4 mg IZ EDTA)*

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

 ** Surrogate >50% deviation 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 kph

NE - Not Extracted B = Lost during extraction NA = Not Applicable NV = Not Verified LOQ - Limit of quantitation

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PFOS = Perfluorooctanesulfonnie PFOSA = Perfluorooctane sulfonnie PFHS = Perfluorohexanesulfonate POAA = Perfluorooctanoete

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ETS-8-5.1 Excel Version 5/95

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GEN-030-sera als

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Study: Product Number(Test Substance): Matrix: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Filename: R-Squared Value: Slope: Y-Intercept: Dates of Extraction/Analyst: Dates of Analysis/Analyst: Dates of Analysis/Analyst: Date of Data Reduction/Analyst:

GEN030 MSU Environmental Samples Various Various Blood ETS-8-4.1 & ETS-8-5.1 using unextracted-curves Soup020199 Masslynx 3.3 See Attachments See Attachments See Attachments See Attachments 12/14/99 SAL/SRP/KKK 01/06/00, 01/07/00 MMH/LAS 01/07/00, 01/10/00 LAS/MMH

Sample Data BLOOD

	Group	Sample #	Concentration	Mean	RSD	Concentration	Mean	RSD
	Dose		+FPOAA	POAA	Std. Dev.	of PTOSA	PTOSA	Std. Dev.
			ug/mL or % Rec.	eg/mL	MS/MSD RPD	ug/mL or % Rec.	eg/mL	MS/MSD RPD
h	Method Blk	MSU12129-H2Obik unfiltered 5-3	<loq (0.00240="" ml)<="" td="" up=""><td></td><td>NA</td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.000625="" ml)<="" td="" ug=""><td></td><td>NA</td></loq>		NA
		MSU12129-H2Oblk filtered 5-4	<loq (0.00240="" ml)<="" td="" ug=""><td><l0q< td=""><td>NA</td><td><loq (0.000625="" ml)<="" td="" ug=""><td><.00</td><td>' NA</td></loq></td></l0q<></td></loq>	<l0q< td=""><td>NA</td><td><loq (0.000625="" ml)<="" td="" ug=""><td><.00</td><td>' NA</td></loq></td></l0q<>	NA	<loq (0.000625="" ml)<="" td="" ug=""><td><.00</td><td>' NA</td></loq>	<.00	' NA
1	Matrix Blk	HIMB12129-blood bik-5-1	<loq (0.00240="" ml)<="" td="" w=""><td>ľ</td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td><u>_</u></td><td></td></loq></td></loq>	ľ		<loq (0.000625="" ml)<="" td="" ug=""><td><u>_</u></td><td></td></loq>	<u>_</u>	
		HMB12129-blood blk-5-2	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
		HMB12129-blood blk-5-3	<loq (0.00240="" ml)<="" td="" ug=""><td>1</td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td>•</td></loq></td></loq>	1		<loq (0.000625="" ml)<="" td="" ug=""><td></td><td>•</td></loq>		•
		HMB12129-blood blk-5-4	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td>••</td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td>••</td></loq>		••
		HMB12129-blood blk-5-5	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>I</td><td></td><td>•</td></loq>			I		•
		HMB12129-blood blk-5-6*	<loq (0.00951="" mg="" ml)<="" td=""><td></td><td></td><td>1 1</td><td>1</td><td></td></loq>			1 1	1	
		HMB12129-blood blk-5-7	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td> I </td><td></td><td></td></loq>			I		
		HMB12129-blood blk-5-5	<loq (0.00958="" ml)<="" td="" up=""><td></td><td>NA</td><td>I</td><td></td><td>NA</td></loq>		NA	I		NA
		HIMB12129-blood blk-5-9	<loq (0.00958="" mg="" ml)<="" td=""><td>40Q</td><td>NA</td><td>I I</td><td>I</td><td>NA</td></loq>	40Q	NA	I 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	NA
		SSB-SSL49-250ppb MS-5-1-2	76%			I		
•		SSB-SSL49-250ppb MSD-5-1-2	71%	74%	7%	I I	I	NA
		HMB-FE52189-250 MS-5-1-2	1%			I		
	÷	HMB-FE52189-250 MSD-5-1-2	70%	35%	195%	I I	1	NA
3	Bleed	6255 (Hoperia)	<loq (0.00240="" ml)<="" td="" up=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
4 0	Polar Bear	20436 (NA2 EDTA)	<loq (0.00248="" ml)<="" td="" up=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td>2</td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td>2</td></loq>		2
		20467 (NA2 EDTA)	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.000625="" m="" ml)<="" td=""><td></td><td>· ·</td></loq></td></loq>			<loq (0.000625="" m="" ml)<="" td=""><td></td><td>· ·</td></loq>		· ·
		20468 (EDTA)	<loo (0.00240="" ml)<="" td="" um=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loo>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
		20470	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
		20472	<loq (0.00240="" ml)<="" td="" w=""><td></td><td></td><td><loq (0.000625="" ag="" ml)<="" td=""><td></td><td>•</td></loq></td></loq>			<loq (0.000625="" ag="" ml)<="" td=""><td></td><td>•</td></loq>		•
		20473*	<loq (0.00240="" ml)<="" td="" w=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td>-</td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td>-</td></loq>		-
		20474 (NA2 EDTA)	<loq (0.00240="" ml)<="" td="" w=""><td>1</td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td>_</td></loq></td></loq>	1		<loq (0.000625="" ml)<="" td="" ug=""><td></td><td>_</td></loq>		_
	1	20475 (NA2 EDTA)	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td>••</td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td>••</td></loq>		••
		20476 (NA2 EDTA)	<loo (0.00240="" ml)<="" td="" us=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loo>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
		20477	<loq (0.00240="" ml)<="" td="" ug=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" us=""><td></td><td></td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" us=""><td></td><td></td></loq>		
		20485 (Heparin)	<loq (0.00240="" ml)<="" td="" up=""><td></td><td></td><td><loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq></td></loq>			<loq (0.000625="" ml)<="" td="" ug=""><td></td><td></td></loq>		
		20486 (Heparin)	<loq (0.00240="" m="" ml)<="" td=""><td></td><td>NA</td><td><loq (0.000625="" ml)<="" td="" ug=""><td>-</td><td>NA</td></loq></td></loq>		NA	<loq (0.000625="" ml)<="" td="" ug=""><td>-</td><td>NA</td></loq>	-	NA
		20487 (Heparin)	<loq (0.00240="" ml)<="" td="" up=""><td>⊲.00</td><td>NA</td><td><loq (0.000625="" ml)<="" td="" ug=""><td>4.00</td><td>NA</td></loq></td></loq>	⊲.00	NA	<loq (0.000625="" ml)<="" td="" ug=""><td>4.00</td><td>NA</td></loq>	4.00	NA
	Blood	SSL49 (7.2 mg K2 EDTA)	<loq (0.00958="" m="" ml)<="" td=""><td></td><td></td><td></td><td>~~~~~</td><td></td></loq>				~~~~~	
S	tellar Sealion	SSL50 (7.2 mg K2 EDTA)	<loq (0.00958="" ml)<="" td="" up=""><td></td><td></td><td></td><td></td><td></td></loq>					
		SSL51 (7.2 mg K2 EDTA)	<loo (0.00958="" ml)<="" td="" up=""><td></td><td></td><td>1</td><td></td><td>•</td></loo>			1		•
		SSL52 (7.2 mg K2 EDTA)	<loq (0.00958="" ml)<="" td="" ug=""><td></td><td></td><td>1 î</td><td></td><td></td></loq>			1 î		
		SSL53 (7.2 mg K2 EDTA)	<loo (0.00958="" ml)<="" td="" up=""><td></td><td></td><td>1 i</td><td></td><td></td></loo>			1 i		
		SSL54 (7.2 mg K2 EDTA)	<loq (0.00958="" ml)<="" td="" up=""><td></td><td></td><td>· · ·</td><td></td><td>1</td></loq>			· · ·		1
		SSL55 (5.4 mg K2 EDTA)	<loq (0.00958="" ml)<="" td="" up=""><td></td><td></td><td></td><td></td><td></td></loq>					
		SSL56 (5.4 mg K2 EDTA)**	<loq (0.00958="" ml)<="" td="" up=""><td></td><td></td><td>1</td><td></td><td></td></loq>			1		
		SSL57 (5.4 mg K2 EDTA)	<loq (0.00958="" ml)<="" td="" w=""><td></td><td></td><td></td><td></td><td></td></loq>					
		SSL58 (5.4 mg K2 EDTA)*	<loq (0.00958="" ml)<="" td="" up=""><td></td><td></td><td></td><td></td><td>1</td></loq>					1
		SSL59 (5.4 mg K2 EDTA)	<loq (0.00958="" ml)<="" td="" w=""><td></td><td>NA</td><td></td><td></td><td> N2</td></loq>		NA			N2
		SSL60 (5.4 mg K2 EDTA)*	<loq (0.00958="" m="" ml)<="" td=""><td>₹00</td><td>NA NA</td><td> ;</td><td></td><td>NA</td></loq>	₹00	NA NA	;		NA
		FOSA, interferent present in both malv		yuur	A71			NA

I = May need to rerun all samples for PFOSA, interferent p ** Surrogane >50% deviation confirmed * Surrogane >50% deviation, not confirmed ** PFOS NOT confirmed; MS transitions variation > 30% Date Entered/By: Date Verified/ By: 1/13/00, 01/21/00 MMH/LAC

02/10/00 kjh

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

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PPOS = Perfluorooc PPOSA = Perfuorooctume sulfonamide PFHS = Perfuorohexanesulfonate

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POAA - Perfluoroco te.

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BTS-8-5.1 **Bacel Version 5/95**

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WilsonJones - Quick Reformed Index System

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FACT-GEN-030 MSU Environmental Samples

Study: Product Number(Test Substance): Matrix: Method/Revision: Analytical Equipment System Number-Instrument Software/Version: Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

GEN030 MSU Environmental Samples Various Various Vanous ETS-8-6.0 & ETS-8-7.0 Soup 020199, Amelia 062498 Massiynx 3.3 12/12/99 SAL/KK/SRP/CSH 01/10/00 MMH

MUSCLE

Group	Sample #	PFOS	Concentration	Mesu	RSD	Concentration	Mean	RSD
17036		Vertilied	of PPOS	PFOS	Std. Dev.	of PFHS	PFHS	Std. Dev.
Method Blk		N1.4	ug/g or % Rec.	ur/r	MS/MSD RPD	ug/g or % Rec.	• g/ g	MS/MSD RPI
Unfiltered water	MSU12149-H2Ounfil 5-1* MSU12149-H2Ounfil 5-2*	NA	0.0163		NA	<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
Method Bik	MSU12149-H2Ofil 5-1*	NA NA	<loq (="" 0.00696)<="" td=""><td>NA</td><td>NA</td><td><loq (="" 0.0342)<="" td=""><td><loq< td=""><td>NA</td></loq<></td></loq></td></loq>	NA	NA	<loq (="" 0.0342)<="" td=""><td><loq< td=""><td>NA</td></loq<></td></loq>	<loq< td=""><td>NA</td></loq<>	NA
Filtered water	MSU12149-H2Ofil 5-2*	NA NA	<loq (="" 0.00696)<="" td=""><td></td><td>NA</td><td><loq (0.0342)<="" td=""><td></td><td>NA .</td></loq></td></loq>		NA	<loq (0.0342)<="" td=""><td></td><td>NA .</td></loq>		NA .
Matrix Blk	MSU12149-H20H1 5-2* MSU12149-Fighbik 5-1	NA NA	0.0257	NA	NA	<loq (="" 0.0342)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
Fish Liver	MSU12149-Fishbik 5-2	NA	0.00948 0.00985			<loq (="" 0.0342)<="" td=""><td></td><td></td></loq>		
TISH LIVE	MSU12149-Fishbik 5-2 MSU12149-Fishbik 5-3*	NA	0.00454	0.00000	37.2	<loq (="" 0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
QC	CPM-BIN7-MS 5-1-2	NA		0.00796	0.00296	<loq (="" 0.0342)<="" td=""><td><1.0Q</td><td>NA</td></loq>	<1.0Q	NA
250 ng/g	CPM-B1N7-MS 5-1-2 CPM-B2N6-MS 5-1-2	NA	59%			60%		
200 MP/E	CSM-1999030-03-01-MS 5-1-2	NA NA	31%	45%	63%	45%	53%	28%
	CSM-1999030-03-01-MS 5-1-2		160%			148%		
	LWM-1999029-16-MS 5-1-2	NA	127%	143%	23%	183%	166%	21%
		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%		
Maria	BTM-1999040-10-MS 5-1-2	NA	166%	157%	11%	168%	1.56%	15%
Muscle	BINI	X	0.101			<loq (="" 0.0342)<="" td=""><td></td><td></td></loq>		
Carp	BIN2	X	0.0784			<loq (="" 0.0342)<="" td=""><td></td><td></td></loq>		
	BIN7	х	0.0905			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
,	BINIO	X	0.0878			<loq (="" 0.0342)<="" td=""><td></td><td></td></loq>		
	B2N2	x	0.105			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	B2N6 B2N8	x	0.0894			<loq (0.0342)<="" td=""><td></td><td>÷</td></loq>		÷
		x	0.0596			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	B2N10	x	0.0838			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	Carp1*	x	0.297		64.2	<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
	Carp2*	x	0.243	0.124	0.0793	<loq (0.0342)<="" td=""><td><1.00</td><td>NA</td></loq>	<1.00	NA
Muscle	1999030-01	NA	0.189			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
Chinook Salmon	1999030-02	NA `	0.126			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	1999030-02-01	x	<loq (="" 0.00696)<="" td=""><td></td><td></td><td><loq (0.0342)<="" td=""><td></td><td>÷</td></loq></td></loq>			<loq (0.0342)<="" td=""><td></td><td>÷</td></loq>		÷
	1999030-02-04	x	0.113			<loq (0.0342)<="" td=""><td></td><td>h.</td></loq>		h.
	1999030-03-01	x	0.0514		0.524	<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
	1999030-03-04	x	0.0573	0.107 - 1 outlier	0.0562	<loo (="" 0.0342)<="" td=""><td><1.00</td><td>NA -</td></loo>	<1.00	NA -
Muscle	1999029-11*	X	0.168			<loo (="" 0.0342)<="" td=""><td></td><td></td></loo>		
Lake Whitefish	1999029-12*	x	0.130			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	1999029-13	x	0.0967			<loq (0.0342)<="" td=""><td></td><td>-</td></loq>		-
	1999029-14	x	0.0983		26.4	<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
	1999029-16	x	0.1659	0.132	0.0348	<loq (="" 0.0342)<="" td=""><td>⊲.00</td><td>NA NA-</td></loq>	⊲.00	NA NA-
Muscie	1999040-01	X	<loq (="" 0.00696)<="" td=""><td></td><td></td><td><loq (0.0342)<="" td=""><td>-2002</td><td></td></loq></td></loq>			<loq (0.0342)<="" td=""><td>-2002</td><td></td></loq>	-2002	
Brown Trout	1999040-02 **	x ••	<loq (="" 0.00696)<="" td=""><td></td><td></td><td><loq (0.0342)<="" td=""><td></td><td>111</td></loq></td></loq>			<loq (0.0342)<="" td=""><td></td><td>111</td></loq>		111
	1999040-03	NA -	<loq (="" 0.00696)<="" td=""><td></td><td></td><td><loq (0.0342)<="" td=""><td>1</td><td></td></loq></td></loq>			<loq (0.0342)<="" td=""><td>1</td><td></td></loq>	1	
	1999040-04	NA	<loq (0.00696)<="" td=""><td></td><td></td><td><loq (="" 0.0342)<="" td=""><td>1</td><td>12</td></loq></td></loq>			<loq (="" 0.0342)<="" td=""><td>1</td><td>12</td></loq>	1	12
	1999040-05	x	<loq (0.00696)<="" td=""><td></td><td></td><td><loq (0.0342)<br=""><loq (0.0342)<="" td=""><td></td><td></td></loq></loq></td></loq>			<loq (0.0342)<br=""><loq (0.0342)<="" td=""><td></td><td></td></loq></loq>		
	1999040-06	x	<loq (="" 0.00696)<="" td=""><td></td><td></td><td><loq (0.0342)<br=""><loq (0.0342)<="" td=""><td>1</td><td></td></loq></loq></td></loq>			<loq (0.0342)<br=""><loq (0.0342)<="" td=""><td>1</td><td></td></loq></loq>	1	
	1999040-07	NA	<loq (0.00696)<="" td=""><td></td><td></td><td></td><td></td><td></td></loq>					
	1999040-08	x	0.0460	1		<loq (0.0342)<="" td=""><td>l</td><td></td></loq>	l	
	1999040-09	NA	<loq (="" 0.00696)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0342)<="" td=""><td>1</td><td>l</td></loq></td></loq>		NA	<loq (="" 0.0342)<="" td=""><td>1</td><td>l</td></loq>	1	l
	1999040-10	NA	<loq (0.00696)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>NA</td><td><loq (0.0342)<="" td=""><td></td><td>NA</td></loq></td></loq></td></loq>	<loq -="" 1="" outlier<="" td=""><td>NA</td><td><loq (0.0342)<="" td=""><td></td><td>NA</td></loq></td></loq>	NA	<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
High (>50%) surrogate			(0.00090)		NA NB = Not Extracted	<loq (="" 0.0342)<="" td=""><td>Q0_</td><td>NA</td></loq>	Q0_	NA

 ruga (>30%) surrogate acviations
 ** 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 B = Lost during extinction NA = Not Applicable LOQ = Limit of Quantitation X = Verified PFOS concentration

PFOSA = Pefluorooctane sulfonamide PFHS = Perfluorohexanesulfonate

POAA = Perfluorooctanoste

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FACT-GEN-030 **MSU Environmental Samples**

Study Product Number(Test Substance) Matrix: Method/Revision: Analytical Equipment System Number Instrument Software/Version Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

GEN030 MSU Environmental Samples GEN030 MSU Environmental Various ETS-8-6.0 & ETS-8-7.0 Soup 020199, Amelia 062498 Massiynx 3.3 12/12/99 SAL/KK/SRP/CSH 01/10/00 MMH 01/12/00 LAS

MUSCLE

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Group Dose	Sample #	Concentration	Mean	RSD	Concentration	Mean	RSD
Dose		of POAA	POAA	Std. Dev.	of PFOSA	PFOSA	Std. Dev.
		ug/g or % Rec.	₩E/E	MS/MSD RPD	IET or % Rec.	===/2	
Method Blk	MSU12149-H2Ounfil 5-1*	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td></td><td>MS/MSD RPD</td></loq></td></loq>		NA	<loq (="" 0.0188)<="" td=""><td></td><td>MS/MSD RPD</td></loq>		MS/MSD RPD
Unfiltered water	MSU12149-H2Ounfil 5-2*	<loq (="" 0.0359)<="" td=""><td><100</td><td>NA</td><td><loq (0.0153)<="" td=""><td>100</td><td>NA</td></loq></td></loq>	<100	NA	<loq (0.0153)<="" td=""><td>100</td><td>NA</td></loq>	100	NA
Method Blk	MSU12149-H2Ofil 5-1*	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<="" td=""><td><1.0Q</td><td>NA</td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td><1.0Q</td><td>NA</td></loq>	<1.0Q	NA
Filtered water	MSU 2149-H2Ofil 5-2*	<loq (="" 0.0359)<="" td=""><td><.00</td><td>NA</td><td><loq (0.0188)<="" td=""><td>100</td><td>, NA</td></loq></td></loq>	<.00	NA	<loq (0.0188)<="" td=""><td>100</td><td>, NA</td></loq>	100	, NA
Matrix Blk	MSU12149-Fishbik 5-1	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td><1.0Q</td><td>NA</td></loq></td></loq>			<loq (0.0188)<="" td=""><td><1.0Q</td><td>NA</td></loq>	<1.0Q	NA
Fish Liver	MSU12149-Fishbik 5-2	<loq (0.0359)<="" td=""><td>1</td><td>NA</td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1	NA	<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	MSU12149-Fishbik 5-3*	<loq (="" 0.0359)<="" td=""><td><.00</td><td>NA</td><td><loq (0.0188)<="" td=""><td><.00</td><td>NA</td></loq></td></loq>	<.00	NA	<loq (0.0188)<="" td=""><td><.00</td><td>NA</td></loq>	<.00	NA
QC	CPM-B1N7-MS 5-1-2	6%			11%	LUQ	NA
250 ng/g	CPM-B2N6-MS 5-1-2	39%	23%	148%	13%		
	CSM-1999030-03-01-MS 5-1-2	139%		1-47,6	77%	12%	19%
	CSM-1999030-03-01-MSD 5-1-2	178%	158%	25%	69%		
	LWM-1999029-16-MS 5-1-2	146%				73%	11%
	LWM-1999029-11-MS 5-1-3	105%	125%	33%	61%		
	BTM-1999040-09-MS 5-1-2	147%		3374	76%	68%	22%
	BTM-1999040-10-MS 5-1-2	152%	150%	3%	80%		
Muscle	BINI	<loq (="" 0.0359)<="" td=""><td>1.50 /4</td><td>378</td><td>87%</td><td>83%</td><td>8%</td></loq>	1.50 /4	378	87%	83%	8%
Carp	B1N2	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	BIN7	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	BINIO	<loq (="" 0.0359)<="" td=""><td>l I</td><td></td><td><loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>	l I		<loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	B2N2	<loq (0.0359)<br=""><loq (0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq></loq>			<loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
\$	B2N6				<loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	B2N8	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	B2NI0	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0118)<="" td=""><td> · </td><td></td></loq></td></loq>			<loq (0.0118)<="" td=""><td> · </td><td></td></loq>	·	
	Carp1*	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0138)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (0.0138)<="" td=""><td> </td><td></td></loq>		
	Carp2*	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td> </td><td>NA</td></loq></td></loq>		NA	<loq (="" 0.0188)<="" td=""><td> </td><td>NA</td></loq>		NA
Muscle		<loq (="" 0.0359)<="" td=""><td>₹.00</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td> ⊲.00 </td><td>NA</td></loq></td></loq>	₹.00	NA	<loq (="" 0.0188)<="" td=""><td> ⊲.00 </td><td>NA</td></loq>	⊲.00	NA
Chinook Salmon	1999030-01	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
Currook Samon	1999030-02	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td> </td><td></td></loq>		
	1999030-02-01	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td> </td><td></td></loq>		
	1999030-02-04	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0138)<="" td=""><td>1 1</td><td></td></loq></td></loq>			<loq (="" 0.0138)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	1999030-03-01	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<="" td=""><td> </td><td>NA</td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td> </td><td>NA</td></loq>		NA
	1999030-03-04	<loq (="" 0.0359)<="" td=""><td>₹00</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td><.00</td><td>NA</td></loq></td></loq>	₹00	NA	<loq (="" 0.0188)<="" td=""><td><.00</td><td>NA</td></loq>	<.00	NA
Muscie	1999029-11*	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
Lake Whitefish	1999029-12*	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td>!!!</td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td>!!!</td><td></td></loq>	!!!	
	1999029-13	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td> </td><td>•</td></loq></td></loq>			<loq (0.0188)<="" td=""><td> </td><td>•</td></loq>		•
	1999029-14	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<="" td=""><td>1 1</td><td>NA</td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td>1 1</td><td>NA</td></loq>	1 1	NA
	1999029-16	<loq (="" 0.0359)<="" td=""><td><00₽</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td><⊾00</td><td>NA</td></loq></td></loq>	<00₽	NA	<loq (="" 0.0188)<="" td=""><td><⊾00</td><td>NA</td></loq>	<⊾00	NA
Muscle	1999040-01	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td>NA.</td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td>NA.</td></loq>		NA.
Brown Trout	1999040-02 **	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0138)<="" td=""><td>1 1</td><td></td></loq></td></loq>			<loq (0.0138)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	1999040-03	<loq (0.0359)<="" td=""><td>1</td><td></td><td><loq (0.0188)<="" td=""><td>] </td><td></td></loq></td></loq>	1		<loq (0.0188)<="" td=""><td>] </td><td></td></loq>]	
	1999040-04	<loq (0.0359)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td> </td><td></td></loq>		
	1999040-05	<loq (="" 0.0359)<="" td=""><td></td><td></td><td></td><td></td><td></td></loq>					
	1999040-06	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0138)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0138)<="" td=""><td></td><td></td></loq>		
	1999040-07	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-08	<loq (0.0359)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-09	<loq (0.0359)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-10	<loq (0.0359)<="" td=""><td><.00</td><td></td><td><loq (="" 0.0188)<="" td=""><td> </td><td>NA</td></loq></td></loq>	<.00		<loq (="" 0.0188)<="" td=""><td> </td><td>NA</td></loq>		NA
igh (>50%) surrogate of			1 2007	NA NE = Not Extracted	<loq (="" 0.0138)<="" td=""><td> ⊲.00 </td><td>NA</td></loq>	⊲.00	NA

** 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

E = Lost during extraction NA = Not Applicable LOQ = Limit of Quantitation X = Verified PFOS concentration PFOS - Perfluorooctanesulfonate PFOSA - Perfluorooctane sulfonamide PFHS = Perfluorobexanesulfonate

POAA = Perfluorooctanoete

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Study: Product Number(Test Substance): Matrix: Method/Revision: Method/Revision: Analytical Equipment System Number Instrument Software/Version: Date of Extraction/Analyst: Date of Data Reduction/Analyst: Date of Data Reduction/Analyst: Sample Data

GEN030 MSU Environmental Samples GEN030 MSU Environmental Various ETS-8-6.0 & ETS-8-7.0 Asnetia 062498 Massiyax 3.3 12/1299 SAL/KK/SRP/CSH 01/10/00 MMH 01/11/00 IAS

MUSCLE and EGGS

Group	Sample #	PFOS	Concentration	Mean	RSD	Concentration	Mean	RSD
Dose		Verified	of PFOS	PTOS	Std. Dev.	of PFHS	PFHS	Std. Der.
			ug/g or % Rec.	wg/g	MS/MSD RPD	wg/g or % Rec.	-2/1	MS/MSD RPI
Method Blk	MSU12159-H2OBlk-mfiltered-5-9	NA	<loq (0.0174)<="" td=""><td></td><td>NA</td><td><loq (0.0342)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
Water	MSU12159-H2OBIE-Elitered-5-9	NA	<loq (0.0174)<="" td=""><td><1.0Q</td><td>NA</td><td><loq (0.0342)<="" td=""><td><l00< td=""><td>NA</td></l00<></td></loq></td></loq>	<1.0Q	NA	<loq (0.0342)<="" td=""><td><l00< td=""><td>NA</td></l00<></td></loq>	<l00< td=""><td>NA</td></l00<>	NA
Matrix Blk	EggBik-5-1	NA	<loq (0.0174)<="" td=""><td></td><td></td><td><loq (0.0342)<="" td=""><td>· · · · · · · · · · · · · · · · · · ·</td><td></td></loq></td></loq>			<loq (0.0342)<="" td=""><td>· · · · · · · · · · · · · · · · · · ·</td><td></td></loq>	· · · · · · · · · · · · · · · · · · ·	
es.	EggBik-5-2	NA	<loq (0.0174)<="" td=""><td></td><td>NA</td><td><loo (0.0342)<="" td=""><td></td><td>NA</td></loo></td></loq>		NA	<loo (0.0342)<="" td=""><td></td><td>NA</td></loo>		NA
	EggBlk-5-3	NA	<loq (0.0174)<="" td=""><td><loq< td=""><td>NA</td><td><loq (0.0342)<="" td=""><td><1.00</td><td>NA</td></loq></td></loq<></td></loq>	<loq< td=""><td>NA</td><td><loq (0.0342)<="" td=""><td><1.00</td><td>NA</td></loq></td></loq<>	NA	<loq (0.0342)<="" td=""><td><1.00</td><td>NA</td></loq>	<1.00	NA
QC	PGW-ALSWCR-TD2 6/25/98-MS-5-1-1	x	44%			16%		
250 ng/g	FGW-ALSWCR-TD2 6/25/98-MSD-5-1-2	х	47%	46%	8%	16%	16%	3%
	FGE-AL118-HP26/25/96-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-MSID-5-1-2	x	167%	144%	32%	57%	57%	155
Carp	Diet I (Carpo) 🐃 🔪	X **	<loq (0.0174)<="" td=""><td></td><td></td><td><loq (0.0342)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	Diet 2 (Carp6)	x	4.00 (0.0174)			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	Diet 3 (Carp4)	x	0.0267		NA	<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
	Diet 4 (Carp3)	х	0.0278	0.0272 - 2 outliers	NA	₹.00 (0.0342)	4.00	NA
Frog Muscle	AL-118-YOY 08/26/96 **	X **	0.00243			<loq (0.0342)<="" td=""><td>- www.y</td><td></td></loq>	- www.y	
Wholebody	ALSWCR-TD 06/03/96 **	X **	<loo (0.0174)<="" td=""><td>1</td><td></td><td><loq (0.0342)<="" td=""><td></td><td></td></loq></td></loo>	1		<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	ALSWCR-TD2 06/25/99 **	X **	4.00 (0.0174)			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	KZCKDM-JUV 08/26/98 **	X **	<loo (0.0174)<="" td=""><td></td><td></td><td><loo (0.0342)<="" td=""><td></td><td></td></loo></td></loo>			<loo (0.0342)<="" td=""><td></td><td></td></loo>		
	KZCKDM-TD 06/05/98 **	X ++	0.0216			<loo (0.0342)<="" td=""><td></td><td>· · · ·</td></loo>		· · · ·
	KZCKDN-TD-2 06/25/98 **	X **	<loq (0.0174)<="" td=""><td></td><td></td><td><loq (0.0342)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	SJ0002-TD-2 06/05/98 **	X **	<lo0 (0.0174)<="" td=""><td></td><td>NA</td><td>4.00 (0.0342)</td><td></td><td>NA</td></lo0>		NA	4.00 (0.0342)		NA
	SJ0002-TD-2 06/25/98 **	X **	<loq (0.0174)<="" td=""><td><loq -="" 2="" outliers<="" td=""><td>NA</td><td><loq (0.0342)<="" td=""><td>⊲.00</td><td>NA</td></loq></td></loq></td></loq>	<loq -="" 2="" outliers<="" td=""><td>NA</td><td><loq (0.0342)<="" td=""><td>⊲.00</td><td>NA</td></loq></td></loq>	NA	<loq (0.0342)<="" td=""><td>⊲.00</td><td>NA</td></loq>	⊲.00	NA
Green Frog Legs	AL-118-HP89 06/25/96	X	<loq (0.0174)<="" td=""><td></td><td></td><td><loq (0.0342)<="" td=""><td></td><td>NA</td></loq></td></loq>			<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
	AL-111-10794 06/25/98	x	<1.00 (0.0174)			<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	SJ0001 06/03/98 **	X **	<loq (0.0174)<="" td=""><td></td><td>NA</td><td><loq (0.0342)<="" td=""><td></td><td></td></loq></td></loq>		NA	<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	SJ0001 06/05/98	x	<loq (0.0174)<="" td=""><td><1.00</td><td>NA</td><td></td><td>-</td><td>NA</td></loq>	<1.00	NA		-	NA
Lake Whitefish Eggs	1999029-13		0.145			<loq (0.0342)<="" td=""><td>4.0Q</td><td>NA</td></loq>	4.0 Q	NA
	1999029-14	x	0.145	0.263	63.5 0.167	<loq (0.0342)<="" td=""><td></td><td>NA</td></loq>		NA
Brown Trout Legs	1999040-01		0.381	0.203	0,167	<loq (0.0342)<="" td=""><td>4.0Q</td><td>NA</td></loq>	4.0Q	NA
STORE FOR FEE	1999040-04	x			l	<loq (0.0342)<="" td=""><td></td><td></td></loq>		
	1999040-04	x	0.0675		21.1	<loq (0.0342)<="" td=""><td></td><td> NA</td></loq>		NA
inh (>60%)		<u>x</u>	0.0488	0.0637	0.0134	<loq (0.0342)<="" td=""><td><1.00</td><td>NA</td></loq>	<1.00	NA

* High (>50%) survogate deviations ** FFOS 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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NE - Not Extracted E = Lost during extraction NA = Not Applicable LOQ = Limit of Quantitation X = Verified PFOS concentration

PPOS = Perfluorooctanesulfonate PPOS = Perfluorooctanesui romme PPOSA = Perfluorooctane salfonamide PFHS = Perfluorobecanesalfonate POAA = Perfluorooctanoste

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Study: Product Number(Test Substance): Matrix: Method/Revision: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Data Reduction/Analyst: Sample Data

GEN030 MSU Environmental Samples GEN030 MSU Environmental Various ETS-8-6.0 & ETS-8-7.0 Annelia 052491 Masilymx 3.3 12/1299 SAL/KK/SRP/CSH 01/10/00 MMH 01/11/00 IAS

MUSCLE and EGGS

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Group Dose	Sample #	Concentration of POAA	Mean POAA	RSD Std. Dev.	Concentration	Mesa	RSD
		ug/g or % Rec.	42/1	MS/MSD RPD	of PFOSA Bg/g or % Rec.	PFOSA	Std. Dev.
Method Blk	MSU12159-H2OBik-unfiltered-5-9	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td>42/2</td><td>MS/MSD RPD</td></loq></td></loq>		NA	<loq (="" 0.0188)<="" td=""><td>42/2</td><td>MS/MSD RPD</td></loq>	42/2	MS/MSD RPD
Water	MSU12159-H2OBik-filtered-5-9	<loq (="" 0.0359)<="" td=""><td><1.00</td><td>NA</td><td><loq (0.0188)<="" td=""><td>-100</td><td>NA</td></loq></td></loq>	<1.00	NA	<loq (0.0188)<="" td=""><td>-100</td><td>NA</td></loq>	-100	NA
Matrix Blk	EggBik-S-I	<loq (0.0359)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td><100</td><td>NA</td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td><100</td><td>NA</td></loq>	<100	NA
888	EggBik-5-2	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td><loq (0.0(88)<="" td=""><td></td><td></td></loq></td></loq>		NA	<loq (0.0(88)<="" td=""><td></td><td></td></loq>		
	EggB&-5-3	<loo (="" 0.0359)<="" td=""><td>4.00</td><td>NA</td><td><loq (0.0188)<="" td=""><td>-</td><td>NA</td></loq></td></loo>	4.00	NA	<loq (0.0188)<="" td=""><td>-</td><td>NA</td></loq>	-	NA
QC	FGW-ALSWCR-TD2 6/25/98-MS-5-1-2	34%			49%	<1.0Q	· NA
250 ng/g	FGW-ALSWCR-TD2 6/25/98-MSD-5-1-2	33%	34%	4%	49%		
	FGE-AL118-HP26/25/98-MS-5-1-2	22%	2414	*/*	46%	49%	1%
	FGE-AL118-HP26/25/98-MSD-5-1-2	26%	24%	15%	54%		
	LWE-19999029-13-MS-5-1-2	90%			64%	50%	16%
	LWE-19999029-13-MSD-5-1-2	83%	86%	9%	64%	66%	
	BTE-19999040-01-MS-5-1-2	98%			104%	00.76	6%
	BTE-19999040-01-MSD-5-1-2	112%	105%	13%	108%	106%	
Сытр	Dist 1 (Garp5) **	<loq (="" 0.0359)<="" td=""><td>-</td><td></td><td><loq (="" 0.0188)<="" td=""><td>10076</td><td>4%</td></loq></td></loq>	-		<loq (="" 0.0188)<="" td=""><td>10076</td><td>4%</td></loq>	10076	4%
	Diet 2 (Carp6)	<loq (="" 0.0359)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	Dist 3 (Curp4)	<loq (="" 0.0359)<="" td=""><td></td><td>NA</td><td>4.00 (0.0188)</td><td></td><td></td></loq>		NA	4.00 (0.0188)		
	Diet 4 (Carp3)	<loq (="" 0.0359)<="" td=""><td>4.00</td><td>NA</td><td><loq (="" 0.0158)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	4.00	NA	<loq (="" 0.0158)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
Frog Muscle	AL-118-YOY 08/26/98 **	<loq (="" 0.0180)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td>400</td><td>NA</td></loq></td></loq>			<loq (0.0188)<="" td=""><td>400</td><td>NA</td></loq>	400	NA
Wholebody	ALSWCR-TD 06/03/98 **	<loq (="" 0.0150)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	ALSWCR-TD2 06/25/99 **	<loq (="" 0.0110)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	KZCKDM-JUV 08/26/98 **	<loq (="" 0.0180)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	KZCKDM-TD 06/05/98 **	<loq (="" 0.0110)<="" td=""><td></td><td></td><td><loq (0.0128)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0128)<="" td=""><td></td><td></td></loq>		
• .	KZCKDM-TD-2 06/25/98 **	<loq (="" 0.0180)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	SJ0002-TD-2_06/05/98 **	<loq (="" 0.0180)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>		NA	<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	SJ0002-TD-2 06/25/98 **	<loo (="" 0.0180)<="" td=""><td>₹.00</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td>400</td><td>NA</td></loq></td></loo>	₹.00	NA	<loq (="" 0.0188)<="" td=""><td>400</td><td>NA</td></loq>	400	NA
irem Frog Eggs	AL-118-HP89 06/25/98	<loq (="" 0.0180)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td><1.0Q</td><td>NA</td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td><1.0Q</td><td>NA</td></loq>	<1.0Q	NA
	AL-118-HP94 06/25/98	<loq (="" 0.0180)<="" td=""><td></td><td></td><td></td><td></td><td></td></loq>					
	SJ0001 06/03/98 **	<loq (="" 0.0180)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	SJ0001 06/05/98	<loq (0.0180)<="" td=""><td>4.00</td><td>NA</td><td><loq (0.0188)<="" td=""><td></td><td>NA</td></loq></td></loq>	4.00	NA	<loq (0.0188)<="" td=""><td></td><td>NA</td></loq>		NA
ke Whitefish Eggs	1999029-13	<loq(0.0180)< td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td><10Q</td><td>NA</td></loq></td></loq(0.0180)<>		NA	<loq (="" 0.0188)<="" td=""><td><10Q</td><td>NA</td></loq>	<10Q	NA
	1999029-14	<loq (0.0180)<="" td=""><td>₹00</td><td>NA NA</td><td><loq (0.0188)<="" td=""><td></td><td>NA</td></loq></td></loq>	₹00	NA NA	<loq (0.0188)<="" td=""><td></td><td>NA</td></loq>		NA
rows Trout Eggs	1999040-01	<loq (0.0180)<="" td=""><td>~~~</td><td>NA.</td><td><loq (="" 0.0188)<="" td=""><td><10Q</td><td>NA</td></loq></td></loq>	~~~	NA.	<loq (="" 0.0188)<="" td=""><td><10Q</td><td>NA</td></loq>	<10Q	NA
	1999040-04	<loq (0.0180)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td>1</td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td>1</td></loq>		1
	1999040-06	<loq (0.0180)<="" td=""><td>₹.00</td><td>NA NA</td><td><loq (0.0188)<="" td=""><td></td><td>NA</td></loq></td></loq>	₹.00	NA NA	<loq (0.0188)<="" td=""><td></td><td>NA</td></loq>		NA
(>50%) surrogate devi				NA NE = Not Extracted	<loq (="" 0.0188)<="" td=""><td><loq< td=""><td>NA</td></loq<></td></loq>	<loq< td=""><td>NA</td></loq<>	NA

** PFOS NOT confirmed; MS transitions variation > 30% Date Entered/Analyst: Date Verified/Analyst: 01/21/00, 01/24/00 LAC 02/10/00 bjb

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 NE = Not Extracted

 E = Lost during extraction

 NA = Not Applicable

 LOQ = Limit of Quantitation

 X = Verified PFOS concentration

PFOS = Perfluorooctanesulfonate PFOSA = Perfuorooctane sulfonamide PFHS = Perfuorobcumerulfonate POAA = Perfluorooctanoate

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Study Product Number(Test Substance) Matrix: Method/Revision Analytical Equipment System Number Instrument Software/Version: Date of Extraction/Analyst. Date of Analysis/Analyst: Date of Data Reduction/Analyst Sample Data

GEN030 MSU Environmental Samples Various Various ETS-8-6.0 & ETS-8-7.0 Soup 020199, Amelia 062498 Solp 20179, Ameria 002498 Massiyux 3 3 12/12/99 SAL/KK/SRP/CSH 12/17/99, 12/20/99, 12/28/99, 12/29/99, 01/03/00, 01/06/00 LAS/MMH 12/20/99, 12/21/99, 12/22/99, 12/30/99, 01/03/00, 01/05/00, 01/07/00 MMH//IAS

Group Dose	Sample #	PFOS Vertified	Concentration of PFOS ug/g or % Rec.	Mean PFOS ug/g	RSD Std. Dev. MS/MSD RPD	Concentration of PFHS	Mean PFHS	RSD Std. Dev.
Method Blk	MSU12129-H2OBik-unfiltered-5-1	NA	NA			BE/E or % Rec.	<u>"2/2</u>	MS/MSD RI
Unfiltered water	MSU12129-H2OBlk-unfiltered-5-2	NA	NA			NA		
	MSU12129-H2OBlk-unfiltered-5-3	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td>i.</td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td>i.</td></loq>		i.
	MSU12129-H2OBlk-unfiltered-5-4	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td>: .'</td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td>: .'</td></loq>		: .'
	MSU12129-H2OBlk-unfiltered-5-5	NA	E			E		
	MSU12129-H2OBlk-unfiltered-5-6	NA	<loq (0.0696)<="" td=""><td></td><td>1</td><td><loq (0.0171)<="" td=""><td></td><td></td></loq></td></loq>		1	<loq (0.0171)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBlk-unfiltered-5-7	NA	<loq (0.0696)<="" td=""><td></td><td>NA</td><td><loq (0.0171)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0171)<="" td=""><td></td><td>NA</td></loq>		NA
	MSU12129-H2OBlk-unfiltered-5-8	NA	<loq (0.0696)<="" td=""><td><l0q< td=""><td>NA</td><td><loq (0.0171)<="" td=""><td><loq< td=""><td>NA</td></loq<></td></loq></td></l0q<></td></loq>	<l0q< td=""><td>NA</td><td><loq (0.0171)<="" td=""><td><loq< td=""><td>NA</td></loq<></td></loq></td></l0q<>	NA	<loq (0.0171)<="" td=""><td><loq< td=""><td>NA</td></loq<></td></loq>	<loq< td=""><td>NA</td></loq<>	NA
Method Blk	MSU12129-H2OBik-filtered-5-1	NA	NA			NA		
Filtered water	MSU12129-H2OBlk-filtered-5-2	NA	NA			NA		
	MSU12129-H2OBik-filtered-5-3	NA	E			· E		
	MSU12129-H2OBik-filtered-5-4	NA NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td>1 1</td><td></td></loq></td></loq>			<loq (0.00683)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	MSU12129-H2OBik-fiitered-5-5	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td>1 1</td><td>-</td></loq></td></loq>			<loq (0.00683)<="" td=""><td>1 1</td><td>-</td></loq>	1 1	-
	MSU12129-H2OBik-filtered-5-6	NA	<loq (0.0696)<="" td=""><td></td><td></td><td><loq (0.0171)<="" td=""><td>1 1</td><td></td></loq></td></loq>			<loq (0.0171)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	MSU12129-H2OBik-filtered-5-7	NA	<loq (0.0696)<="" td=""><td></td><td>NA</td><td><loq (0.0171)<="" td=""><td>1 1</td><td>NA</td></loq></td></loq>		NA	<loq (0.0171)<="" td=""><td>1 1</td><td>NA</td></loq>	1 1	NA
	MSU12129-H2OBik-filtered-5-8	NA	<loq (0.0696)<="" td=""><td><700</td><td>NA</td><td><loq (0.0171)<="" td=""><td>1 4.00</td><td>I NA</td></loq></td></loq>	<700	NA	<loq (0.0171)<="" td=""><td>1 4.00</td><td>I NA</td></loq>	1 4.00	I NA
Matrix Blk	FSH12129-LvrBlk-5-1	NA	0.0305			<loq (0.0171)<="" td=""><td></td><td></td></loq>		
Fish Liver	FSH12129-LvrBlk-5-2	NA	0.0331		32.1	<loq (0.0171)<="" td=""><td></td><td>NA</td></loq>		NA
•	FSH12129-LvrBlk-5-3	NA	0.0170	0.0269	0.00862	<loq (0.0171)<="" td=""><td>100</td><td>NA</td></loq>	100	NA
Marix Blk	RBL12129-LvrBlk-S-1	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td>+</td><td>. 14 %</td></loq></td></loq>			<loq (0.00683)<="" td=""><td>+</td><td>. 14 %</td></loq>	+	. 14 %
Rabbit Liver	RBL12129-LvrBik-5-2	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (0.00683)<="" td=""><td> </td><td></td></loq>		
.4	RBL12129-LyrBik-5-3	NA	<loq (0.0696)<="" td=""><td></td><td></td><td><loq (0.0171)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0171)<="" td=""><td></td><td></td></loq>		
	RBL12129-LvrBik-5-4	NA	<loq (0.0696)<="" td=""><td></td><td></td><td><loq (0.0171)<="" td=""><td>1</td><td>•</td></loq></td></loq>			<loq (0.0171)<="" td=""><td>1</td><td>•</td></loq>	1	•
	RBL12129-LvrBik-5-5*	NA	<loq (0.0696)<="" td=""><td></td><td>NA</td><td><loq (0.0171)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0171)<="" td=""><td></td><td>NA</td></loq>		NA
	RBL12129-LvrBlk-5-6*	NA	<loq (0.0696)<="" td=""><td><loq< td=""><td>NA</td><td><loq (0.0171)<="" td=""><td><1.0Q</td><td>NA</td></loq></td></loq<></td></loq>	<loq< td=""><td>NA</td><td><loq (0.0171)<="" td=""><td><1.0Q</td><td>NA</td></loq></td></loq<>	NA	<loq (0.0171)<="" td=""><td><1.0Q</td><td>NA</td></loq>	<1.0Q	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%	
	LWL-1999029-12-MS-5-1-2*	NA	321%			57%	1	
	LWL-1999029-12-MSD-5-1-2*	NA	287%	304%	11%	68%	63%	18%
	BTL-1999040-01-MS-5-1-2*	NA	138%		1	81%		;
	BTL-1999040-01-MSD-5-1-2*	NA	132%	135%	5%	84%	83%	4%
	TNL-TU54-MS-5-1-2	NA	68%		1	\$5%		
	TNL-TU54-MSD-5-1-2	NA	E	68%	NA	Е	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-S-1-2	NA '	93%			82%		
	GFL-KZCKDM-DI-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		1	<loq (0.0171)<="" td=""><td>-</td><td></td></loq>	-	
Chinook Salmon	1999030-02	x	0.169			<loq (0.0171)<="" td=""><td></td><td></td></loq>		
	1999030-02-01	NA	0.0328		1	<loq (0.0171)<="" td=""><td>1</td><td></td></loq>	1	
	1999030-02-04	NA	0.126		1	<loq (0.0171)<="" td=""><td>1</td><td></td></loq>	1	
	1999030-03-01	NA	0.173		56.1	<loq (0.0171)<="" td=""><td></td><td>NA</td></loq>		NA
	1999030-03-04	NA	0.0405	0.108	0.0608	<loq (0.0171)<="" td=""><td><l00< td=""><td>NA NA</td></l00<></td></loq>	<l00< td=""><td>NA NA</td></l00<>	NA NA
Liver	1999029-11	NA	0.0679	· · · · · · · · · · · · · · · · · · ·		<loq (0.0171)<="" td=""><td></td><td></td></loq>		
Lake Whitefish	1999029-12	NA	0.0812			<loq (0.0171)<br=""><loq (0.0171)<="" td=""><td></td><td>1 2</td></loq></loq>		1 2
	1999029-13	x	0.0738					1
	1999029-14	Î	0.0329		29.3	<loq (0.0171)<br=""><loq (0.0171)<="" td=""><td></td><td>·</td></loq></loq>		·
	1999029-16	NA	0.0778	0.0667	0.0195		1	NA
Liver	1999040-01	NA		0.0001	0.0155	<loq(0.0171)< td=""><td><1.0Q</td><td>NA</td></loq(0.0171)<>	<1.0Q	NA
Brown Trout	1999040-02	NA	<loq (="" 0.0174)<br=""><loq (="" 0.0174)<="" td=""><td></td><td>1</td><td><loq (0.0171)<="" td=""><td>1</td><td></td></loq></td></loq></loq>		1	<loq (0.0171)<="" td=""><td>1</td><td></td></loq>	1	
	1999040-03	NA	<loq (="" 0.0174)<="" td=""><td>1</td><td></td><td><loq (0.0171)<="" td=""><td>1</td><td>1</td></loq></td></loq>	1		<loq (0.0171)<="" td=""><td>1</td><td>1</td></loq>	1	1
	1999040-04	NA	<loq (="" 0.0174)<="" td=""><td></td><td></td><td><loq (0.0171)<="" td=""><td>- F</td><td>·</td></loq></td></loq>			<loq (0.0171)<="" td=""><td>- F</td><td>·</td></loq>	- F	·
	1999040-05	NA				<loq (0.0171)<="" td=""><td></td><td>-</td></loq>		-
	1999040-05	1	0.0255			<loq (0.0171)<="" td=""><td></td><td>1</td></loq>		1
		NA	<loq (="" 0.0174)<="" td=""><td></td><td></td><td><loq (="" 0.0171)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0171)<="" td=""><td></td><td></td></loq>		
	1999040-07	NA	<loq (="" 0.0174)<="" td=""><td></td><td>· ·</td><td><loq (="" 0.0171)<="" td=""><td></td><td> </td></loq></td></loq>		· ·	<loq (="" 0.0171)<="" td=""><td></td><td> </td></loq>		
	1999040-08	NA	<loq (="" 0.0174)<="" td=""><td>· ·</td><td></td><td><loq (="" 0.0171)<="" td=""><td></td><td>1</td></loq></td></loq>	· ·		<loq (="" 0.0171)<="" td=""><td></td><td>1</td></loq>		1
	1999040-09	NA	<loq (="" 0.0174)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0171)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (="" 0.0171)<="" td=""><td></td><td>NA</td></loq>		NA
lich /> 508/	1999040-10	NA	<loq (="" 0.0174)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>NA</td><td><loq (="" 0.0171)<="" td=""><td><l0q< td=""><td>NA</td></l0q<></td></loq></td></loq></td></loq>	<loq -="" 1="" outlier<="" td=""><td>NA</td><td><loq (="" 0.0171)<="" td=""><td><l0q< td=""><td>NA</td></l0q<></td></loq></td></loq>	NA	<loq (="" 0.0171)<="" td=""><td><l0q< td=""><td>NA</td></l0q<></td></loq>	<l0q< td=""><td>NA</td></l0q<>	NA
high (>50%) surrogate of	seviations			NE - Not Extracted		PFOS = Perfluorooctanceu		
				E = Lost during extraction		PFOSA = Pefluorooctane :		
te Entered/Analyst:	12/22/99, 12/28/99, 12/29/99, 12/30/99, 01/12/00, 01/17/00, 01/18/00 LAC			NA = Not Applicable		PFHS = Perfluorohexanen	lifonate	
				LOQ = Limit of Quantitation				

ETS-8-7.0 Excel Version 3/95

GEN-030-liver.xls

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Study: Product Number(Test Substance) Matrix: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

4

GEN030 MSU Environmental Samples Various Various ETS-8-6.0 & ETS-8-7.0 Soup 020199, Amelia 062498 Soup 020199, Ameria 062498 Massiynx 3.3 12/1299 SAL/KK/SRP/CSH 12/1799, 12/2099, 12/28/99, 12/29/99, 01/03/00, 01/05/00 IAS/MMH 12/20/99, 12/21/99, 12/22/99, 12/30/99, 01/03/00, 01/05/00, 01/07/00 MMH/IAS

Group	Sample #	Concentration	Mean	RSD	Concentration	Mean	
Dose		of POAA	POAA	Std. Dev.	of PFOSA	PFOSA	RSD
		ug/g or % Rec.	42/2	MS/MSD RPD	ug/g or % Rec.		Std. Dev.
Method Blk	MSU12129-H2OBlk-unfiltered-5-1	NA			NA	<u>"#/8</u>	MS/MSD RPD
Unfiltered water	MSU12129-H2OBlk-unfiltered-5-2	NA	1 1		NA	1 1	
	MSU12129-H2OBlk-unfiltered-5-3	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td> </td><td></td></loq>		
	MSU12129-H2OBik-unfiltered-5-4	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.018b)<="" td=""><td>1</td><td></td></loq></td></loq>			<loq (="" 0.018b)<="" td=""><td>1</td><td></td></loq>	1	
	MSU12129-H2OBlk-unfiltered-5-5	E	1		E	1 1	
	MSU12129-H2OBlk-unfiltered-5-6	<loq (0.0719)<="" td=""><td>1 1</td><td></td><td><loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>	1 1		<loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	MSU12129-H2OBlk-unfiltered-5-7	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td>1 1</td><td>NA .</td></loq></td></loq>		NA	<loq (="" 0.0188)<="" td=""><td>1 1</td><td>NA .</td></loq>	1 1	NA .
	MSU12129-H2OBIk-unfiltered-5-8	<loq (0.0719)<="" td=""><td><100</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td><.00</td><td>NA</td></loq></td></loq>	<100	NA	<loq (="" 0.0188)<="" td=""><td><.00</td><td>NA</td></loq>	<.00	NA
Method Blk	MSU12129-H2OBik-filtered-5-1	NA			NA	- <u> </u>	
Filtered water	MSU12129-H2OBIk-filtered-5-2	NA	1		NA	1	
	MSU12129-H2OBIE-filtered-5-3	B	1		E	1 1	
	MSU12129-H2OBlk-filtered-5-4	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	MSU12129-H2OBlk-Siltered-5-5	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	MSU12129-H2OBlk-filtered-5-6	<loq (0.0719)<="" td=""><td>1 1</td><td></td><td><loq (0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>	1 1		<loq (0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	MSU12129-H2OBik-Eltered-5-7	<loq (0.0719)<="" td=""><td>1</td><td>NA</td><td><loq (0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>	1	NA	<loq (0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
	MSU12129-HZOBlk-filtered-5-8	<loq (0.0719)<="" td=""><td><.00</td><td>NA</td><td><loq (0.0188)<="" td=""><td><.00</td><td>NA</td></loq></td></loq>	<.00	NA	<loq (0.0188)<="" td=""><td><.00</td><td>NA</td></loq>	<.00	NA
Matrix Blk	FSH12129-LvrBik-5-1	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td>1</td><td>NA</td></loq></td></loq>			<loq (0.0188)<="" td=""><td>1</td><td>NA</td></loq>	1	NA
Fish Liver	FSH12129-LvrBlk-5-2	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>		NA	<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	FSH12129-LvrBlk-5-3	<loq (0.0719)<="" td=""><td><.00</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td><100</td><td>NA</td></loq></td></loq>	<.00	NA	<loq (="" 0.0188)<="" td=""><td><100</td><td>NA</td></loq>	<100	NA
Matrix Blk	RBL12129-LvrBik-5-1	<loq (0.0719)<="" td=""><td>+</td><td>.41</td><td><loq (="" 0.0188)<="" td=""><td>1</td><td>NA</td></loq></td></loq>	+	.41	<loq (="" 0.0188)<="" td=""><td>1</td><td>NA</td></loq>	1	NA
Rabbit Liver	RBL12129-LvrBlk-5-2	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td> </td><td></td></loq>		
	RBL12129-LvrBik-5-3	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td> </td><td></td></loq>		
	RBL12129-LvrBlk-5-4	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	RBL12129-LvrBlk-5-5*	<loq (0.0719)<="" td=""><td>1</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1	NA	<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	RBL12129-LvtBlk-5-6*	<loq (0.0719)<="" td=""><td><1.00</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td>1 7 00 1</td><td>NA</td></loq></td></loq>	<1.00	NA	<loq (="" 0.0188)<="" td=""><td>1 7 00 1</td><td>NA</td></loq>	1 7 00 1	NA
QC	Mink Liver, D530, MS-5-1-1	86%			43%	<100	NA
250 ng/g	Mink Liver, D530, MSD-5-1-2*	84%	85%	3%	81%	(74)	
	CSL-1999030-03-01-MS-5-1-2*	131%	-		64%	62%	. 60%
	CSL-1999030-03-01-MSD-5-1-2*	111%	121%	17%	52%		
	LWL-1999029-12-MS-5-1-2*	133%			77%	58%	21%
	LWL-1999029-12-MSD-5-1-2*	145%	139%	9%	83%		
	BTL-1999040-01-MS-5-1-2*	127%		774	94%	80%	7%
	BTL-1999040-01-MSD-5-1-2*	120%	124%	6%	92%		
-	TNL-TUS4-MS-5-1-2	84%		0/4	71%	93%	3%
	TNL-TU54-MSD-5-1-2	E	B4%	NA			•
	FSL-P295-MS-5-1	72%			E	71%	NA
	FSL-P295-MSD-5-1	80%	76%	10%	82%		
	PBL-980390LB-MS-5-1	85%			71%	85%	7%
	PBL-980390LB-MSD-5-2*	129%	107%	41%	65%	1 1001	
	GFL-KZCKDM-D1-MS-5-1-2	82%	1		82%	68%	9%
	GFL-KZCKDM-DI-MSD-S-1-	103%	92%	23%	93%	88%	104/
	TTL-LCPTR99503C-MS-5-1-2*	73%			78%	0070	12%
	TTL-LCPTR99503C-MSD-5-1-2	64%	69%	13%	71%	74%	104/
	MTL-10Vancleave98-MS-5-1-2	100%			74%	1970	10%
	MTL-10Vancleave98-MSD-5-1-2	75%	88%	29%	74%	1	
Liver	1999030-01	<loq (0.0719)<="" td=""><td>++</td><td>~ ~ ~ ~</td><td><loq (="" 0.0188)<="" td=""><td>74%</td><td>0%</td></loq></td></loq>	++	~ ~ ~ ~	<loq (="" 0.0188)<="" td=""><td>74%</td><td>0%</td></loq>	74%	0%
Chinook Salmon	1999030-02	<loq (0.0719)<="" td=""><td></td><td></td><td></td><td>1</td><td></td></loq>				1	
	1999030-02-01	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (0.0188)<="" td=""><td> </td><td></td></loq></td></loq>	1		<loq (0.0188)<="" td=""><td> </td><td></td></loq>		
	1999030-02-04	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (0.0118)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (0.0118)<="" td=""><td> </td><td></td></loq>		
	1999030-03-01	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	1999030-03-04	<loq (0.0719)<="" td=""><td>1.00</td><td>NA NA</td><td><loq (0.0188)<="" td=""><td> </td><td>NA</td></loq></td></loq>	1.00	NA NA	<loq (0.0188)<="" td=""><td> </td><td>NA</td></loq>		NA
Liver	1999029-11	<loq (0.0719)<="" td=""><td>+</td><td></td><td><loq (="" 0.0188)<="" td=""><td><100</td><td>NA</td></loq></td></loq>	+		<loq (="" 0.0188)<="" td=""><td><100</td><td>NA</td></loq>	<100	NA
Lake Whitefish	1999029-12	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	1999029-13	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (="" 0.0188)<="" td=""><td> </td><td></td></loq></td></loq>	1		<loq (="" 0.0188)<="" td=""><td> </td><td></td></loq>		
	1999029-14		1 1		<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	1999029-16	<loq (0.0719)<="" td=""><td>100</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td></td><td>NA</td></loq></td></loq>	100	NA	<loq (="" 0.0188)<="" td=""><td></td><td>NA</td></loq>		NA
Liver	1999040-01	<loq (0.0719)<="" td=""><td>+ ~~~~+</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td><r00< td=""><td>NA</td></r00<></td></loq></td></loq>	+ ~~~~+	NA	<loq (="" 0.0188)<="" td=""><td><r00< td=""><td>NA</td></r00<></td></loq>	<r00< td=""><td>NA</td></r00<>	NA
Brown Trout	1999040-02	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-02	<loq (0.0719)<="" td=""><td>1 1</td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1 1		<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-03	<loq (0.0719)<="" td=""><td>1 1</td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1 1		<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-05	<loq (0.0719)<="" td=""><td>1 1</td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1 1		<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-06	<loq (0.0719)<="" td=""><td>1 1</td><td></td><td><loq (="" 0.0188)<="" td=""><td>1</td><td></td></loq></td></loq>	1 1		<loq (="" 0.0188)<="" td=""><td>1</td><td></td></loq>	1	
	1999040-06	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1		<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
		<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-08	<loq (0.0719)<="" td=""><td> 1</td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1		<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	1999040-09	<loq (0.0719)<="" td=""><td> </td><td>NA</td><td><loq (0.0188)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td></td><td>NA</td></loq>		NA
L (> (01()	1999040-10	<loq (0.0719)<="" td=""><td><1.0Q</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td><l0q< td=""><td>NA</td></l0q<></td></loq></td></loq>	<1.0Q	NA	<loq (="" 0.0188)<="" td=""><td><l0q< td=""><td>NA</td></l0q<></td></loq>	<l0q< td=""><td>NA</td></l0q<>	NA
th (>50%) surrogate de	;viacions		NE = Not E	•	PFOS = Perfluorooctanes		
Entered/Ar-1	12/22/20 12/28/20 10/28/20 12/28/20 12/28/20 12/28/20 12/28/20 12/28/20 12/28/20 12/28/2000 12/28/2000 12/2000 12/28/2000 12/28/2000000000000000000000000000000000			ring extraction	PFOSA = Pefluorooctane		
Entered/Analyst:	12/22/99, 12/28/99, 12/29/99, 12/30/99, 01/12/00, 01/17/00, 01/18/00 LAC		NA = Not A		PFHS = Perfluorohexene		
			LOQ = Lim				

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Miscellaneous Liver

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GEN030 MSU Environmental Samples Various
Various
ET3-4-6.0 & ET3-4-7.0
Serup 020199, Amerila 062498
Manalynx 33
12/12/99 SAL/KK/SRP/CSH
2/17/99, 12/20/99, 12/22/99, 12/29/99, 01/03/00, 01/05/00, 01/06/00, 01/06/00 EAS/MINH
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 MMEVIAS

Group Dom	Sampin d	PFOS Verified	Convertation of PEOS	Mine PPOS	RSD Std. Dev.	Concentration of PFES	Mesa PFES	RSD Stat. Dave.
Method Blk	MSU12129-H2OB0	NA	vg/g or % Res.		MS/MSD R/D	NA		MS/MSD R/1
Infiltered water	MSU11139-HDOBik-en/Devel-5-2	NA	NA			NA		
	MSU12129-H2DBB:-m/08mmi-3-3	NA	<loq (0.0147)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	MSU12129-H3OB0:-mdlmrei-5-4	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBBm6hmul-5-5 MSU12129-H2OBBm6hmul-5-6	NA	E			E		
	MSU12129-HEDBitz-method-5-7	NA NA	<0.00(0.0696)			<loq (0.0171)<="" td=""><td></td><td></td></loq>		
	MSU12129-H3OBit-mailtored-5-8	NA	<loq (0.0696)<br=""><loq (0.0696)<="" td=""><td>4.00</td><td>NA NA</td><td>4.0Q (0.0171)</td><td></td><td>NA</td></loq></loq>	4.00	NA NA	4.0Q (0.0171)		NA
Method Bilt	MSU12129-HSOB@c-@immed-5-1	NA	NA	- CLAQ	· NA	<loq (0.0171)<br="">NA</loq>	100	
Filtered water	MSU12129-H2OBBr-filment-5-2	NA	NA			NA		
	MSU12129-H3OBBt-filtered-5-3	NA	E			B		
	MSU12129-H20BBc-Bland-5-4 MSU12129-H20BBc-Bland-5-5	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	MSU12129-H20Blk-@www.5-6	NA NA	4LOQ (0.0347)			<loq (0.00683)<="" td=""><td> </td><td></td></loq>		
	MSU12129-H2088t-Bland-5-7	NA	<pre>4LOQ (0.0696) 4LOQ (0.0696)</pre>			⊄.0Q(0.0171)		
	MSU12129-H20BBr-dilared-5-4	NA	4.00 (0.0696)	4.00	NA NA	<.0Q (0.0171)		NA
Matrix Blz	FSH12129-Lvr8ik-5-1	NA	0.0305			4.0Q(0.0171) 4.0Q(0.0171)	4.00	NA
Figh Liver	PSH12129-L-m8th-5-2	NA	0.0331		32.1	LOQ (9.0171)		NA
	FSH12129-Lvr82-3-3	NA	0.0170	0.0269	0.00862	4.0Q(0.0171)	₹ .0Q	NA
Matrix Blk Rebbit Liver	RBL12129-Lvr88-5-1 RBL12129-Lvr88-5-2 ~	NA	<loq (0.0347)<="" td=""><td>-</td><td></td><td><loq (0.00683)<="" td=""><td></td><td></td></loq></td></loq>	-		<loq (0.00683)<="" td=""><td></td><td></td></loq>		
Name of Calvar	88L12129-LV188-3-3	NA NA	4.00 (0.0347)			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	XBL12129-LvrBE-5-4	NA	<loq (0.0696)<br=""><loq (0.0696)<="" td=""><td></td><td></td><td>4.0Q (0.0171)</td><td>1 </td><td></td></loq></loq>			4.0Q (0.0171)	1	
	REL12125-LwRE-5-5*	NA	4.00 (0.0696)		NA	4.0Q(0.0171)		
	RBL12129-LvrBB-5-6*	NA	4.00 (0.0006)	00	NA NA	4.0Q (0.0171) 4.0Q (0.0171)		NA
90	Mints Liver, 0530, 348-5-1-1	NA	145%			61%	4.00	NA
250 mg/g	Mink Liver, D530, MSD-5-1-2*	NA	538%	342%	115%	\$0%	55%	21%
	C31-1999030-03-01-NS-5-1-2"	NA	258%			116%		4179
Ĺ	CSL-1999030-03-01-348D-3-1-2*	NA	202%	230%	24%	112%	114%	-
	LWL-1999829-12-465-5-1-2*	NA	151%			\$7%		
–	LWL-1999029-12-3680-5-1-2*	NA	317%	334%	10%	68%	63%	18%
	BTL-1999040-01-MS-5-1-2* BTL-1999040-01-MSD-5-1-2*	NA	138%			81%		
	TRL-199000-01-460-5-1-2	NA	132%	135%			83%	4%
•	TNL-TU34-M3D-3-1-2	NA	96% E	67%.	NA	55%		
H	FSL-P295-M3-5-1	NA	111%			<u> </u>	55%	NA
L	PSL-P295-MSD-5-1	NA	106%	108%	5%	84%	90%	15%
	PBL-900390(,B-MS-S-1	NA	77%			70%		
<u> </u>	PSL-980390[_B-MSD-5-2*	NA	238%	158%	102%	78%	73%	9%
	GPL-K2CKD44-D1-448-3-1-2	NA	101%			12%		
	CFL-K2CKDM-D1-MSD-5-1- TTL-LCP(R9950)C-MS-5-1-2*	NA NA	112%	107%	11%		86%	9%
1	TTL-LCPTR9903C-M0D-5-1-2	NA	92%	97%	10%	66% 72%	ا شما	
	MTL-10Vascianve98-bd8-5-1-2	NA	108%		1974	14%	69%	1%
ł	MTL-10Vateleave98-MSD-5-1-2	NA	118%	113%	1%	14%	79%	13%
Liver	P233	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td>1374</td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td>1374</td></loq>		1374
forthern Fur Seal	P285**	×	9.122			<loq (0.00683)<="" td=""><td> </td><td></td></loq>		
	P295	NA	0.0547			<loq (0.00683)<="" td=""><td>1</td><td></td></loq>	1	
1	97 CU 02	NA	NE			NE	1	
	96 CU KB 02 96 CU KB 03	NA	<0.0347)			<loq (0.00683)<="" td=""><td></td><td>. •</td></loq>		. •
1	96 CU KB 03 96 CU KB 07	NA NA	4.00 (0.0347)	• •	1	0.9587		-
	96 CU KB 09	NA	<			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	94 CU X3 10	NA	<000 (0.0347)			<pre><loq(0.00683) <loq(0.00683)< pre=""></loq(0.00683)<></loq(0.00683) </pre>		
	91 CU KB 11	NA	4.00 (0.0347)	i		<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	90 CU XCB 12	NA	<loq (0.0347)<="" td=""><td>1</td><td></td><td>4.0Q (0.00683)</td><td></td><td></td></loq>	1		4.0Q (0.00683)		
	96 CUKB 13	NA	<lo0 (0.0347)<="" td=""><td>}</td><td></td><td>4.00 (0.00683)</td><td>1</td><td></td></lo0>	}		4.00 (0.00683)	1	
		NA .	<loq (0.03477)<="" td=""><td></td><td>NA</td><td><loq (0.00683)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.00683)<="" td=""><td></td><td>NA</td></loq>		NA
	96 CU KB 15	NA	<loq (0.0347)<="" td=""><td><loq -="" 2="" outlines<="" td=""><td>NA</td><td>0.0858</td><td>4.0Q-2 outlines</td><td>NA</td></loq></td></loq>	<loq -="" 2="" outlines<="" td=""><td>NA</td><td>0.0858</td><td>4.0Q-2 outlines</td><td>NA</td></loq>	NA	0.0858	4.0Q-2 outlines	NA
Liver Polar Bear	970012	x	0,456		1	<loq (0.00683)<="" td=""><td>1</td><td></td></loq>	1	
· viar Sear	970201 980341	×	9_301	1		4.0Q (0.00683)	1	
	903341 692-PLBR-0033	x	9.678	1	1	<loq (0.00683)<="" td=""><td>1</td><td></td></loq>	1	
	9001271_B	x	0.471	1	1	<_OQ(0.00643)	1	
	900387LB	x	0.221			4.0Q(0.00683)	1	
	980390LB**	x	1.309	1	ł	<loq (0.00683)<="" td=""><td>I</td><td></td></loq>	I	
ļ	940563LA	x	1.438		1	<loq (0.00883)<="" td=""><td>1</td><td></td></loq>	1	
1	900565LB	x I	9.328	i	1	4.0Q (0.00683)	1	
	990112LB	X	R.175		1	4.00 (0.00683)	1	
	990592 <u>1</u> .A	XX	0.313	1	1	4.00 (0.00683)	1	
1	990594LB	x	0.356	1	1	<loq (0.00683)<="" td=""><td>1</td><td>1</td></loq>	1	1
	STOSSAL B	i x	0.436	1	1	4.00(0.0083)	1	
1	990600LB 990610LB	×	0.224	1		<loq (0.00083)<="" td=""><td>1</td><td> </td></loq>	1	
1	990652LC	x x	8.295	1		4.00 (0.00683)	1	
1	9906542.B	x	0.235	0.350	38.6	4.00 (0.00633)		NA
Liver	D0530	x	0.974	+		<.00 (0.00683) <.00 (0.0171)	0010	NA
Mink	D0566*	x	2.68	1		4.00 (0.0171)	1	1
1	D0590	x	2.38	1	1	4.00 (0.0171)	1	
	DOGIE	×	0.974	1	1	<loq (0.0171)<="" td=""><td>1</td><td>1</td></loq>	1	1
	D0630*	x	2.75	1		<loq (0.0171)<="" td=""><td>1</td><td>ł</td></loq>	1	ł
	D0684*	×	3.50	1		<loq (0.0171)<="" td=""><td>1</td><td></td></loq>	1	
	101000 101024	x	3.42	1	1	<.00 (0.0171)	1	ŀ
	Digs	x x	213	1		<loq (0.0171)<="" td=""><td>1</td><td>1</td></loq>	1	1
	D1092	x	3.20	1	1	<00 (0.0171)	1	ł
1	Ditio	x	3.35	1	1	<loq (0.0171)<="" td=""><td>1</td><td>1</td></loq>	1	1
1	D1134	Â	3.67		1		1	
	DIISOX*	x	1.96	1	1	<loq (0.0171)<br=""><loq (0.0171)<="" td=""><td>1</td><td>1</td></loq></loq>	1	1
	D1194	x x	1.54		1 .	<pre>4.00((0.0171) 4.00((0.0171))</pre>	1	
1	D119	x x	2.82	1	1	<loq (0.0171)<="" td=""><td>1</td><td>1</td></loq>	1	1
	DI344	x	3.42	1		4.00 (0.0171)	1	
	DISAN	×	1.21	1	35.0	<loq (0.0171)<="" td=""><td>1</td><td>NA</td></loq>	1	NA
	D1660*	×	3.66	2.63	0.919	4.00 (0.0171)	400	NA
1.0.0001	the second s				NE - Not Extracted		IPOS - Perdeoroo	
n (>50%) marrogate dev	ation and and and and and and and a data and a							
	ation was not achieved, used 499 ->99 trunction.				E - Lost during extraction		PFOSA - Pullaceo	octana sel ferenció
positive sealyte confirms Entered/Analyse: 12	tion was not achieved, used 499 -> 99 transition. 2/22/99, 12/28/99, 12/29/99, 12/20/99, 12/20/99, 12/28/99, 12/28/99, 12/29/99, 12/20/99, 12/20/09 1_AC						PFOSA - Perfecto PFHS - Perfecto POAA - Perfecto	estatus sel fonemid enancesificanis

ETS-8-7.0 Exact Version 5/95

OEN-030-liver.s.la

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فيك فساله ara/Versson. w/Aoalyst 'Aaalyst' instrument Soft Date of Estracti Date of Analysis Date of Data Re

Sample Data

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4

 CEN030
 MSU Exvironmental Samples

 Various
 Various

 Strate 45.0 & ETS-8-7.0

 Strate 203109, Aumite 002409

 Manahyua 13

 Johnson 203109, Aumite 002409

 Johnson 203109, MARCKUSER

 J21709, 122099, MARKUSER

 J27709, 122090, 122099, 122099, 01/03/00, 01/05/00

Gesup Dune	Sample #	Concentration of POAA	NOAA	RSD Std. Day.	Concentration of PFOSA	Mens PPOSA	RSD Sel, Dev.
		my/g or % Rec.	-11	MS/MSD RPD	upy or % Ret.		MIMMSD RP
Method Bits stiltered water	MSU12129-H2OBlk-anfiltered-5-1 MSU12129-H2OBlk-anfiltered-5-2	NA			NA		
CUMPED WINE	MSU12129-H2OBb -mfiltered-3-2 MSU12129-H2OBb -mfiltered-3-3	NA <loq (0.0719)<="" td=""><td></td><td>(</td><td>NA <loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>		(NA <loq (0.0188)<="" td=""><td></td><td></td></loq>		
ł	MSU12129-H2OBit-militared-5-4	4.00 (0.0719)			4L0Q (0.0134)		
	MSU12129-H2OBB:-mfiltered-5-5	E			E		
	MSU13129-H2082c-msShered-5-6	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBB	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (="" 0.0188)<="" td=""><td></td><td>NA</td></loq>		NA
Made d DO	MSU12129-H208ik-anfiltered-5-8	<loq [0.0719]<="" td=""><td>100</td><td>NA</td><td><loq (="" 0.01="" h)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	100	NA	<loq (="" 0.01="" h)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
Method Blk Filtered water	MSU12129-H2OBik-filtered-3-1 MSU12129-H2OBik-filtered-5-2	NA NA			NA NA		
	MSU12129-H2OBB-ditared-5-3	E			g		
1	MSU12129-H2OBB:- filmed-5-4	<loq (0.0719)<="" td=""><td></td><td></td><td><1.00 (0.0188)</td><td></td><td></td></loq>			<1.00 (0.0188)		
	MSU12129-H2OB0k-@tweed-5-5	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBik-filtered-5-6	<loq (0.0719)<="" td=""><td></td><td></td><td>4.0Q (0.0180)</td><td></td><td></td></loq>			4.0Q (0.0180)		
	MSU12129-H2OBit-filtered-5-7	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td></td><td>NA</td></loq>		NA
Matrix Bit	MSU12129-H2OBit-Gitered-5-8 FSH12129-LvrBit-3-1	4.00(00719)	400	NA	<pre><loq (0.0180)<="" pre=""></loq></pre>	4.00	NA
Fide Liver	FSH12129-LvrBik-5-2	<loq (0.0719)<br=""><loq (0.0719)<="" td=""><td></td><td>NA</td><td>4.00 (1.0188)</td><td></td><td>NA</td></loq></loq>		NA	4.00 (1.0188)		NA
	930H12129-Lvr80k-5-3	4.00 (0 0719)	40Q	NA	4.00 (0.0187)	4.00	NA
Matrix Blk	RBL12129-LvrBit-5-1	4.0Q (0.0719)			4.0Q (3.0188)		
Rebbit Liver	RBL12129-LvrBik-5+2	4.0Q (0.0719)			<loq (0.0188)<="" td=""><td>1</td><td></td></loq>	1	
	RBL12129-L+B&-3-3	≪LOQ (0.0719)			≺LCQ (0.9188)		
	RBL12129-LvrBit-3-4	4.0Q (0.0719)			4.0Q (0.0188)		
	RBL12129-LvxBitt-5-5* RBL12129-LvxBitt-5-6*	<loq (0.0719)<br=""><loq (0.0719)<="" td=""><td>اممه</td><td>NA NA</td><td><loq (0.0188)<br=""><loq (0.0188)<="" td=""><td>400</td><td>NA NA</td></loq></loq></td></loq></loq>	اممه	NA NA	<loq (0.0188)<br=""><loq (0.0188)<="" td=""><td>400</td><td>NA NA</td></loq></loq>	400	NA NA
oc	Mink Liver, D530, MS-5-1-1	1000 (0.0/19) 16%	~~~		43%		NA
250 ng/g	Minh Liver, D510, MSD-5-1-2*	\$4%	85%	3%	81%	62%	60%
	CSL-1999030-01-01-H3-3-1-2*	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%		
L	LWL-1999029-12-MSD-5-1-2*	14.5%	119%	9%.	73%	70%	8%
1	BTL-1999040-01-X4S-5-1-2* BTL-1999040-01-X4SD-5-1-2*	127%		æ	13%	1	***
<i>.</i> ∵ ⊢	BTL-199040-01-MSD-5-1-2* TNL-TU54-MS-5-1-2	120%	124%	~~	£1%. 59%	\$2%	3%
·.•	TNL-TU54-MISD-5-1-2	B	2.4%	NA	59% E	5976	NA
	FSL-7:95-MS-5-1	72%			76%		
	FSL-P295-MSD-5-1	80%	76%	10%	71%	73%	856
	PBL-900390LB-MS-5-1	85%			60%		
	PBL-900390LB-MSD-5-2*	129%	107%	41%	54%	\$7%	11%
1	GFL-KZCKDH-D1-MS-5-1-2	12%			71%		
	OFL-KZCKDM-D1-MSD-5-1- 1TL-LCPTR9900C-MS-5-1-2*	103%	27%	23%	12% 67%	77%	14%
	TTL-LCPTR9900C-MSD-3-1-2	64%	69%	13%	39%	43%	12%
	MTL-10Vancharve98-MS-5-1-2	100%	1 1		63%		12.7
	MTL-10Vanciesve98-MSD-5-1-2	75%	10%	29%	63%	63%	196
Liver	P283	4LOQ (0.0719)			<loq (0.01111)<="" td=""><td></td><td></td></loq>		
Northern Far Seal	P285**	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	P295	<loq (0.0719)<="" td=""><td>1 1</td><td></td><td><.OQ (0.0188)</td><td></td><td></td></loq>	1 1		<.OQ (0.0188)		
1	97 CU 02	NE			NE		
l l	98 CU KB 02 98 CU KB 03	<loq (0.0719)<="" td=""><td></td><td></td><td></td><td></td><td></td></loq>					
Í	96 CU KB 03 96 CU KB 07	<loq (0.0719)<br=""><loq (0.0719)<="" td=""><td>1 · I</td><td></td><td> <0.00 (0.0188) <0.00 (0.0188) </td><td></td><td></td></loq></loq>	1 · I		 <0.00 (0.0188) <0.00 (0.0188) 		
	98 CU KB 09	4.00 (0.0719)	1 1		4.00 (0.01MB)		
	96 CU KB 10	4LOQ (0.0719)	1		4.00 (0.0188)		
	98 CU KB 11	<loq (0.0719)<="" td=""><td>1 1</td><td></td><td><loq (="" 0.0158)<="" td=""><td></td><td>1</td></loq></td></loq>	1 1		<loq (="" 0.0158)<="" td=""><td></td><td>1</td></loq>		1
	50 CU KB 12	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0134)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0134)<="" td=""><td></td><td></td></loq>		
	SECUKE 13	<loq (0.0719)<="" td=""><td>1</td><td></td><td>4.0Q(0.0188)</td><td></td><td></td></loq>	1		4.0Q(0.0188)		
	SECUKB 14	<loq (0.0719)<="" td=""><td>1 1 1</td><td>NA NA</td><td></td><td>400</td><td>NA</td></loq>	1 1 1	NA NA		400	NA
Liver	96 CU KB 15 970012	4LOQ (0.0719) 4LOQ (0.0719)	4.00		4.0Q(0.0188) 4.0Q(0.0188)	4.0Q	NA
Poler Bear	970201	4.00 (0.0719)			4.0Q (0.0188)		
	900341	4.00 (0.0719)			4LOQ (0.0188)		
ļ	692-FLBR-0033	4.00 (0.0719)	1		4LOQ (0.0188)		
	900127LB	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq(0.0188)< td=""><td>ł</td><td> </td></loq(0.0188)<></td></loq>	1		<loq(0.0188)< td=""><td>ł</td><td> </td></loq(0.0188)<>	ł	
1	9801871.3	<loq (0.0719)<="" td=""><td>1</td><td>1</td><td>4.0Q(4.0130)</td><td>1</td><td>l</td></loq>	1	1	4.0Q(4.0130)	1	l
	980390LB+*	<loq (0.0719)<="" td=""><td>1</td><td>1</td><td>4.0Q(0.0188)</td><td>ł</td><td>1</td></loq>	1	1	4.0Q(0.0188)	ł	1
1	900563LA	4.00 (0.0719)	1		4.00(4.6183)	1	ł
	SHOULD B	4.00 (0.0719)	1	1	4.00(6.01M)	1	1
	9901 12LB 990397LA	<loq (0.9719)<br=""><loq (0.0719)<="" td=""><td>1</td><td>1</td><td><loq(0.0188) <loq(0.0188)< td=""><td>1</td><td>1</td></loq(0.0188)<></loq(0.0188) </td></loq></loq>	1	1	<loq(0.0188) <loq(0.0188)< td=""><td>1</td><td>1</td></loq(0.0188)<></loq(0.0188) 	1	1
	79039428	<loq (0.0719)<="" td=""><td>1</td><td>1</td><td>4L0Q(8.0186)</td><td> </td><td>1</td></loq>	1	1	4L0Q(8.0186)		1
	190596LB	4.00 (0.0719)	1		4.0Q(0.0188)	Į	1
1	990600L.B	<loq (0.0719)<="" td=""><td>1</td><td>1</td><td><loq (="" 0.0188)<="" td=""><td>1</td><td>1</td></loq></td></loq>	1	1	<loq (="" 0.0188)<="" td=""><td>1</td><td>1</td></loq>	1	1
	990610LB	<loq (0.0719)<="" td=""><td>1</td><td></td><td>4LOQ (0.0188)</td><td>1</td><td></td></loq>	1		4LOQ (0.0188)	1	
	990652LC	4.00 (0.0719)	1	NA	4.0Q(0.0188)	4	NA
	9906588,3	4.0Q (8.0719)	4.0Q	NA	4.0Q(0.0188)	900	NA
Léver Mink	D0530 D0566*	<loq (0.0719)<br=""><loq (0.0719)<="" td=""><td>1</td><td>1</td><td>0.0334 0.0266</td><td>1</td><td>1</td></loq></loq>	1	1	0.0334 0.0266	1	1
MI CAR	10390°	4.00 (0.0719)	1	1	0.0209		1
	20590	4.00 (6.0719)			0.0581		
1	D0630*	4.00 (0.0719)		1	0.0703	1	1
	D0684*	<loq (0.0719)<="" td=""><td>1</td><td>1</td><td>0.0612</td><td>1</td><td>1</td></loq>	1	1	0.0612	1	1
	D1000	4LOQ (0.0719)			0.0294	1	1
	D1024	<loq (0.0719)<="" td=""><td>1</td><td>ł</td><td><loq (0.0181)<="" td=""><td>1</td><td>1</td></loq></td></loq>	1	ł	<loq (0.0181)<="" td=""><td>1</td><td>1</td></loq>	1	1
	DIGSO	4.0Q (0.0719)		1	<loq (0.0188)<="" td=""><td>1</td><td>1</td></loq>	1	1
	D1092	<loq (0.0719)<="" td=""><td>1</td><td>Į.</td><td>√LOQ (0.0143)</td><td>1</td><td>1</td></loq>	1	Į.	√LOQ (0.0143)	1	1
	D1 110* D1 134	<loq (0.0719)<="" td=""><td></td><td>1</td><td>00367 4 00 00 00 00 00</td><td>1</td><td>1</td></loq>		1	00367 4 00 00 00 00 00	1	1
	D1134 D1150X*	4.00 (6.0719)		1		1	1
	D1150X*	<loq (0.0719)<br=""><loq (0.0719)<="" td=""><td></td><td>1</td><td>0.0340</td><td>1</td><td>1</td></loq></loq>		1	0.0340	1	1
	DIIS	4.00 (0.0719)		l	0.0242	1	1
	D1244	4.00 (0.0717)	1	1	<loq (0.0122)<="" td=""><td></td><td>1</td></loq>		1
4	D1248	<loq (0.0717)<="" td=""><td></td><td>NA</td><td>0.0345</td><td>1</td><td>0.5</td></loq>		NA	0.0345	1	0.5
	D1660*	1.00 (0.0719)	⊲.00	NA	0.0621	0.0442 - 5 outliers	0.020
ligh (>50%) surregate de				NE - Not Extracted		PFOS - Perflecrecote	
againing analyte confirm	ation was not achieved, used 499 -> 99 transition.			E - Lout during extra		PPOBA - Pedeorood	
ne Entered/Analyst: 1	2/22/99, 12/28/99, 12/29/99, 12/30/99, 1/12/00, 01/17/00, 01/11/00, 01/19/00, 01/28/00, LAC			NA - Not Applicable LOQ - Limit of Ques		P7H3 - Perfleorohea POAA - Perfleorocci	

ETS-8-7.0 Easel Version 5/95

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GEN303 MSU Environmental Samplet Various ETS-6-6.0 & ETS-6-7.0 Soup 020199, Amelia 062498 Masslynx 3.3 12/1299 SAL/KK/SRP/CSH 12/1799, 12/2099, 12/2199, 12/2999, 01/03/00, 01/06/00, 02/01/00 IAS/MMH/MEE 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

GEN030 MSU Environmental Samples

Study: Product Number(Test Substance): Matrix: Method/Revision: Assilytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Analysis/Analysi: Date of Analysis/Analysi: Date of Data Reduction/Analysi: Sample Data

Group	Sample#	PFOS	Concentration	Menn	RSD	Concentration	Mean	RSD
Dose		Verified	of PFOS	PFOS	Std. Dev.	of PFHS	PFHS	Std. Dev.
			ug/g or % Rec.	====	MS/MSD RPD	ug/g or % Rec.		MS/MSD RPD
Method Blk	MSU12129-H2OBlk-onfiltered-5-1	NA	NA		1	NA		
infiltered water	MSU12129-H2OBlk-unfiltered-5-2	NA	NA			NA		
	MSU12129-H2OB0k-unfiltered-5-3	NA	<loq (0.0347)<="" td=""><td></td><td>1</td><td><loq (0.00683)<="" td=""><td></td><td></td></loq></td></loq>		1	<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBik-unfiltered-5-4	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBik-unfiltered-5-5	NA	E COMPANY			<loq (0.0171)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBik-unfilecred-5-6	NA	<loq (0.0696)<="" td=""><td></td><td>NA</td><td><loq (0.0171)<="" td=""><td></td><td></td></loq></td></loq>		NA	<loq (0.0171)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBik-unfiltered-5-7	NA NA	<loq (0.0696)<br=""><loq (0.0696)<="" td=""><td>4.00</td><td>NA</td><td><loq (0.0171)<="" td=""><td><l00< td=""><td>NA NA</td></l00<></td></loq></td></loq></loq>	4.00	NA	<loq (0.0171)<="" td=""><td><l00< td=""><td>NA NA</td></l00<></td></loq>	<l00< td=""><td>NA NA</td></l00<>	NA NA
Method Blk	MSU12129-H2OBik-unfiltered-5-8 MSU12129-H2OBik-filtered-5-1	NA	NA NA	~~~~		NA		n A
Filtered water	MSU12129-H2OBik-filered-5-2	NA	NA			NA		
Pillered water	MSU12129-H2OBit-Ghard-5-3	NA	B			B		
	MSU12129-H2OBik-filtered-5-4	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OB1k-filtered-5-5	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00683)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBIt-filtered-5-6	NA	<loq (0.0696)<="" td=""><td></td><td></td><td><loq (0.0171)<="" td=""><td>1</td><td></td></loq></td></loq>			<loq (0.0171)<="" td=""><td>1</td><td></td></loq>	1	
	MSU12129-H2OBlk-filtered-5-7	NA	<loq (0.0696)<="" td=""><td></td><td>NA</td><td><loq (0.0171)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0171)<="" td=""><td></td><td>NA</td></loq>		NA
	MSU12129-H2OBit-filured-5-4	NA	<loq (0.0696)<="" td=""><td>₹00</td><td>NA</td><td><loq (0.0171)<="" td=""><td>⊲.00</td><td>NA</td></loq></td></loq>	₹00	NA	<loq (0.0171)<="" td=""><td>⊲.00</td><td>NA</td></loq>	⊲.00	NA
Matrix Blk	FSH12129-LvrBlk-5-1	NA	0.0305			<loq (0.0171)<="" td=""><td></td><td>4.</td></loq>		4.
Fish Liver	PSH12129-LwBlk-5-2	NA	0.0331		32.1	<loq (0.0171)<="" td=""><td></td><td>NA</td></loq>		NA
1010.00	FSH12129-L+rBlk-5-3	NA	0.0170	0.0269	0.00862	<loq (0.0171)<="" td=""><td><1.00</td><td>NA</td></loq>	<1.00	NA
Matrix Blk	RBL12129-LvrBlk-5-1	NA	<loq (0.0347)<="" td=""><td></td><td></td><td><loq (0.00613)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.00613)<="" td=""><td></td><td></td></loq>		
Rabbit Liver	RBL12129-LvrBlt-5-2	NA	<loq (0.0347)<="" td=""><td>1</td><td></td><td><loq (0.00643)<="" td=""><td></td><td></td></loq></td></loq>	1		<loq (0.00643)<="" td=""><td></td><td></td></loq>		
	RBL12129-LvrBlk-5-3	NA	<loq (0.0696)<="" td=""><td>1</td><td></td><td><loq (0.0171)<="" td=""><td>I</td><td></td></loq></td></loq>	1		<loq (0.0171)<="" td=""><td>I</td><td></td></loq>	I	
	- RBL12129-LvrBlk-5-4	NA	<loq (0.0696)<="" td=""><td>1</td><td></td><td><loq (0.0171)<="" td=""><td>1</td><td>•</td></loq></td></loq>	1		<loq (0.0171)<="" td=""><td>1</td><td>•</td></loq>	1	•
	RBL12129-LwBB-5-5*	NA	<loq (0.0696)<="" td=""><td> </td><td>NA</td><td><loq (0.0171)<="" td=""><td>1</td><td>NA</td></loq></td></loq>		NA	<loq (0.0171)<="" td=""><td>1</td><td>NA</td></loq>	1	NA
	RBL12129-LwBlk-5-6*	NA	<loq (0.0696)<="" td=""><td>4.00</td><td>NA</td><td><loq (0.0171)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	4.00	NA	<loq (0.0171)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
QC	Mink Liver, D530, MS-5-1-1	NA	145%			61%		
250 ng/g	Mink Liver, D530, MSD-S-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%	456
	LWL-1999029-12-MS-5-1-2*	NA	321%			57%		
	LWL-1999029-12-MSD-5-1-2*	NA	287%	304%	11%	68%	63%	28%
	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	65%			55%		
•	TNL-TUS4-MSD-5-1-2	NA	Ē	68%	NA	E	55%	NA
	FSL-P295-MS-5-1	NA	91%			97%		
	FSL-P295-MSD-5-1	NA	\$6%	\$\$%	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-S-1-2	NA	93%			82%		
	GFL-KZCKDM-D1-MSD-5-1-	NA	105%	99%	12%	89%	\$6%	
	TTL-LCPTR99503C-MS-5-1-2*	NA	90%			66%		
	TTL-LCPTR99503C-MSD-5-1-2	NA	100%	95%	11%	72%	69%	
	MTL-10Vancleave98-MS-5-1-2	NA	79%			74%		13%
	MTL-10Vancleave98-MSD-5-1-2	NA	19%	84%	11%	84%	79%	1376
Liver	P, #10, Vancieave 98	NA	0.0601			<loq (0.00683)<="" td=""><td></td><td>2</td></loq>		2
Map Turtle	F, #09, Vanciesve 98	NA	0.0514			<loq (="" 0.00683)<="" td=""><td></td><td>•</td></loq>		•
	F, #02, Lecksville 98	NA	0.0739			<loq (0.00683)<="" td=""><td></td><td></td></loq>		
	P, #06, Looksville 99	NA	0.0394			<loq (0.00683)<br=""><loq (0.00683)<="" td=""><td></td><td></td></loq></loq>		
	M. (-1)	NA	0.179	1		<loq (0.00683)<="" td=""><td></td><td></td></loq>		
Liver	F, (89, 8912) LCPTR 9503C	NA NA	4.00 (0.0347)		1	<loq (0.00643)<="" td=""><td></td><td></td></loq>		
Terrapin	LCPTR 9509C	NA	4.00 (0.0347)	1	0.730	<loq (0.00683)<="" td=""><td>1 1</td><td>NA</td></loq>	1 1	NA
rentha	LCPTR 9505C	NA	<.00(0.0347)	0.188 - 3 outliers	0.257	<loq (0.00683)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
Liver	TU25*	+ x	<.00((0.0696)			<pre>d.0Q(0.0171)</pre>		
	1025*	l î	<loq (0.0696)<="" td=""><td>1</td><td>1</td><td><loq (0.0171)<="" td=""><td>1</td><td></td></loq></td></loq>	1	1	<loq (0.0171)<="" td=""><td>1</td><td></td></loq>	1	
Tuna	104*	Î	<.00 (0.0696)	1	1	<loq (0.0171)<="" td=""><td></td><td></td></loq>		
	TUIS	Â	<loq (0.0696)<="" td=""><td></td><td></td><td><loq (0.0171)<="" td=""><td></td><td>•</td></loq></td></loq>			<loq (0.0171)<="" td=""><td></td><td>•</td></loq>		•
	TU49	NA NA	<loq (="" 0.0696)<="" td=""><td>1</td><td></td><td><loq (0.0171)<="" td=""><td></td><td></td></loq></td></loq>	1		<loq (0.0171)<="" td=""><td></td><td></td></loq>		
	TUS4*	x	<loq (0.0696)<="" td=""><td>1</td><td></td><td><loq(0.0171)< td=""><td></td><td></td></loq(0.0171)<></td></loq>	1		<loq(0.0171)< td=""><td></td><td></td></loq(0.0171)<>		
	TUSE	NA NA	<loq (="" 0.0696)<="" td=""><td></td><td>I</td><td><loq(0.0171)<="" td=""><td>1</td><td></td></loq(></td></loq>		I	<loq(0.0171)<="" td=""><td>1</td><td></td></loq(>	1	
	TU63*	x	<loq (0.0696)<="" td=""><td></td><td>1</td><td><loq(0.0171)< td=""><td></td><td> i '</td></loq(0.0171)<></td></loq>		1	<loq(0.0171)< td=""><td></td><td> i '</td></loq(0.0171)<>		i '
	τυ66	Â	0.00698	1		<loq(0.0171)< td=""><td>1</td><td></td></loq(0.0171)<>	1	
	TUM	NA	<loq (="" 0.0696)<="" td=""><td>1</td><td></td><td><loq(0.0171)< td=""><td></td><td></td></loq(0.0171)<></td></loq>	1		<loq(0.0171)< td=""><td></td><td></td></loq(0.0171)<>		
	TUBS	x	<loq (0.00696)<="" td=""><td>1</td><td>NA</td><td><loq(0.0171)< td=""><td></td><td>NA</td></loq(0.0171)<></td></loq>	1	NA	<loq(0.0171)< td=""><td></td><td>NA</td></loq(0.0171)<>		NA
	TU90	NA	<loq (="" 0.0696)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>NA</td><td><loq(0.0171)< td=""><td><.00</td><td>NA</td></loq(0.0171)<></td></loq></td></loq>	<loq -="" 1="" outlier<="" td=""><td>NA</td><td><loq(0.0171)< td=""><td><.00</td><td>NA</td></loq(0.0171)<></td></loq>	NA	<loq(0.0171)< td=""><td><.00</td><td>NA</td></loq(0.0171)<>	<.00	NA
Liver	KZCKDM-D1	NA	<loq (0.0347)<="" td=""><td>1</td><td>1</td><td><loq (="" 0.00683)<="" td=""><td>T</td><td>· · · · · · · · · · · · · · · · · · ·</td></loq></td></loq>	1	1	<loq (="" 0.00683)<="" td=""><td>T</td><td>· · · · · · · · · · · · · · · · · · ·</td></loq>	T	· · · · · · · · · · · · · · · · · · ·
Green Frog	KZCKDM-D2	x	0.285	1		<loq (="" 0.00683)<="" td=""><td>1</td><td></td></loq>	1	
GROUP FIVE	Pool of 4	ŇĂ	<loq (0.0347)<="" td=""><td>1</td><td>NA</td><td><loq (="" 0.00683)<="" td=""><td>ļ</td><td>NĂ</td></loq></td></loq>	1	NA	<loq (="" 0.00683)<="" td=""><td>ļ</td><td>NĂ</td></loq>	ļ	NĂ
	SJ6001	x	<loq (0.0347)<="" td=""><td><loq -="" 1="" outlier<="" td=""><td>- NA</td><td><loq (="" 0.00683)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq></td></loq>	<loq -="" 1="" outlier<="" td=""><td>- NA</td><td><loq (="" 0.00683)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	- NA	<loq (="" 0.00683)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
gh (>50%) surrogate de	······································	<u> </u>		1	NE = Not Expacted		PFOS = Perfuoroc	
TH (~ 20.00) SOLLONGING OC	* 1944-04128				E = Lost during extraction			octane sulfonntoide
Entered Analyst:	12/22/99, 12/28/99, 12/29/99, 12/30/99,				NA = Not Applicable		PFHS = Parthuorol	
					· · · · · · · · · · · · · · · · · · ·			octanosie

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Study: Product Number(Test Substance): Matrix: Matrix: Method/Revision: Analytical Equipment System Number: Instrument Software/Version: Date of Extraction/Analyst: Date of Data Reduction/Analyst. Sample Data

Miscellaneous Liver

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GEN030 MSU Environmental Samples Various Various Various ETS-4-6.0 & ETS-1-7.0 Soup 020199, Amelia 062498 Masslysex 3.3 12/12/99 SAL/KK/SRP/CSH 12/17/99, 12/20/99, 12/29/99, 01/03/00, 01/06/00, 02/01/00 LAS/MMH/MEE 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/LAS/MEE

Group Dose	Sample #	of POAA	Mean POAA	RSD Std. Dev.	Concentration of PFOSA	Mean	RSD
		ug/g or % Rec.		MS/MSD RPD		PFOSA	Std. Dev.
Method Bilk	MSU12129-H2OB1k-unfiltered-5-1	NA NA		MENNED KPU	ug/g or % Rec. NA		MS/MSD RPD
infiltered water	MSU12129-H2OBTk-unfiltered-5-2	NA					
	MSU12129-H2OBlk-unfiltered-5-3	<loq (0.0719)<="" td=""><td></td><td></td><td>NA <loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			NA <loq (0.0188)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBIk-unfiltered-5-4	<loq (0.0719)<="" td=""><td></td><td>1</td><td></td><td></td><td></td></loq>		1			
	MSU12129-H2OBIk-unfiltered-5-5	-coq(a.c.())		1	<loq (0.0188)<br="">E</loq>		
	MSU12129-H2OBIk-unfiltered-5-6	<loq (0.0719)<="" td=""><td></td><td></td><td>-</td><td></td><td>•</td></loq>			-		•
	MSU12129-H2OBIk-unfiltered-S-7	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<br=""><loq (0.0188)<="" td=""><td></td><td></td></loq></loq></td></loq>		NA	<loq (0.0188)<br=""><loq (0.0188)<="" td=""><td></td><td></td></loq></loq>		
	MSU12129-H2OBIk-unfiltered-5-8	<loq (0.0719)<="" td=""><td>4.00</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td><l0q< td=""><td>NA</td></l0q<></td></loq></td></loq>	4.00	NA	<loq (="" 0.0188)<="" td=""><td><l0q< td=""><td>NA</td></l0q<></td></loq>	<l0q< td=""><td>NA</td></l0q<>	NA
Method Blk	MSU12129-H2OBlk-filtered-5-1	NA	~~~~		NA NA		NA
Filtered water	MSU12129-H2OBlk-filtered-5-2	NA		1	NA		
	MSU12129-H2OBlk-filtered-5-3	E			R		
	MSU12129-H2OBik filtered-5-4	4.0Q (0.0719)			<loq (="" 0.0111)<="" td=""><td></td><td></td></loq>		
	MSU12129-H2OBik-filtered-5-5	<loq (0.0719)<="" td=""><td></td><td></td><td>4LOQ (0.0188)</td><td></td><td></td></loq>			4LOQ (0.0188)		
	MSU12129-H2OBIL filtered-5-6	<loq (0.0719)<="" td=""><td></td><td></td><td>4.0Q (0.0188)</td><td></td><td></td></loq>			4.0Q (0.0188)		
	MSU12129-H2OB&-filmed-5-7	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (="" 0.0188)<="" td=""><td></td><td>NA</td></loq>		NA
	MSU12129-H2OBik-filtered-5-8	<loq (0.0719)<="" td=""><td> ⊲.00</td><td>NA</td><td><loq (0.0114)<="" td=""><td>⊲.00</td><td>NA</td></loq></td></loq>	⊲.00	NA	<loq (0.0114)<="" td=""><td>⊲.00</td><td>NA</td></loq>	⊲.00	NA
Matrix Blk	FSH12129-LvrBDc-5-1	COQ (0.0719)			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
Fish Liver	FSH12129-Lvr#11-5-2	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (0.0160)<="" td=""><td></td><td>NA</td></loq></td></loq>		NA	<loq (0.0160)<="" td=""><td></td><td>NA</td></loq>		NA
	FSH12129-Lvr81k-5-3	<loq (0.0719)<="" td=""><td> ⊲∞ </td><td>NA</td><td><loq (0.0184)<="" td=""><td>⊲.00</td><td>NA</td></loq></td></loq>	⊲ ∞	NA	<loq (0.0184)<="" td=""><td>⊲.00</td><td>NA</td></loq>	⊲.00	NA
Matrix Blk	RBL12129-LvrBlk-5-1	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td>140</td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td>140</td></loq>		140
Rabbit Liver	RBL12129-LvrBlk-5-2	<loq (0.0719)<="" td=""><td> </td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	RBL12129-LvrBik-5-3	4.0Q (0.0719)			<loq (0.0188)<="" td=""><td> </td><td></td></loq>		
	RBL12129-LvrBik-5-4	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td> </td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td> </td><td></td></loq>		
	RBL12129-LvrBik-S-S*	<loq (0.0719)<="" td=""><td></td><td>NA</td><td><loq (0.0188)<="" td=""><td> </td><td>NA</td></loq></td></loq>		NA	<loq (0.0188)<="" td=""><td> </td><td>NA</td></loq>		NA
	RBL12129-LvrBlk-5-6*	4LOQ (0.0719)	⊲.0 Q	NA	<loq (="" 0.0188)<="" td=""><td> ⊲.00</td><td>NA</td></loq>	⊲.00	NA
QC	Mink Liver, D530, MS-5-1-1	\$6%			43%		
250 ng/g	Mink Liver, D530, MSD-5-1-2*	84%	85%	3%	\$1%	62%	60%
	CSL+1999030-03-01-MS-5-1-2*	131%			64%	<u>├──</u>	
	CSL-1999030-03-01-MSD-5-1-2*	111%	121%	17%	52%	58%	21%
	LWL-1999029-12-MS-5-1-2*	133%	1		77%		
	LWL-1999029-12-MSD-5-1-2*	145%	139%	9%	\$3%	80%	7%
	BTL-1999040-01-MS-5-1-2*	127%			94%	<u> </u>	
	BTL-1999040-01-MSD-5-1-2*	120%	124%	6%	92%	93%	3%
	TNL-TU54-MS-5-1-2	84%			71%	t	
	TNL-TU54-MSD-5-1-2	E	84%	NA	E	71%	NA
	FSL-P295-MS-5-1	72%	1		87%		
	FSL-P295-MSD-5-1	80%	76%	10%	82%	85%	7%
	PBL-980390LB-MS-5-1	85%	1		71%	1	•
	PBL-980390LB-MSD-5-2*	129%	107%	41%	65%	68%	9%
	GFL-KZCKDM-DI-MS-5-1-2	82%	1		\$2%		
	GFL-KZCKDM-D1-MSD-5-1-	103%	92%	23%	93%	88%	12%
	TTL-LCPTR99503C-MS-5-1-2*	73%	1		78%		
	TTL-LCPTR99503C-MSD-5-1-2	64%	69%	13%	71%	74%	10%
	MTL-10Vanciesve98-MS-5-1-2	100%			74%		
f in an	MTL-10Vancleave91-MSD-5-1-2	75%	88%	29%	74%	74%	0%
Liver	F, #10, Vancleave 98	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1		<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
Map Turtle	F, #09, Vancleave 98	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (0.01\$\$)<="" td=""><td>I I</td><td></td></loq></td></loq>			<loq (0.01\$\$)<="" td=""><td>I I</td><td></td></loq>	I I	
	F, #02, Locksville 98	<loq (0.0719)<="" td=""><td></td><td>1</td><td><loq (0.0188)<="" td=""><td> </td><td></td></loq></td></loq>		1	<loq (0.0188)<="" td=""><td> </td><td></td></loq>		
	F, #06, Leeksville 99	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (0.0188)<="" td=""><td>I </td><td></td></loq></td></loq>			<loq (0.0188)<="" td=""><td>I </td><td></td></loq>	I	
	M, (-1)	<loq (0.0719)<="" td=""><td>1</td><td>ļ </td><td><loq (0.0188)<="" td=""><td>1 1</td><td></td></loq></td></loq>	1	ļ	<loq (0.0188)<="" td=""><td>1 1</td><td></td></loq>	1 1	
. .	F, (89, 8912)	<loq (0.0719)<="" td=""><td>1</td><td>1</td><td><loq (0.0188)<="" td=""><td>1</td><td></td></loq></td></loq>	1	1	<loq (0.0188)<="" td=""><td>1</td><td></td></loq>	1	
Liver	LCPTR 9503C	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1		<loq (0.0188)<="" td=""><td></td><td></td></loq>		
Tempin	LCPTR 9504C	<loq (0.0719)<="" td=""><td>1</td><td>NA</td><td><loq (0.0183)<="" td=""><td>1</td><td>NA</td></loq></td></loq>	1	NA	<loq (0.0183)<="" td=""><td>1</td><td>NA</td></loq>	1	NA
	LCPTR 9505C	<loq (0.0719)<="" td=""><td><u> ₹00</u></td><td>NA</td><td><loq (0.0133)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	<u> ₹00</u>	NA	<loq (0.0133)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
Liver	TU25*	<loq (0.0719)<="" td=""><td></td><td></td><td></td><td></td><td></td></loq>					
Tuna	TU34*	<loq (0.0719)<="" td=""><td>1</td><td></td><td>4.0Q (0.0188)</td><td></td><td></td></loq>	1		4.0Q (0.0188)		
	TU41*	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (0.0158)<="" td=""><td> </td><td></td></loq></td></loq>	1		<loq (0.0158)<="" td=""><td> </td><td></td></loq>		
	TU48*	<loq (0.0719)<="" td=""><td>1</td><td>j l</td><td><loq (0.0188)<="" td=""><td></td><td></td></loq></td></loq>	1	j l	<loq (0.0188)<="" td=""><td></td><td></td></loq>		
	TU49	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0133)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0133)<="" td=""><td></td><td></td></loq>		
	TU\$4*	<loq (0.0719)<="" td=""><td>1</td><td>1</td><td><loq (0.0138)<="" td=""><td></td><td></td></loq></td></loq>	1	1	<loq (0.0138)<="" td=""><td></td><td></td></loq>		
	TUSB	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (0.0158)<="" td=""><td></td><td></td></loq></td></loq>	1		<loq (0.0158)<="" td=""><td></td><td></td></loq>		
	TU63*	<loq (0.0719)<="" td=""><td>1</td><td>1</td><td><loq (0.0158)<="" td=""><td></td><td></td></loq></td></loq>	1	1	<loq (0.0158)<="" td=""><td></td><td></td></loq>		
	TU66	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (="" 0.01="" 88)<="" td=""><td></td><td></td></loq></td></loq>	1		<loq (="" 0.01="" 88)<="" td=""><td></td><td></td></loq>		
	TU84	<loq (0.0719)<="" td=""><td>1</td><td></td><td><loq (0.0158)<="" td=""><td>I </td><td></td></loq></td></loq>	1		<loq (0.0158)<="" td=""><td>I </td><td></td></loq>	I	
	TUSS	<loq (0.0719)<="" td=""><td>1</td><td>NA</td><td><loq #)<="" (0.01="" td=""><td>j l</td><td>NA</td></loq></td></loq>	1	NA	<loq #)<="" (0.01="" td=""><td>j l</td><td>NA</td></loq>	j l	NA
	TU90	<loq (0.0719)<="" td=""><td>4.00</td><td>NA</td><td><loq (="" 0.0188)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	4.00	NA	<loq (="" 0.0188)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
Liver	KZCKDM-D1	<loq (0.0719)<="" td=""><td>T</td><td>1</td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>	T	1	<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
Green Frog	KZCKDM-D2	<loq (0.0719)<="" td=""><td></td><td></td><td><loq (="" 0.0188)<="" td=""><td></td><td></td></loq></td></loq>			<loq (="" 0.0188)<="" td=""><td></td><td></td></loq>		
	Pool of 4	<loq (0.0719)<="" td=""><td>1</td><td>NA</td><td><loq (0.0118)<="" td=""><td></td><td>NA</td></loq></td></loq>	1	NA	<loq (0.0118)<="" td=""><td></td><td>NA</td></loq>		NA
	SJ0001	<loq (0.0719)<="" td=""><td>400</td><td>NA .</td><td><loq (0.0188)<="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	400	NA .	<loq (0.0188)<="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
	stione			NB = Not Extracted		PPOS - Perfuon	
(>50%) surrogate devi	-504g						
(>50%) surrogate devi				E = Lost during extraction			rooctane sulfonemide
(>50%) surrogate devi atered/Analyst:	12/22/99, 12/28/99, 12/29/99, 12/30/99,						rooctane sulfonamide

ETS-8-7.0 Excel Version 5/95

GEN-030-liver.xla

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Study: et Number(Test Substance): Fred Matrix: Method/Revisi Analytical Equipment System Instrument Software/Version: al System N Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

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Various Livers Group

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FACT-GEN-033

GEN033, MSU - Liver Samples NA Various livers - Unextracted Curves 8TS-8-6.0 and 8TS-8-7.0 Devey 070799, Amelia 062498
 Dareg 070/99, Affeste 02495
 Junpan

 Massiynx 3.3
 Y-intercept:

 03/1400 SAL/CSH/KKK
 03/1400 SAL/CSH/KKK

 03/16/00, 03/17/00, J/19/00, 03/20/00, 03/21/00, 03/29/00, 04/07/00 IAS/MMH

 03/20/00, 03/22/00, 03/20/00, 03/21/00, 03/29/00, 04/01/00 IAS/MMH

R-Squa Slope: red Value:

See Attach See Attach See Attach mis

RSD

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Concentration Mean RSD Concentration Mean

Dees		of PFOS	PFOS	Std. Dev.	of POAA	POAA	Std. Dev.
		ug/g or % Rec	*S/E	MS/MSD RPD	ug/g or % Rec	-1/2	MS/MSD RPD
Mink Liver	(23 (45)	0.801			< LOQ (0.0359 ug/g)		
	C26 (44)	0.443	1		< LOQ (0.0359 ug/g)		
· •	C27 (49)	0.145	1		< LOQ (0.0359 ug/g)		
1	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	1		< 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)		
	. 121 (57)	1.03			< LOQ (0.0359 ug/g)		
	F24 (58)	0.868					
		2.16	·		< LOQ (0.0359 ug/g)		
	P01~(60)				<1.00 (0.0359 ug/g)		
1	P03 (64)	4.80			< LOQ (0.0359 ug/g)		
1	P09 (61)	0.841			< LOQ (0.0359 wg/g)		
	\$11 (37)	0.902			< LOQ (0.0359 ug/g)	ł	•
	\$15 (41)	1.27			< LOQ (0.0359		
1	S18 (40)	1.99			< LOQ (0.0359 ug/g)		
	S19 (59)	2.68			< LOQ (0.0359 ug/g)	1	
I	\$25 (39)	0.509			< LOQ (0.0359 ug/g)	1	
	\$30 (36)	0,186			< LOQ (0.0559 ug/g)	1	
	\$35 (42)	0.0933			< LOQ (0.0359 ug/g)	ł	
	\$39 (38)	0.317	[1		< LOQ (0.0359 wg/g)	1	
	T01 (51)	0.633			< LOQ (0.0359 ug/g)		
	T04 (53)	1.35			< LOQ (0.0359 ug/g)		1
	T03 (52)	0.565			< LOQ (0.0359 w/g)	I.	
	V12 (62)	4.87			< LOQ (0.0359 ug/g)	1	
1	V03 (65)	1.52	1	103	< LOQ (0.0359 ug/g)		NA
	V06 (63)	3.65	1.23	1.27	< LOQ (0.0359 ug/g)	<.00	NA
Baikal Seat Liver	JOE (81)	0.0127	1	[< LOQ (0.0359 ag/g)		\ \
	J09 (87)	0.0228	{	1	< LOQ (0.0718 wg/g)		
	J10 (86)	<loq (0.0347="" g)<="" td="" ug=""><td></td><td></td><td>< LOQ (0.0715 ug/g)</td><td></td><td>1</td></loq>			< LOQ (0.0715 ug/g)		1
·	J12 (89)	0.0141		1	< LOQ (0.0359 ug/g)	1	
	J19 (84)	0.00931	1		< LOQ (0.0359 ug/g)	1	
	720 (88)	0.0154			< LOQ (0.0718 ug/g)	1	
	J24 (82)	<loq (0.0347="" g)<="" td="" ug=""><td>1</td><td></td><td>< LOQ (0.0715 m/g)</td><td>1</td><td>ì</td></loq>	1		< LOQ (0.0715 m/g)	1	ì
	J27 (83)	<loq (0.0347="" g)<="" m="" td=""><td></td><td>1</td><td>< LOQ (0.0718 mp/g)</td><td>1</td><td></td></loq>		1	< LOQ (0.0718 mp/g)	1	
	J36 (85)	0.0146			< LOQ (0.0359 w/g)	1	· ·
	J37 (10)	<loq (0.0347="" g)<="" td="" w=""><td>1</td><td></td><td>< LOQ (0.0718 wg/g)</td><td></td><td></td></loq>	1		< LOQ (0.0718 wg/g)		
	R04 (69)	<loq (0.0347="" g)<="" td="" ug=""><td></td><td>1</td><td>< LOQ (0.0718 ug/g)</td><td></td><td>1</td></loq>		1	< LOQ (0.0718 ug/g)		1
	R13 (78)	0.0100	1		< LOQ (0.0359 ug/g)	1	
	R14 (74)	<loq (0.0347="" g)<="" td="" ug=""><td></td><td></td><td>< LOQ (0.0718 u/g)</td><td>1</td><td>Į</td></loq>			< LOQ (0.0718 u/g)	1	Į
		0.00608	1.				
	R16 (71)			4	<1.0Q (0.0359 ug/g)		
	R29 (72)	0.00795	1		< LOQ (0.0359 ug/g)	1	
	R42 (66)		1	1	< LOQ (0.0359 ug/g)	1	1
	R43 (73)	0.0156	1		< LOQ (0.0359 ug/g)	1	
	R45 (79)	0.00648	1	1	< LOQ (0.0359 mg/g)	1	1
	R46 (77)	0.00778	1		< LOQ (0.0359 mg/g)	1	
	B 47 (70)	<loq (0.0347="" g)<="" mg="" td=""><td>1</td><td></td><td>< LOQ (0.0718 ug/g)</td><td>1</td><td></td></loq>	1		< LOQ (0.0718 ug/g)	1	
	R54 (67)	4.0Q (0.0347 💓 g)	1	1	< LOQ (0.0718 ug/g)	1	1
	R55 (75)	0.0133	1		< LOQ (0.0718 wg/g)	1	
	R57 (76)	0.0158	1	33.7	< LOQ (0.0359 mg/g)	1	NA
	R.64 (68)	0.00786	0.0123	0.00416	< LOQ (0.0359 w/g)	400	NA
Acres Achieved and	LO4 (91)	4.0Q (0.0347 w/g)	T	NA	4.0Q (0.0718 m/m)		NA
Gangas Delphin Liver							

NA = Not Applicable

Date Entered/Analyst: Date Verified/Analyst:

03/28/00, 04/05/00, 04/07/00, 05/07/00 NDMER/LAC

Sample #

PFOSA = Perfluorooctanesulfo PFHS = Perfluorohecume sulfor

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POAA = Perfluorooctas

ETS-4-7.0 Excel Version 5/95

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GEN-053-liver.xls

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Study en(Text Substance): м. vision: M Analytical Equipment System Instrument Software/Version: nt System Number: Date of Extraction/Analyst: Date of Extraction/Analyst: Date of Analysis/Analyst: Date of Data Reduction/Analyst: Sample Data

Various Livers

GEN033, MSU - Liver Samples

See Below See Attachments See Attachments

See Attachmente

 GEN033, MSU - Liver Samples

 NA

 Various livers - Uscattracted Curves
 Filename:
 See Be

 ET3-8-0.0 and ET3-8-7.0
 R-Squared Value:
 See Al

 Davey 070799, Amelia 062498
 Slope:
 Sea Al

 Massiynu 3.3
 Y-Instructure
 See Al

 03/14/00
 SAL/CSH/KKK
 03/24/00, 03/25/00, 03/25/00, 03/25/00
 GA/07/00

 03/20/00, 03/22/00, 03/23/00, 03/24/00, 06/04/00, 04/11/00
 LAS/MMH
 03/20/00, 03/22/00, 03/23/00, 03/24/00, 06/04/00, 04/11/00

FACT-GEN-033

Group Dore	Sample #	Concentration of PPOSA	Mean PFOSA	RSD Std. Dev.	Concentration of PFHS	Mena PFHS	RSD Std. Dev.
		ug/g or % Rec	-9/2	MS/MSD RPD	ug/g or % Rec	-1/2	MS/MSD RP
Mint Liver	(23 (45)	0.0383			< LOQ (0.00683 ug/g)		In the complete For
	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	1	1
	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)	0.0628			< LOQ (0.00683 ug/g)		
	F15 (55)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	F19 (56)	< LOQ (0.0376 mg/g)			< LOQ (0.00683 mg/g)		
	721 (57)	0.0579			< LOQ (0.00683 ug/g)	1	
	F24 (58)	< LOQ (0.0376 mg/g)			< LOQ (0.00683 ug/g)		
	101 (60)	0.551			0.0315		
	103 (64)	0.590			0.0652		
	P09 (61)	< LOQ (0.0376 w/g)			< LOQ (0.00683 ug/g)		÷.
	\$11 (37)	< LOQ (0.0376 w/g)			< LOQ (0.00683 mg/g)		د بذ
	515 (41)	< LOQ (0.0376 mg/g)	1		< LOQ (0.00683 ug/g)		
	S18 (40)	0.0414			< LOQ (0.00683 mg/g)		
	\$19 (59)	0.132			0.0102		
	525 (39)	< LOQ (0.0376 wg/g)			< LOQ (0.00683 mg/g)		
•	\$30 (36)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 w/g)		
•	A 535 (62)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 wg/g)		
	339 (38) TDI (51)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	1	< LOQ (0.0376 ug/g)	1		< LOQ (0.00683 ug/g)		
ų.	T04 (53)	< LOQ (0.0376 ug/g)			0.0104		
	T03 (52)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	¥12 (62)	0.345			< LOQ (0.00683 ug/g)		
	V03 (65)	. 0.0594		NA	< LOQ (0.00683 mg/g)		NA
	Y06 (63)	0.0586	<loq -="" 10="" outliers<="" th=""><th>NA</th><th>< LOQ (0.00683 ug/g)</th><th><loq -="" 5="" outliers<="" th=""><th>NA</th></loq></th></loq>	NA	< LOQ (0.00683 ug/g)	<loq -="" 5="" outliers<="" th=""><th>NA</th></loq>	NA
Baikal Seal Liver	,04 (\$1)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 w/g)		<u>.</u>
	309 (87)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
•	J10 (\$6)	< LOQ (0.0376 ug/g)	1		< LOQ (0.00683 ug/g)		
•	J12 (89)	< LOQ (0.0376 wg/g)			< LOQ (0.00683 m/g)		
	J19 (84)	< LOQ (0.0376 mg/g)			< LOQ (0.00683 ug/g)		
	120 (65)	< LOQ (0.0376 ug/g)			<loq (0.00683="" g)<="" td="" ug=""><td>1</td><td>i.</td></loq>	1	i.
	724 (82)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		t.
	.27 (83)	< LOQ (0.0376 mg/g)			< LOQ (0.00683 ug/g)		
	,36 (85)	< LOQ (0.0376 ug/g)			< LOQ (0.00643 ug/g)		
		< LOQ (0.0376 ug/g)	1		< LOQ (0.00683 wg/g)		
	R04 (69)	< LOQ (0.0376 ug/g)	1	1	< LOQ (0.00683 ug/g)		
	R.13 (78)	< LOQ (0.0376 ug/g)		1	< LOQ (0.00683 ug/g)		
	R14 (74)	< LOQ (0.0376 ug/g)		ļ	< LOQ (0.00683 ug/g)		
	R16(71) .	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		
	129 (72)	< LOQ (0.0376 ug/g)	· · · · ·		< LOQ (0.00613 mg/g)		
	R42 (66)	< LOQ (0.0376 ug/g)			< LOQ (0.00613 ug/g)		
	R43 (73)	< LOQ (0.0376 ug/g)			< LOQ (0.00683 ug/g)		:
	R45 (79)	<loq (0.0376="" g)<="" td="" up=""><td>1</td><td>l</td><td>< LOQ (0.00613 ug/g)</td><td></td><td>·-</td></loq>	1	l	< LOQ (0.00613 ug/g)		·-
	R46 (77)	<loq (0.0376="" g)<="" td="" w=""><td>1</td><td>1</td><td>< LOQ (0.00643 mg/g)</td><td></td><td>•</td></loq>	1	1	< LOQ (0.00643 mg/g)		•
	1,47 (70)	<loq (0.0376="" g)<="" td="" w=""><td></td><td>1</td><td>< LOQ (0.00583 ug/g)</td><td>1</td><td></td></loq>		1	< LOQ (0.00583 ug/g)	1	
	R54 (67)	<loq (0.0376="" g)<="" td="" w=""><td></td><td></td><td>< LOQ (0.00683 w/g)</td><td>1</td><td></td></loq>			< LOQ (0.00683 w/g)	1	
	R55 (75)	<loq (0.0376="" g)<="" mg="" td=""><td>1</td><td></td><td>< LOQ (0.00613 m/g)</td><td>1</td><td></td></loq>	1		< LOQ (0.00613 m/g)	1	
	157 (76)	<loq (0.0376="" g)<="" td="" w=""><td>1</td><td>NA</td><td>< LOQ (0.00613 ug/g)</td><td></td><td>.</td></loq>	1	NA	< LOQ (0.00613 ug/g)		.
	R64 (68)	<loq (0.0376="" g)<="" td="" w=""><td>4.00</td><td>NA</td><td></td><td>1 400</td><td>NA</td></loq>	4.00	NA		1 400	NA
Ganges Dolphin Liver	L04 (91)	<loq (0.0376="" g)<="" td="" w=""><td>+</td><td>NA</td><td>< LOQ (0.00683 w/g)</td><td>Q</td><td>NA</td></loq>	+	NA	< LOQ (0.00683 w/g)	Q	NA
·····	L05 (90)	<loq (0.0376="" g)<="" m="" td=""><td>4.00</td><td>NA</td><td>< LOQ (0.00613 w/g) < LOQ (0.00613 w/g)</td><td></td><td>NA NA</td></loq>	4.00	NA	< LOQ (0.00613 w/g) < LOQ (0.00613 w/g)		NA NA
DQ - Limit of Quantitation							

LOQ = Limit of Quantitation NA = Not Applicable

Date Entered/Analyst: Date Verified/Analyst:

03/28/00, 04/05/00, 04/07/00, 05/07/00 MMH/LAC

PFOS = Perfluorooctanesulfanate PFOSA = Perfluorooctanesulfanate PFHS = Perfluorobecane sulfonate

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POAA = Perfluoroocta

BTS-8-7.0 Excel Version 5/95

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Date of Data Red Sample Data

GEN033, MSU - Liver Samples	
NA	
Verices livers - uncuracied curves	Filenane:
ETS-8-6.0 and ETS-8-7.0	R-Square
Davey 070799, Amaila 062498	Slope:
Massiyas 3.3	Y-Interce
99/14/00 SAL/CSHYKKK	

FACT-GEN-033

83/16/00, 03/17/00, 3/19/00, 03/20/00, 03/21/00, 03/29/00, 04/07/00 1AS/MMH 83/20/00, 03/22/00, 03/23/00, 03/24/00, 04/04/00, 04/11/00 IAS/MMH

See Attache See Attache

Group	Sample 8	Concentration	Mean	RSD	Concentration	Maga	110
Deet		of 2208	P706	Std. Dev.	erzona	7044	Shi Dev.
		agy or % Rec	-971	MS/MSD R/D	myg ar % Rac		MS/MSD R21
Comerent Liver	e ()) D	0.0432			0.0414		
Pennik, Adult	29# (S),F,A**	0.0565			0,102		
	30F (6),F,A	0.0485			0.0506		
	32 # (7), # ,A	0.0977			0.0897		
	34F (5),F,A**	0.0335		61.9	0.0297		56.9
	42F (L1),FA	0.150	0.0715	0.0442	0.143	0.0761	9,0432
Comerant Liver	77 (2),51	0.0913			0.0641		4002
Female, Juvenile	9F (3),FJ	0.468		113	0.444		. 115
	36F (10),F,J	0.0499	0.203	0.230	0.0467	0.192	0.219
Comerant Liver	227 (4),34,3	0.0433			0.0454	~	V.417
Main, Juvenile	33# (II),M_J	0.0316		17.2	0.0303		25.7
	44F (12),M_3	0.0337	0.0362	0.00623	0.0293	0.0350	0.00899
ettienese Deighin Liver	T15/91 (13)	0.181			<loq (0.0359="" m)<="" mm="" td=""><td></td><td>0.00077</td></loq>		0.00077
- 1	THE 91 (17)	0,296			<loq (0.0718="" g)<="" mg="" td=""><td></td><td></td></loq>		
	THE (14)	0.159			<loq (0.0718="" mmg)<="" td=""><td></td><td></td></loq>		
	TALLOG	0.425		44.5	<1.00 (0.0718 m/g)		
1	TUCT2 (15)	<.00 (0.00696 w/m)	0.258 - One Outline	0.119	<1.00 (0.0718 mm)	4.00	NA NA
Striped Deighin Liver	SCR02 (10)	0.161		w.117	<loq (0.0718="" m="" m)<="" td=""><td>4000</td><td>NA</td></loq>	4000	NA
	SCV1 (19)	0.0891			<loq (0.9718="" mm)<="" td=""><td></td><td></td></loq>		
	SCP03 (20)	0.0944		40.2	4.00 (0.0718 mm)		
	SCPO4 (31)	0.06/7	0,102	0.0410	<100 (0.0718 m/m)		NA NA
Weddell Seel Liver	W31 (22)	<1.00 (0.0347 m/p)	4.00	NA	<loq (0.0718="" m="" m)<="" td=""><td></td><td>NA NA</td></loq>		NA NA
Swardlick Liver	\$23 (23)	<loq (0.00006="" g)<="" td="" w=""><td>~~~~</td><td></td><td></td><td></td><td>NA</td></loq>	~~~~				NA
	324 (24)	<1.00 (0.00006 w/m)			4.0Q (0.0359 m/m)	1	•
	\$23 (25)	0.00714			4.00 (0.0359 ur/g)	!!	
1	\$32 (27)	<1.00 (0.00496 m/s)	1	NA	<0.00 (0.0359 up/g)		
	546 (26)	0.0133	<loq -="" outlines<="" td="" two=""><td>NA</td><td><loq (0.0359="" g)<br="" mg=""><loq (0.0359="" g)<="" mg="" td=""><td>400</td><td>NA</td></loq></loq></td></loq>	NA	<loq (0.0359="" g)<br="" mg=""><loq (0.0359="" g)<="" mg="" td=""><td>400</td><td>NA</td></loq></loq>	400	NA
Tunning Liver	TICOD	<1.00 (9.6347 - 2/2)	-cog- involutions				NA
· · · · · ·	TILFISE (SS)	0.0433			<pre><loq (0.0359="" (0.0715="" <loq="" g)="" g)<="" pre="" up=""></loq></pre>		
	17 (29)	0.0874					
<i>.</i>	T15 (20)	0.0568			<loq (0.0359="" g)<="" td="" w=""><td></td><td></td></loq>		
	T17 O1)	0.0491			<000 (0.0359 m/m)	1 · 1	
	T20 (32)	0.0207			<100 (0.0359 m/s)		
	T25 (30)	0.0560		46.2	<loq (0.0359="" g)<="" td="" up=""><td> </td><td></td></loq>		
1	T25 (34)	0.0250	0.0483 - One Outlier	9.0223	<loq (0.0715="" g)<="" td="" w=""><td>400</td><td>NA NA</td></loq>	400	NA NA
Backtalled Gall Liver	BHG01 (100)	0.292		0.1225	<00 (0.0359 unit)		~~
	BHG02 (101)	0.260			<loq (0.0359="" n)<="" td="" up=""><td>1 1</td><td></td></loq>	1 1	
	BHG03 (102)	0.148			<loq (0.0359="" g)<="" td="" w=""><td>1</td><td></td></loq>	1	
	BHOM (105)	0.503	1		<0.00 (0.0359 m/g)		• •
l	BHC05 (104)	0.271	1		<loq (0.0359="" g)<="" td="" up=""><td> </td><td></td></loq>		
l	BT(34305 (92)	0.0681	1		<loq (0.0359="" g)<="" td="" up=""><td> </td><td></td></loq>		
	BTG9906 (99)	9.107	1		<loq (0.0359="" g)<="" mm="" td=""><td> </td><td></td></loq>		
	BT(9319 (96)	0.215			<loq (0.0359="" g)<="" td="" us=""><td>1 1</td><td></td></loq>	1 1	
1	BT(9511 (96)	0.143			<loq (0.0359="" g)<="" td="" ug=""><td>1 </td><td></td></loq>	1	
1	BTG9512 (97)	0.0705			<loq (0.0359="" g)<="" td="" up=""><td>I. I</td><td></td></loq>	I. I	
1	BTG9401 (90)	0.126			<000 (0.0359 cm/g)		
	BTChongdol (94)	0.0737			<loq (0.0359="" g)<="" td="" w=""><td>1</td><td>•</td></loq>	1	•
	STGmadol (95)	0.0707	[.		<loq (0.0359="" m)<="" mm="" td=""><td></td><td></td></loq>		
	HIRG04 (106)	0.116	1	69.2	<loq (0.0359="" m)<="" td="" w=""><td> </td><td></td></loq>		
	HDLG09 (105)	0.0939	0.172	0,119	<loq (0.0359="" g)<="" td="" w=""><td>ا ممه ا</td><td>NA NA</td></loq>	ا ممه ا	NA NA

** NO PPOS confini LOQ - Limit of Qui NA - Not Applicable

Dete Entered/Analyst Dete Verified/Analyst

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03/28/00, 04/05/00, 04/07/00, 05/07/00 MMH/LAC

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PPOS - Perface POAA - Perface PPOSA - Perface PFHS - Perface

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Study: Product N riTest 5 Date Date of Analysis Date of Data Red Sample Data و بيد



GEN033, NSU - Liver Samples National livers - massimicated curves Plannamic ST3-4-60 and ST3-4-7.0 R. Squeered Values: Davey 070799, Annalia 062/98 Slopic Materiyas. 1.0 Y dimensionic 03/1 400 SAL/CSM/KCK
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Group Dest	Sample #	Concentration of PPOBA	Nona PPOSA	RSD Sid, Der.	Concentration of PPRS	Mess	RSD
Dese				MS/MSD RPD		PPHS	Std. Dev.
		upger % Ret		Maymau Kiro	ugig or % Rec		ME/MSD R
Comerant Liver	of (I),F.A	<loq (0.0376="" g)<="" td="" w=""><td>1</td><td></td><td>< LOQ (0.00683 ug/g)</td><td></td><td></td></loq>	1		< LOQ (0.00683 ug/g)		
Fomale. Adult	29 ₽ (5),F,A**	<loq (0.0376="" g)<="" td="" up=""><td>1</td><td></td><td>< LOQ (0.00613 ug/g)</td><td></td><td></td></loq>	1		< LOQ (0.00613 ug/g)		
	30F (6),P.A	<loq (0.0376="" g)<="" td="" w=""><td></td><td></td><td>< LOQ (0.00683 ug/g)</td><td></td><td></td></loq>			< LOQ (0.00683 ug/g)		
	32 F (7),F.A	<1.00 (0.0376 up/g)			< LOQ (0.00683 ug/g)		
	34F (9),F.A**	<loq (0.0376="" g)<="" td="" ug=""><td></td><td>NA</td><td>< LOQ (0.00683 ug/g)</td><td></td><td>NA</td></loq>		NA	< LOQ (0.00683 ug/g)		NA
	42F (11).F.A	<loq (0.0376="" g)<="" td="" w=""><td>40Q</td><td>NA</td><td>< LOQ (0.00683 ug/g)</td><td><.0Q</td><td>NA</td></loq>	40Q	NA	< LOQ (0.00683 ug/g)	<.0Q	NA
Comorant Liver	7 # (2),#J	0.0688			< LOQ (0.00683 vg/g)		
Female, Jevenile	9 F (0),FJ	<loq (0.0076="" g)<="" td="" ug=""><td></td><td>NA</td><td>< LOQ (0.00683 ug/g)</td><td></td><td>NA</td></loq>		NA	< LOQ (0.00683 ug/g)		NA
	348F (10),FJ	<loq (0.0376="" g)<="" td="" ug=""><td><loq -="" one="" outlier<="" td=""><td>NA</td><td>< LOQ (0.00683 wg/g)</td><td><∆oo</td><td>NA</td></loq></td></loq>	<loq -="" one="" outlier<="" td=""><td>NA</td><td>< LOQ (0.00683 wg/g)</td><td><∆oo</td><td>NA</td></loq>	NA	< LOQ (0.00683 wg/g)	<∆oo	NA
Comprised Liver	22# (4),M,J	<loq (0.9376="" g)<="" td="" ug=""><td></td><td></td><td>< LOQ (0.00683 ug/g)</td><td></td><td></td></loq>			< LOQ (0.00683 ug/g)		
Maie, Jevenile	33F (0),MJ	<loq (0.0376="" g)<="" td="" ug=""><td></td><td>NA</td><td>< LOQ (0.00683 up/g)</td><td></td><td>NA</td></loq>		NA	< LOQ (0.00683 up/g)		NA
	44F (12),MLI	<loq (0.0376="" g)<="" td="" ug=""><td>Q0,</td><td>NA</td><td>< LOQ (0.00681 ug/g)</td><td>⊲Ω⊘</td><td>NA</td></loq>	Q0,	NA	< LOQ (0.00681 ug/g)	⊲ Ω⊘	NA
Bettleness Delabin Liver	T591 (13)	0.224			< LOO (0.00683 up/s)		_
	THEFT (17)	0.358			< LOO (0.00683 up/g)		
	To61 (14)	0.129			< LOQ (0.00683 ug/s)		4.
	TALL (16)	0.129		<u> </u>	< LOO (0.00603 wa/a)		S NA
	TUXT2 (15)	9,115	6,191	0.103	< LOQ (0.00413 w/g)	4.00	NA
Strined Delphia Liver	SCP02 (19)	<0.00 (0.0376 1/2)			< LOO (0.00683 wa/s)		
	SCV1 (19)	<loq (0.0376="" g)<="" td="" w=""><td>•</td><td></td><td>< LOQ (0.00683 ug/g)</td><td></td><td></td></loq>	•		< LOQ (0.00683 ug/g)		
	90903 (20)	4.00 (0.076 m/s)		NA	< LOQ (0.00681 ug/g)	ŕ	NA
	SCP04 (21)	<loq (***))<="" (1.03%="" td=""><td><.0Q - One Outlier</td><td>NA</td><td>0.0270</td><td></td><td>NA</td></loq>	<.0Q - One Outlier	NA	0.0270		NA
Weddell Seet Liver	W\$1 (22)	4.00 (0.00% w/p)	400	NA 1	< LOQ (0.00683 up/p) -	400	NA
Swertige Liver	\$23 (23)	<			0.09954		TIA .
			1	í I		· ·	1.1
	\$24 (24)	<00 (8.0076 w/g)			and former of the		
•	SES (2.9)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td></td><td>< LOQ (0.00683 ug/g)</td><td></td><td></td></loq>			< LOQ (0.00683 ug/g)		
1	\$32 (27)	<loq (0.0376="" g)<="" td="" w=""><td><00⊳</td><td>NA NA</td><td>< LOQ (0.00683 w/h)</td><td></td><td>NA</td></loq>	<00⊳	NA NA	< LOQ (0.00683 w/h)		NA
	\$41 (24)	<00 (0.03% w/g)	1 400	NA	< LOQ (0.00583 mg/g)	<0.00.0m0mm	NA
Tunefish Liver	T1 (03)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td>1 1</td><td>< LOQ (0.00683 wg/g)</td><td></td><td></td></loq>		1 1	< LOQ (0.00683 wg/g)		
	TILF156 (05)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td>1 1</td><td>< LOQ (0.00683 wg/g)</td><td></td><td>i i</td></loq>		1 1	< LOQ (0.00683 wg/g)		i i
	T2 (29)	<loq (0.0576="" g)<="" td="" up=""><td></td><td>1 1</td><td>< LOQ (0.00683 ug/g)</td><td></td><td></td></loq>		1 1	< LOQ (0.00683 ug/g)		
•	T15 (28)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td>1 1</td><td>< LOQ (0.00663 wg/g)</td><td></td><td>1</td></loq>		1 1	< LOQ (0.00663 wg/g)		1
	T17(01)	<1.0Q (0.0376 ug/g)			< LOQ (0.00683 ug/g)		1
	T20 (32)	<loq (0.0376="" g)<="" td="" vg=""><td></td><td></td><td>< LOQ (0.00683 ug/g)</td><td>1</td><td>,</td></loq>			< LOQ (0.00683 ug/g)	1	,
	T23 (30)	<loq (0.0076="" g)<="" td="" w=""><td></td><td>NA</td><td>< LOQ (0.00683 wg/g)</td><td></td><td>NA</td></loq>		NA	< LOQ (0.00683 wg/g)		NA
	T25 (34)	<loq (0.0376="" g)<="" td="" ug=""><td>Q00</td><td>NA</td><td>< LOQ (0.00683 wg/g)</td><td>Q</td><td>. NA</td></loq>	Q00	NA	< LOQ (0.00683 wg/g)	Q	. NA
Blacktailed Gull Liver	211G01 (100)	<.0Q (0.0076 ug/g)			< LOQ (0.00683 wg/g)		
	BHBC02 (101)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td></td><td>< LOQ (0.00683 vg/g)</td><td></td><td>1</td></loq>			< LOQ (0.00683 vg/g)		1
	BH8303 (102)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td>1 1</td><td>< LOQ (0.00683 w/g)</td><td></td><td>\sim</td></loq>		1 1	< LOQ (0.00683 w/g)		\sim
	BHG04 (103)	<loq (0.0376="" g)<="" td="" w=""><td></td><td></td><td>< LOQ (0.00683 w/g)</td><td></td><td></td></loq>			< LOQ (0.00683 w/g)		
	BHQ05 (104)	<loq (0.0376="" g)<="" td="" wg=""><td>ļ</td><td></td><td>< LOQ (0.00683 ug/g)</td><td>1</td><td></td></loq>	ļ		< LOQ (0.00683 ug/g)	1	
	BTC9305 (92)	<loq (0.0376="" g)<="" td="" ug=""><td>1</td><td></td><td>< LOQ (0.00683 mg/g)</td><td>1</td><td></td></loq>	1		< LOQ (0.00683 mg/g)	1	
	BTC9304 (99)	<loq (0.0376="" g)<="" td="" w=""><td></td><td></td><td>< LOQ (0.00683 w/g)</td><td></td><td></td></loq>			< LOQ (0.00683 w/g)		
1	STG9310 (%)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td> </td><td>< LOQ (0.00683 w/g)</td><td>1</td><td>1</td></loq>			< LOQ (0.00683 w/g)	1	1
	BTG9311 (90)	<loq (0.0376="" g)<="" mg="" td=""><td></td><td></td><td>< LOQ (0.00613 w/m)</td><td>1</td><td>1</td></loq>			< LOQ (0.00613 w/m)	1	1
1	BTG9912 (97)	<loq (0.0376="" g)<="" td="" ug=""><td>1</td><td></td><td>< LOQ (0.00613 w/z) -</td><td></td><td>1</td></loq>	1		< LOQ (0.00613 w/z) -		1
	BTC9401 (93)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td></td><td>< LOQ (0.00613 wint)</td><td></td><td>-1</td></loq>			< LOQ (0.00613 wint)		-1
	BTGhongdo1 (94)	<loq (0.0376="" g)<="" td="" ug=""><td></td><td>1</td><td>< LDQ (0.00613 mg/g)</td><td>· ·</td><td></td></loq>		1	< LDQ (0.00613 mg/g)	· ·	
	BTGmendol (95)	<loq (0.0376="" g)<="" td="" ug=""><td>•</td><td></td><td>< LOQ (0.00613 mg/g)</td><td>1</td><td>1</td></loq>	•		< LOQ (0.00613 mg/g)	1	1
1	HR(004 (106)	<loq (0.0376="" m)<="" td="" ug=""><td></td><td>NA</td><td>< LOQ (0.00683 ug/g)</td><td>1</td><td>NA</td></loq>		NA	< LOQ (0.00683 ug/g)	1	NA
1	HIRG09 (105)	<loq (0.0376="" g)<="" td="" up=""><td>- 1 400</td><td>NA</td><td>< LOQ (0.00683 up)</td><td><.00</td><td>NA NA</td></loq>	- 1 400	NA	< LOQ (0.00683 up)	<.00	NA NA
NO PPOS configuration performed.				PFOS - Perfectocta			
Q - Limit of Quantitation				POAA - Perfectocca			
• Not Applicable				PFOSA - Perdeorooc			

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er(Test Substance): Method/Revision: Analytical Equipment System Instrument Software/Version: Date of Analysis/Analysi: Date of Analysis/Analysi: Date of Data Reflection/Analy Sample Data

 GEN033, MSU - Liver Samples

 NA
 Filesam

 Varioss livers - anextinated carves
 Filesam

 ET3-E-6.0 and ET3-F-7.0
 R. Squar

 Davry 070798, Amelia 062498
 Stope:

 Mashyar 3.3
 Y-later

 03/16/00 SAL/CSH/KKK
 03/25/00, 03/21/00, 03/25/00, 04/07/00

 03/16/00, 03/17/00, 31/9/00, 03/25/00, 03/21/00, 03/25/00, 04/07/00
 LAS/AbAH

 03/20/00, 03/22/00, 03/23/00, 04/07/00
 LAS/AbAH

FACT-GEN-033

See Attachments See Attachments See Attachments See Attachments

Various Livers

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Group	Sample #	Concentration of PPOS	Mesa	RSD Std. Dev.	Concentration of POAA	Mesa POAA	RSD
		of PFUS	**/1	SEL DEV. MS/MSD RPD	of POAA		SML Dev. MS/MSD RPD
Method Bilk	RBL03140-H20 BB-5-1	< LOQ (0.00696 up/g)		Marked RPD		*#1	MS/MSD RPD
Mentione Dex	RBL03140-H20 BE-5-1				<loq (0.0359="" g)<="" mg="" td=""><td>1 </td><td></td></loq>	1	
	RBL03140-H20 BB-5-3	<loq (0.00696="" g)<="" sg="" td=""><td></td><td></td><td>4.0Q (0.0359 up/g)</td><td>1 1</td><td></td></loq>			4.0Q (0.0359 up/g)	1 1	
	RBL03140-HD7 BB-5-4	< LOQ (0.0694 ug/g)			<loq (0.0718="" g)<="" mg="" td=""><td> </td><td></td></loq>		
		< LOQ (0.0694 mg/g)	1		<loq (8.0718="" g)<="" td="" ug=""><td> </td><td></td></loq>		
	RBL03140-H2O BR-5-5	< LOQ (0.0694 ug/g)			4LOQ (0.0718 ug/g)	1 1	
	KBL03140-H2O 88-5-6	< LOQ (0.0347 mg/g)			<loq (0.00719="" g)<="" mg="" td=""><td>I I</td><td></td></loq>	I I	
	RBL03140-H2O BB-5-7	< LOQ (0.0694 ug/g)	1		<loq (0.0718="" cg="" g)<="" td=""><td>1 </td><td></td></loq>	1	
	RBL03140-H2O B0:-5-4	< LOQ (0.0694 ug/g)	Į		<loq (0.0718="" g)<="" td="" ug=""><td></td><td></td></loq>		
	RBL03140-H2O BB-5-9	< LOQ (0.0694 ug/g)	1	1	<loq (0.0718="" g)<="" td="" ug=""><td></td><td></td></loq>		
	RBL03140-H2O BB-5-10	< LOQ (0.0347 sg/g)			<loq (0.00719="" g)<="" mg="" td=""><td></td><td></td></loq>		
	RBL43140-H20 BB-5-11	< LOQ (0.0347 1g/g)			4.0Q (0.0359 up/g)		
1	RBL03140-H2D BB-5-12	< LOQ (0.0347 ug/g)		NA	<loq (0.0159="" g)<="" sg="" td=""><td></td><td>NA</td></loq>		NA
	RBL03149-H2O BR-S-13	< LOQ (0.0347 ug/g)	<10Q	NA	<loq (0.0359="" g)<="" mp="" td=""><td>40Q</td><td>NA</td></loq>	40Q	NA
Matrix Bik	RBL03140-Liver 38-5-1	< LOQ (0.0694 ug/g)			<loq (0.0718="" 10="" g)<="" td=""><td></td><td></td></loq>		
	RBL03140-Liver BB-5-2	< LOQ (0.0694 ag/g)	1		<loq (0.0718="" g)<="" td="" up=""><td></td><td>ł</td></loq>		ł
	RBL03140-Liver Blc-S-1	< LOQ (0.0694 ug/g)			<loq (0.0711="" g)<="" td="" up=""><td>1</td><td>1</td></loq>	1	1
	RBL03140-Liver Bb-5-4	< LOQ (0.0694 ug/g)	l		<loq (0.0711="" g)<="" td="" w=""><td>1</td><td></td></loq>	1	
	RBL03140-Live 36-5-5	0.00683			4.0Q (0.0359 up/g)	1	
	RBL03140-Live BB-5-6	< LOQ (0.0694 up/g)			4.00 (0.0711 w/p)	l	
1	RELEASE 40-Liver Min-S-7	< LOQ (0.0694 w/g)	1		4.00 (0.0718		
	RBL03140-Liver 38-5-8	< LOO (0.0694 to/s)			4.00 (0.0718 w/g)		
	RBL03140-Liver BB-S-P	< LOQ (0.0694 mm/g)			4.00 (0.0718 w/g)	1	· ·
1	RBL03140-Liver Bit-5-10	< LOQ (8.0694 se/g)			4.00 (0.0711 m/g)	1	
	RBL03140-Liver BB-S-11	<loq (0.0347="" g)<="" m="" td=""><td></td><td></td><td><000 (0.0359 m/g)</td><td></td><td></td></loq>			<000 (0.0359 m/g)		
	RBL03140-Liver 10-5-12	< LOQ (0.0347 mg/m)		NA	<loq (0.0359="" g)<="" td="" us=""><td></td><td>NA</td></loq>		NA
	181.03140-Liver 88-5-13	< LOQ (0.0347 mg/g)	<.00 - Oue Outlier	NA	<loq (8.0359="" au="" g)<="" td=""><td> ⊲.00</td><td>NA</td></loq>	⊲.00	NA
- 20	R31.03140-10-1 pp-5-1*	104%	1		112%		
250 pph	R81.03140-MSD-1 mb-5-2*	11496	109%	7%	109%	109%	
	R.B.L.03140-MC5-250 ppb-3-1	120%	1		128%	1	
1 - E	R3L03140-MSD-250 ppb-5-2	133%	127%	10%	128%	128%	0%
····	442 (12)-145	98%			196%		
	44F (12)-MSD	107%	103%	7%	109%	107%	2%
	Tuto1 (17)-bd	13155	1		59%		***
	T18-91 (17)-MSD	140%	135%	86	64%	61%	9%
	SCV1 (19)-bdS	685			56%	+ ***	<u> </u>
	SCV1 (19-MSD	22%	79%	15%	76%	68%	30%
	WS1 (22)-MS	81%	1	<u> </u>	63%		
	WSI (22)-MSD	73%	77%	10%	62%	GK.	286
	524 (24)-MSD	92%	NA	NA	101%	NA	NA
	T15 (28)-MS	\$4%			\$3%		
	T15 (20)-MSD	103%	94%	21%	105%	99%	12%
	TILF156(35)-MB	93%		1	\$2%		
1	T1LF156 (35)-145D	72%	82%	25%	80%	34%	15%
	\$19 (57)-645	326%			126%	+	+
	S19 (39)-MSD	234%	280%	33%	154%	140%	20%
	P9 (61)-MS	231%	440778		102%	1 197/20	
	P9 (61)-MSD	142%	187%	45%	112%	107%	"
	R47 (70)-MS	699V	10/78		70%		<u>+</u>
1	R47 (70)-MSD	69%	69%	2%	63%	69%	
		1 90776					
1	L4 (91)-MS	69%		l	- 73%	1	78%
	L4 (91)-MSD	187%	128%	92%	165%	119%	
F	BTG9305 (92)-MS	143%	NA	NA	155%	NA	NA
F	BTGhongdol (94)-MS	122%		I		1	
- Limit of Quantitation	BTGhongdol (94)-MS BTGhongdol (94)-MSD	122% 120%	121%	2%	156%	146%	13%

PPOS - Perfinorooclaner POAA - Perfinorooclaner PPOSA - Perfinorooclan PPHS - Perfinorohexane

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

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GEN-003-liver.xie

سفسه مس Date of Data R Sas npie Data

GEN013, MSU + Liver Samples NA		
Various livers - uncatracted curves	Filename:	See Be
ETS-8-60 and ETS-8-7.0	R-Squared Value:	See Al
Devey 070799, Ametia 062498	Slope:	See Al
Mandyux 3.3	Y-Intercept:	See A
03/14/00 SAL/CSH/KKK	-	
03/16/00, 03/17/00, 3/19/00, 03/20/00, 03/2		i/Monet
03/20/00, 03/22/00, 03/23/00, 03/24/00, 04	04/00, 04/11/00 (AS/MMH	

See Attach

FACT-GEN-033

Group	Sample #	Concentration	Mean	RSD	Concentration	Mean	RSD
Deet		# PFOSA	PFOSA	Std. Dev.	of PTHS	PPHS	Std. Dev.
		ugig or % Rac	-9/2	MS/MSD RPD	ug/g or % Roc	*8/2	MS/MSD RPD
fathed Bik	RBL03140-H20 BB-5-1	< LOO (0.0376 up/g)			<loq (0.00613="" g)<="" td="" ug=""><td></td><td></td></loq>		
	R8L03140-H2O 88-5-2	< LOQ (0.0376 mp/g)			<loq (0.00683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	RBL03140-H20 BB-S-3	< LOQ (0.0376 mg/g)			<loo (0.00663="" s)<="" td="" us=""><td></td><td></td></loo>		
	BBL03140-H20 BB-5-4	< LOO (0.0376 w/m)			<loo (0.00683="" m)<="" ms="" td=""><td></td><td></td></loo>		
	RBL03140-H20 Bit-5-5	< LOQ (0.0376 =#/g)		1	<loq (0.00683="" g)<="" td="" wg=""><td>1</td><td>1</td></loq>	1	1
	RBL03140-H20 88-5-6	< LOO (0.0376 ag/g)			<loq (0.00683="" g)<="" td="" ug=""><td></td><td></td></loq>		
	RBL03140-H20 TE-5-7	< LOQ (0.0376 ug/g)			<loq (0.00683="" g)<="" mg="" td=""><td></td><td></td></loq>		
	RBL03140-H20 38-5-8	< LOO (0.0376 mg/g)			<loo (0.00683="" g)<="" td="" ug=""><td></td><td>1 ·</td></loo>		1 ·
	RBL01140-H20 38-5-9	< LOQ (0.0376 mg/g)	1		<loq (0.00683="" s)<="" td="" us=""><td></td><td>· ·</td></loq>		· ·
	28L01140-H20 88-5-10	< LOQ (0.0376 ug/g)			<loq (0.00683="" g)<="" td="" w=""><td></td><td></td></loq>		
	EBL03140-H2O 20-5-11	< LOQ (0.0376 w/g)			<.0Q (0.00683 mp/g)		
	RBL01140-H20 Mb-5-12	< LOO (0.0376 m/z)		NA	<loq (0.00683="" g)<="" mg="" td=""><td></td><td>NA</td></loq>		NA
	RBL01140-H20 Bb.5-13	< LOQ (0.0376 m/g)	<00>	NA	<loq (0.00683="" 19="" g)<="" td=""><td><00⊳</td><td>NA</td></loq>	<00⊳	NA
M	BBL00140-Liver Bb-5-1	< LOQ (0.0376 up/g)			<loq (0.00683="" eg="" g)<="" td=""><td></td><td></td></loq>		
Matrix Bik	RBL03140-Liver BB-5-2	< LOQ (0.076		1	<loq (0.00681="" eg="" g)<="" td=""><td>1</td><td>1</td></loq>	1	1
1	EBL03140-Liver Bit-5-3	< LOQ (0.6376 w/g)	1	1	<loq (8.00683="" g)<="" td="" w=""><td>1</td><td>1</td></loq>	1	1
	RELOS 140-Liver Bit-5-4	< LOQ (0.0076 w/g)			4.00 (0.00683 w/g)	1	1
	BLOS 140-Liver Ma-5-5	< LOQ (0.0376 w/g)			4.00 (0.00683 m/g)	1	
	23L03140-Liver 28-5-6	< LOQ (0.0976 m/s)	1		<.00 (0.00003 up/g)		ļ
	13L03140-Liver 38-5-7	<1.00 (0.0376 up/g)	1		<loq (0.00683="" g)<="" m="" td=""><td></td><td></td></loq>		
	281.03146-Liver 38-5-5	<loq (0.0376="" g)<="" td="" w=""><td>ļ.</td><td></td><td><.00 (0.0060 mg/g)</td><td>1</td><td>1.</td></loq>	ļ.		<.00 (0.0060 mg/g)	1	1.
	28L03140-Liver 38-5-9	< LOQ (0.0376 10/g)			<loq (8.0060="" td="" w="" z)<=""><td></td><td>· ·</td></loq>		· ·
	RBL03140-Live BL-5-10	< LOQ (0.0376 m/g)			4.00 (0.00683 w/g)		
	18L03140-Live 18-5-11	< LOQ (0.0376 m/g)			<loq (0.00683="" -="" 2)<="" td=""><td></td><td></td></loq>		
	101.03140-Liver 10-5-11	< LOQ (0.0376 mg/g)		NA	4.00 (0.00683 w/p)		NA
	20103140-Live 20-5-12 20103140-Live 20-5-13	<loq (0.0376="" g)<="" td="" ug=""><td>4.00</td><td>NA</td><td><loq (0.00683="" g)<="" m="" td=""><td>4.00</td><td>NA</td></loq></td></loq>	4.00	NA	<loq (0.00683="" g)<="" m="" td=""><td>4.00</td><td>NA</td></loq>	4.00	NA
		<100 (000 / 00 / 0			17%		+
90	10100140-605-1 pp5-1*	67%	67%	2%	57%	72%	42%
250 899	RBL03140-MSD-1 gpb-5-2* RBL03140-MSD-1 gpb-5-2*	114%			61%		
* .	28L03140-MSD-299 ppb-5-2	84%	99%	30%	48%	54%	25%
		94%			14%	+	
<i>'</i>	44P (12)-645	87%	92%	5%	17%	80%	3%
	44F (12)-MSD T12/91 (17)-MS	146%	7470		65%	+	
		191%	168%	26%	65%	65%	1%
			144.4		54%	1	
	SCVI (19)-MSD	\$7%	70%	48%	64%	59%	17%
	WSI (22)-MS	69%			59%		1
	WS1 (22)-MSD	69%	69%	0%	60%	60%	· 1%
	\$24 (24)-650	94%	NA	NA	76%	NA	NA
	T15 (28)-ME				6%		
	T15 (28)-MSD	95%	11%	36%	102%	1 10%	40%
	TILF156 (05)-145		4.74		1 275	1 100	
	TIL/156 (35)-MSD	69%	76%	26%	73%	30%	19%
	S19 (59)-MS	140%			\$7%	1	
	S19 (59)-MSD	2%	71%	195%	-1%	48%	200%
	P9 (61)-MS	73%	1	-1	52%	1	1
	P9 (61)-MSD	68%	70%	7%	48%	50%	
	R47 (70)-MS	68%			55%		
		73%	73%	14%	58%	57%	5%
	I.4 (91)-MSD	50%	1		58%		1
		119%	15%	\$2%	145%	102%	16%
	L4 (91)-MSD BTG9345 (92)-MS	70%	NA	NA NA	31%	NA	NA
	BTGhongile1 (94)-MS	625			18%		-1
							20%

LOQ - Limit of Quar NA - Not Applicable

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PFOS - Partinos POAA - Partinos PFOSA - Partino PFOSA - Partino

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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:

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st.

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.

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- ETS-8-4.1 Extraction of PFOS from Serum Page 1 of 14

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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

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- 3.1 PFOS: perfluorooctanesulfonate (anion of potassium salt) $C_8F_{17}SO_3^-$
- 3.2 PFOSA: perfluorooctane sulfonylamide C₈F₁₇SO₂NH₂
- 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 C₈F₁₇SO₂N(CH₂CH₃)CH₂CH₂OH
- 3.5 PFOSEA: perfluorooctane sulfonyl ethylamide $C_8F_{17}SO_2N(CH_2CH_3)H$
- 3.6 M556: C₈F₁₇SO₂N(H)(CH₂COOH)
- 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

- ETS-8-4.1 Extraction of PFOS from Serum

Page 2 of 14

6.1.5 Balance $(\pm 0.100 \text{ g})$

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 μ L to 50 μ L
- 7.10 Graduated pipettes
- 7.11 Syringes, disposable plastic, 3 cc
- 7.12 Syringe filters, nylon, 0.2 µm, 25 mm
- 7.13 Timer

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- 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 (Na₂CO₃), J.T. Baker or equivalent
- 8.5 Sodium bicarbonate (NaHCO₃), 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

ETS-8-4.1 Extraction of PFOS from Serum

Page 3 of 14

3M_MN01666051

- **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 C₈F₁₃SO₃H) molecular weight = 428
- 8.9.8 Other fluorochemicals, as appropriate

8.10 Reagent preparation

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- **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-QTM 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-QTM 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 (Na₂CO₃/NaHCO₃): Weigh approximately 26.5 g of sodium carbonate (Na₂CO₃) and 21.0 g of sodium bicarbonate (NaHCO₃) into a 1 L volumetric flask and bring to volume with Milli-QTM 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.

- ETS-8-4.1 Extraction of PFOS from Serum Page 4 of 14

3M_MN01666052

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, C₈F₁₃SO₃H 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

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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-QTM 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.

ETS-8-4.1 Extraction of PFOS from Serum Page 5 of 14

3M_MN01666053

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

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- 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.

ETS-8-4.1 Extraction of PFOS from Serum

Page 6 of 14

Table 1Approximate spiking amounts for standards and spikesUsing 1.0 mL of matrix								
Working standard	Working standard µL Approx. final conc. of							
(approx. conc.)		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.

- ETS-8-4.1 Extraction of PFOS from Serum Page 7 of 14

3M_MN01666055

- 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

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

<u>mL of standard x concentration of standard ($\mu g/mL$)</u> mL of standard + mL of surrogate standard + initial matrix volume (mL)

Final Concentration (µg/mL) of PFOS in matrix

14.0 METHOD PERFORMANCE

- 14.1 The method detection limit (MDL) is analyte and matrix specific. Refer to MDL report for specific MDL and limit of quantitation (LOQ) values (see Attachments B and C).
- 14.2 The following quality control samples are extracted with each batch of samples to evaluate the quality of the extraction and analysis.
 - 14.2.1 Method blanks and matrix blanks.
 - 14.2.2 Matrix spike and matrix spike duplicate samples to determine accuracy and precision of the extraction.
 - 14.2.3 Continuing calibration check samples to determine the continued accuracy of the initial calibration curve.
- 14.3 Refer to section 14 of ETS-8-5.1 for method performance criteria.

15.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

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

- ETS-8-4.1 Extraction of PFOS from Serum Page 8 of 14

3M_MN01666056

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

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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

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Revision Number	Reason For Revision	Revision Date
1	Section 12.21 Changed to include sample storage at room temperature.	04/02/99
	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.	

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

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- 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.

- ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 3 of 10

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

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- **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.

Page 4 of 10

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

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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 = $10 \mu L$ injection

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 9 minutes

ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 5 of 10

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

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- 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 $10 500 \,\mu$ L/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°

- ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 6 of 10

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- 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:

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13.1.4 Calculate matrix spike percent recoveries using the following equation:

% Recovery = <u>Observed Result - Background Result</u> x 100 Expected Result

13.1.5 Calculate percent difference using the following equation:

% Difference = <u>Expected Conc. - Calculated Conc.</u> x 100 Expected Conc.

13.1.6 Calculate actual concentrations in matrix ($\mu g/g$):

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 \pm 30% of the spiked concentration.

14.5 Continuing Calibration Verification

- 14.5.1 Continuing calibration verification percent recoveries must be within \pm 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.

Page 7 of 10

3M_MN01666062

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

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- 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

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Revision <u>Number</u>

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Reason For Revision

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Revision Date

- ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 9 of 10

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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:

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Group Dose	Sample#	Concentration ng/g	Initial Wt. g	Dilution Factor	Final Conc. ug/g
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Slope: Taken from linear regression equation.

Group/Dose: Taken from the study folder.

Sample#: Taken from the study folder.

Concentration (ng/g): Taken from the MassLynx integration summary.

Initial Wt. (g): Taken from the study folder.

Dilution Factor: Taken from the study folder.

Final Conc. (ug/g): Calculated by dividing the initial volume from the concentration

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Attachment A: Summary Spreadsheet

t - ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 10 of 10

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

Revision Date:

Adoption Date:

Author: Lisa Clemen, Robert Wynne

Approved By:

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Laboratory Manager

Group Leader

Technical Reviewer

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.

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- ETS-8-6.0 Extraction of PFOS from Liver

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Page 1 of 14

Date

Date

Date

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

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- 3.1 PFOS: perfluorooctanesulfonate (anion of potassium salt) C₈F₁₇SO₃
- 3.2 PFOSA: perfluorooctane sulfonylamide C₈F₁₇SO₂NH₂
- 3.3 PFOSAA: perfluorooctane sulfonylamido (ethyl)acetate C₈F₁₇SO₂N(CH₂CH₃)CH₂CO₂
- 3.4 EtFOSE-OH: 2(N-ethylperfluorooctane sulfonamido)-ethyl alcohol C₈F₁₇SO₂N(CH₂CH₃)CH₂CH₂OH
- 3.5 PFOSEA: perfluorooctane sulfonyl ethylamide C₈F₁₇SO₂N(CH₂CH₃)H
- **3.6** M556: C₈F₁₇SO₂N(H)(CH₂COOH)
- 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

• ETS-8-6.0 Extraction of PFOS from Liver Page 2 of 14

- 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 μ L to 50 μ L
- 7.12 Graduated pipettes
- 7.13 Syringes, disposable plastic, 3 cc
- 7.14 Syringe filters, nylon, 0.2 µm, 25 mm
- 7.15 Timer

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- 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-QTM 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 (Na₂CO₃), J.T. Baker or equivalent
- 8.5 Sodium bicarbonate (NaHCO₃), 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

ETS-8-6.0 Extraction of PFOS from Liver Page 3 of 14

- **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 C₈F₁₃SO₃H) molecular weight = 428
- 8.10.8 Other fluorochemicals, as appropriate

Reagent preparation

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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-QTM 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-QTM 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-QTM 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-QTM 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 (Na₂CO₃/NaHCO₃): Weigh approximately 26.5 g of sodium carbonate (Na₂CO₃) and 21.0 g of sodium bicarbonate (NaHCO₃) into a 1 L volumetric flask and bring to volume with Milli-QTM 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.

ETS-8-6.0 Extraction of PFOS from Liver

Page 4 of 14

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- **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, C₈F₁₃SO₃H 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

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

10.0 OUALITY 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.

ETS-8-6.0 Extraction of PFOS from Liver

Page 5 of 14

- **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

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- 11.1.1 Weigh approximately 40 g of liver into a 250 mL Nalgene bottle containing 200 mLs Milli-QTM 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.

Page 6 of 14

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.

Approximate Spiking A	Table 1 Approximate Spiking Amounts for Calibration Standards								
Working Standard	μl	Approx. final conc. of							
(Approx. Conc.)		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

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- 12.1 Obtain frozen liver samples.
- 12.2 Cut approximately 1 g of liver using a dissecting scalpel. This part of the procedure is best performed quickly, not allowing the liver to thaw.
- 12.3 Weigh the sample directly into a tared plastic sampule vial.
- 12.4 Record the liver weight in the study notebook.
- 12.5 Return unused liver portions to freezer.
- 12.6 Add 2.5 mLs of water to sampule vial.
- 12.7 Grind the sample. Put the grinder probe in the sample and grind for about 2 minutes, or until the sample is homogeneous.
- 12.8 Rinse the probe into the sample with 2.5 mLs water using a pipette.
- 12.9 Take the grinder apart and clean it with methanol after each sample. Refer to AMDT-EP-22.
- 12.10 Cap the sample and vortex for 15 seconds. Label the sampule vial with the study number, weight, liver ID, date and analyst initials.

ETS-8-6.0 Extraction of PFOS from Liver Page 7 of 14

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- 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-QTM 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.

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- 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.

Page 8 of 14

13.0 DATA ANALYSIS AND CALCULATIONS

13.1 Calculations:

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13.1.1 Calculate the average density of the liver homogenate by recording each mass of ten separate 1.0 mL aliquots of homogenate.

Average density (mg/mL) = <u>Average mass (mg) of the aliquots</u>

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:

<u>g of Liver x Average density* of homogenate (mg/mL)</u> (g of Liver + g of Water)

- * refer to 13.1.1 for details.
- 13.1.3 Calculate actual concentrations of PFOS and other fluorochemicals in calibration standards using the following equation:

<u>µL of Standard x Concentration (µg /mL)</u> = Final Concentration (µg/g or mg/kg) mg Liver / 1 mL homogenate* 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.

ETS-8-6.0 Extraction of PFOS from Liver

Page 9 of 14

3M_MN01666074

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 AFFÉCTED 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.

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Reason For Revision

Revision Date

- ETS-8-6.0 Extraction of PFOS from Liver Page 10 of 14

Study #	Surrogate Std	FC Mix Std	FC Mix Std	FC Mix Std	Comments
Matrix	approx. ppm	approx. 0.5 ppm	approx. 5 ppm	approx. 50 ppm	
Box #	actual ppm	actual ppm	actual ppm	actual ppm	
Wk/Day	#	#	#	#	
Date Spiked/Analyst					
CCV					
MS					
MSD					
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		-	-	-	
			-	•	
Blank Liver H	Homogenate: Std #	Live	er amount =	g	
Liver Extraction Method					Date & Initials
Spike surrogate and Standard n	nix. 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 ₂ CO ₂ /0		Std. #			
Dispense 5ml of Methyl-t-But		TN-A-			
Shake 20 min.		Shaker Sp	eed		
Centrifuge 20-25 min.		Centrifuge			
Remove a 4 mL aliquot of orga	inic layer				
Put on Nitrogen Evaporator to		Evaporator	Temperature		
Add 1.0 mL of Methanol		TN-A-			
Vortex 30 sec.					
Filter using a 3cc B-D syringe	with a 0.2 µm SRI filter	into autosample vial			

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.

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ETS-8-6.0 Extraction of PFOS from Liver Page 11 of 14

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 for rabbit liver

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

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Compou	nu. 1105						
	Prepared	Range of ·	LCR from	Range of	LCR from	Range of	LCR from
Liver	range of	average	ave curve	low std	low std	high std	high std
matrix	standards	curve		curve	curve	curve	curve
	(ppb) (ng/mL)						
Rabbit	6.19 - 1237	12 - 1200	12 - 1200	6 - 300	12 - 300	60 - 1200	60 - 1200

Compound: PFOSA

	Prepared	Range of	LCR from	Range of	LCR from	Range of	LCR from
Liver	range of	average	ave curve	low std	low std	high std	high std
matrix	standards	curve		curve	сигуе	curve	CUIVE
	(ppb) (ng/mL)						
Rabbit	6.19 - 1237	12 - 1200	12 - 1200	12 - 300	12 - 300	60 - 1200	60 - 1200

Compound: PFOSAA

	Prepared	Range of	LCR from	Range of	LCR from	Range of	LCR from
Liver	range of	average	ave curve	low std	low std	high std	high std
matrix	standards	curve		curve	curve	curve	curve
	(ppb) (ng/mL)						
Rabbit	6.16 - 1232	12 - 1200	30 - 1200	30 - 900	60 - 900	N/A	N/A

- ETS-8-6.0 Extraction of PFOS from Liver

Compound: EtFOSE-OH

	Prepared	Range of	LCR from	Range of	LCR from	Range of	LCR from
Liver	range of	average	ave curve	low std	low std	high std	high std
matrix	standards	curve		curve	спьке	curve	curve
	(ppb) (ng/mL)						
Rabbit	6.17 - 1235	31 - 900	31 - 900	N/A	N/A	N/A	N/A

Compound: PFOSEA

	Prepared	Range of	LCR from	Range of	LCR from	Range of	LCR from
Liver	range of	average	ave curve	low std	low std	high std	high std
matrix	standards	curve		curve	CHIVE	curve	curve
	(ppb) (ng/mL)						
Rabbit	6.17 - 1235	31 - 1200	31 - 1200	N/A	N/A	N/A	N/A

Compound: M556

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	Prepared	Range of	LCR from	Range of	LCR from	Range of	LCR from
Liver	range of	average	ave curve	low std	low std	high std	high std
matrix	, standards	curve		curve	curve	curve	curve
	(ppb) (ng/mL)	(ppb) (mg/ml.)					
Rabbit	6.17 - 1235	31 - 1200	60 - 1200	N/A	N/A	N/A	N/A

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Attachment B: MDL/LOQ Values

ETS-8-6.0 Extraction of PFOS from Liver

Page 13 of 14

Ion Pair Standard Curves – Tissue

Prep date(s): Analyte(s): Sample matrix:

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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	PFOSA	PFOSAA	EtFOSE	PFOSEA	M556		All	All
Std conc	Std conc	Std conc	Std conc	Std conc	Std conc	Std conc	Am't spiked	Density
ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	ug/mL	mL	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

		DEOGAA	PAROCE	PFOSEA	M556		Surrogate	All
PFOS	PFOSA	PFOSAA	EtFOSE					
Final	Final	Final conc	Final	Final	Final	Std conc	Std conc	Am't
conc	conc	ng/g	· conc	conc	conc	ng/g	ng/mL	spiked
ng/g	ng/g		ng/g	ng/g	ng/g			mL
5.99	5.99	5.99	5.99	5.99	5.99		100	0.005
12.0	12.0	12.0	12.0	12.0	12.0			
29.9	29.9	29.9	29.9	29.9	29.9		Surrogate	
59.9	59.9	59.9	59.9	59.9	59.9		Final conc	
120	120	120	120	120	120		ng/mL	
299	299	299	299	299	299		0.500	
599	599	599	599	599	599	<u> </u>		
898	898	898	898	898	898			1
1198	1198	1198	1198	1198	1198			

Validated ranges - approximate concentrations

A PERSONAL PARTY AND A PERSON A	Pop abbi amage									
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,	Estimates only, use rabbit values.								
Rat	Estimates only,	Estimates only, use rabbit values.								
Monkey	Estimates only,	use rabbit values.								

Attachment C: Standard Calculations

ETS-8-6.0 Extraction of PFOS from Liver Page 14 of 14

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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:

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Laboratory Manager

Group Leader

Technical Reviewer

Date

Date

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.

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- ETS-8-5.1 Analysis of Serum Extract Using ES/MS

3M MN01666080

Page 1 of 9

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

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- 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.

Page 2 of 9

3M_MN01666081

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

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- 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-QTM water, all water used in this method should be Milli-QTM 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

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- **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.

Page 4 of 9

3M_MN01666083

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 = $10 \mu L$ 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.

ETS-8-5.1 Analysis of Serum Extract Using ES/MS Page 5 of 9

3M_MN01666084

- 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 $10 - 500 \,\mu$ L/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.
- 12.3.10Click 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:

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13.1.4 Calculate matrix spike percent recoveries using the following equation:

% Recovery = <u>Observed Result - Background Result</u> x 100 Expected Result

13.1.5 Calculate percent difference using the following equation:

% Difference = <u>Expected Conc. - Calculated Conc.</u> x 100 Expected Conc.

13.1.6 Calculate actual concentration of PFOS, or other fluorochemical, in matrix (µg/mL):

(ng of PFOS calc. from std. Curve x Dilution Factor) x <u>1 μg</u> (Initial Volume of matrix (mL) + mL of Surrogate Standard) 1000 ng Final Volume (mL)

> ETS-8-5.1 Analysis of Serum Extract Using ES/MS

Page 6 of 9

3M_MN01666085

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

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14.4.1 Matrix spike percent recoveries are must be within \pm 30% of the spiked concentration.

14.5 Continuing Calibration Verifications

- 14.5.1 Continuing calibration verification percent recoveries must be \pm 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.

ETS-8-5.1 Analysis of Serum Extract Using ES/MS Page 7 of 9

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

1

1

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:

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Group Dose	Sample#	Concentration ug/mL	Initial Vol. mL	Dilution Factor	Final Conc. ug/mL
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			_		
			-		
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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

Attachment A: Summary Spreadsheet

t ETS-8-5.1 Analysis of Serum Extract Using ES/MS Page 9 of 9

3M_MN01666088

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:

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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.

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ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 1 of 10

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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 perfluoroctanesulfonate (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.

Page 2 of 10

3M_MN01666090

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

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- 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-QTM water, mix until all solids are dissolved. Store at room temperature.

ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 3 of 10

3M_MN01666091

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

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- **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.

ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 4 of 10

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

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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 = $10 \mu L$ injection

12.2.2.2 Inject/sample = 1

12.2.2.3 Cycle time = 9 minutes

ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 5 of 10

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

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- 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 $10 500 \,\mu$ L/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°

ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 6 of 10

- 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:

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13.1.4 Calculate matrix spike percent recoveries using the following equation:

% Recovery = <u>Observed Result - Background Result</u> x 100 Expected Result

13.1.5 Calculate percent difference using the following equation:

% Difference = <u>Expected Conc. - Calculated Conc.</u> x 100 Expected Conc.

13.1.6 Calculate actual concentrations in matrix ($\mu g/g$):

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 \pm 30% of the spiked concentration.

14.5 Continuing Calibration Verification

- 14.5.1 Continuing calibration verification percent recoveries must be within \pm 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.

ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 7 of 10

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

4

- 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"

Page 8 of 10

3M_MN01666096

20.0 REVISIONS

Li.

Revision <u>Number</u>

1

4.

Reason For Revision

Revision Date

ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 9 of 10

3M_MN01666097

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:

.4

Group Dose	Sample#	Concentration ng/g	Initial Wt. g	Dilution Factor	Final Conc. ug/g
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			-		

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

Attachment A: Summary Spreadsheet

t ETS-8-7.0 Analysis of Liver Extract Using ES/MS Page 10 of 10

3M_MN01666098