LCMSMS Analysis of Extracts
Reported in: "Preliminary Report Analysis of Perfluorinated Compounds in Environmental Samples" 4/7/99

3M Environmental Technology
and Services

PO Box 3331
St. Paul, MN 55133-3331
612 778 6442

3M

# Laboratory Report

**LCMSMS Analysis of Extracts reported in: "Preliminary Report Analysis of Perfluorinated Compounds in Environmental Samples" by P. Jones and K. Kannan**

**Report number: FACT-GEN-008**

*Testing Laboratory*

3M Environmental Technology & Safety Services
3M Environmental Laboratory
Fluorine Analytical Chemistry Team (FACT)
2-3E-09
935 Bush Avenue,
St. Paul, MN 55106

*Laboratory Contact*

Kris Hansen, Ph.D.
Bldg. 2-3E-09
P.O. Box 3331
St. Paul, MN 55133-3331
Phone: (651) 778-6018
FAX: (651) 778-6176

*Requester*

3M/Battelle Exposure Team (c/o Susan Beach)
3M Company
Environmental Lab
2-3E-09
St. Paul, MN

## 1 Introduction

Sample extracts, prepared by P. Jones and K. Kannan according to FACT-M-4.0, were screened using a Micromass Platform II, a single quadrapole LCMS. These same extracts were re-screened by FACT using a Micromass Quattro II, a triple quadrapole instrument (LCMSMS), which provides a more selective analysis than a single quad instrument. Interferences that may provide a false positive response in LCMS analysis can be eliminated with the LCMSMS.

Extracts from samples of sea eagle plasma, bald eagle plasma, and fish were analyzed quantitatively for PFOS using the LCMSMS. In general, PFOS levels determined by LCMSMS were approximately half of those determined using LCMS. It is likely that matrix interference at or around mass 499 complicated the original LCMS analysis. This type of matrix interference near mass 499 has been observed in other biological samples.For the LCMSMS analysis, quantitation was based on the 499 → 99, 130 daughter ions response.

In the LCMSMS analysis, low levels of PFOS (< 5 ppb) were detected in several of the fish samples; these values are significantly lower than those reported by Jones and Kannan. It is likely that a matrix interferent caused an anomolously high response in the LCMS analysis.

No PFOS above the limit of detection was detected in the remaining fish samples analyzed by LCMSMS. Again, this is in contrast to the LCMS analysis.

Additionally, all samples of eagle plasma were screened for the following fluorochemicals: PFOSA, PFOSAA, M570, M556, POAA, PFHS, and PFDS (see Appendix B for definitions of FACT acronyms). Very low levels (< 5 ppb) of PFOSA were observed in the North American bald eagle plasma samples. No PFOSAA, M556, POAA, or M570 was detected in any samples.

PFHS was detected in every Baltic sea eagle plasma sample, while PFDS was preliminarily (no standard material available for confirmation) detected in every North American bald eagle plasma sample.

This data is screening quality only. Only 2 high level (250-ppb) PFOS matrix spike samples (eagle plasma) were extracted and analyzed.

## 2 Sample Receipt

P. Jones and K. Kannan delivered samples to the Environmental Lab. The samples were extracted and analyzed by them using an Environmental Lab LCMS on March 15-19, 1999. After LCMS analysis, the extracts were submitted to FACT for LCMSMS analysis. There was no extraction paperwork submitted with the samples. It is assumed that sample volume and final extract volume were the same.

## 3 Holding Times

There is no holding time criteria associated with these samples for LC/ESMSMS analysis.

## 4 Methods - Analytical and Preparatory

**Analytical methods**

FACT-M-3.1, Extraction of Potassium Perfluorooctanesulfonate or Other Fluorochemical Compounds from Serum or Other Fluid for Analysis Using HPLC-Electrospray/Mass Spectrometry: modified by NOT extracting a matrix curve for use in quantitation. A pre-existing curve, extracted from rabbit sera, was used for quantitation. This modification will have the greatest effect on the fish samples. Extraction efficiencies of most of the target fluorochemicals from the fish tissues are likely to be significantly lower than those from the plasma. For this reason, LCMSMS reported values for the fish tissue samples should be considered to be minimum.

FACT-M-4.1, Analysis if Potassium Perfluorooctanesulfonate or Other Fluorochemical in Serum or Other Fluid Using HPLC-Electrospray/Mass Spectrometry: modified by quantitating samples versus a standard curve extracted from rabbit sera, not from the sample matrix.

**HPLC/ESMS and HPLC/ESMSMS**

In HPLC, an aliquot of extract is injected and passed through a reverse phase liquid chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. For example, in a standard solution PFOS may elute at 8.0 minutes. Retention times between a standard PFOS solution and the analyte extracted from groundwater in this analysis were matched on the HPLC system to within 1%.

Following HPLC separation, ESMSMS provides a rapid and accurate means for analyzing a wide range of organic compounds, including fluorochemicals. Electrospray, an ionization technique used primarily for detection of molecular ions, is generally operated at relatively mild temperatures. Molecules are ionized, and a primary ion, characteristic of the analyte, is selected. This ion is bombarded with high-energy gas; subsequent collisions create smaller secondary ionic fragments unique to the primary ion, which are detected.

For example, for PFOS ($C_8F_{17}SO_3^-$) analysis, ion 499 is selected as the characteristic primary ion. This ion is fragmented into other ions such as 80 amu (corresponding to $SO_3^-$), and 130 amu ($CF_2SO_3^-$). Each of these secondary fragments is detected and can be used to differentiate PFOS from other compounds that might have the same characteristic 499 amu primary ion, but different chemical compositions and secondary ion fragmentation patterns.

## 5 Analysis

### 5.1 Quantitative Analysis

For both the LCMS and LCMSMS analysis, quantitative results are based upon comparison of analyte peak area in the sample with analyte peak areas generated using a standards extracted from rabbit sera. The extraction efficiency of PFOS from sera of other animals has been determined to be between 75-95% at all levels within the range of quantitation. It is likely that the extraction efficiency of PFOS from eagle plasma is approximately the same.

### 5.2 Qualitative Analysis

Eagle plasma sample extracts were qualitatively screened for PFOSA, PFOSAA, M556, M570, POAA, PFHS, and PFDS. During the analysis, the extracts were bracketed by a minimum number of standards to monitor analyte retention time. Other than relative comparisons, no quantitative results are available.

No PFDS standard material is available. Preliminary identification of PFDS in the extracts is based upon response of 3 reasonable daughter ions (599→80, 99, 130) at a reasonable retention time.

### 5.3 Blanks

Two container blanks were provided and extracted by Jones and Kannan. Extraction blanks were also prepared. No analytes were detected in the extracts of either the container blanks or the extraction blanks.

### 5.4 Matrix Spikes

One PFOS matrix spike at 250 ppb was prepared for each of two eagle plasma samples. Both matrix spikes showed greater than 80% recovery.

### 5.5 Laboratory Control Samples

Laboratory Control Samples are not a component of this study.

### 5.6 Sample Related Comments

All data is screening quality only. All analytical results of fish tissue extracts should be considered to be very preliminary, as the extraction efficiency of the target fluorochemicals from these matrices is not characterized.

## 6 Data Summary

Extracts from samples of sea eagle plasma, bald eagle plasma, and fish were analyzed quantitatively for PFOS using The LCMSMS. In general, PFOS levels determined by LCMSMS were approximately half of those determined using LCMS. It is likely that matrix interference at or around mass 499 complicated the LCMS analysis. For the LCMSMS analysis, quantitation was based on the 499 → 99, 130 daughter ions response. Matrix interference at 499 has been observed in other biological samples.

Low levels of PFOS (< 5 ppb) were detected in several of the fish samples; these values are significantly lower than those reported by Jones and Kannan. It is likely that a matrix interferent caused an anomolously high response in the LCMS analysis. This type of anomaly has been observed by FACT on other samples analyzed by LCMS and LCMSMS.

No PFOS above the limit of detection was detected in the remaining fish samples analyzed by LCMSMS. Again, this is in contrast to the LCMS analysis.

Additionally, all samples of eagle plasma were screened for the following fluorochemicals: PFOSA, PFOSAA, M570, M556, POAA, PFHS, and PFDS (see Appendix B for definitions of FACT acronyms). Very low levels (< 5 ppb) of PFOSA were observed in the North American bald eagle plasma samples. No PFOSAA, M556, POAA, or M570 was detected in any samples.

PFHS was detected in every Baltic sea eagle plasma sample, while PFDS was preliminarily (no standard material available for confirmation) detected in every North American bald eagle plasma sample.

This data is screening quality only. Only 2 high level (250-ppb) PFOS matrix spike samples (eagle plasma) were extracted and analyzed.

## 7 Data / Sample Retention

Samples and extracts will be retained for future evaluation, if necessary.

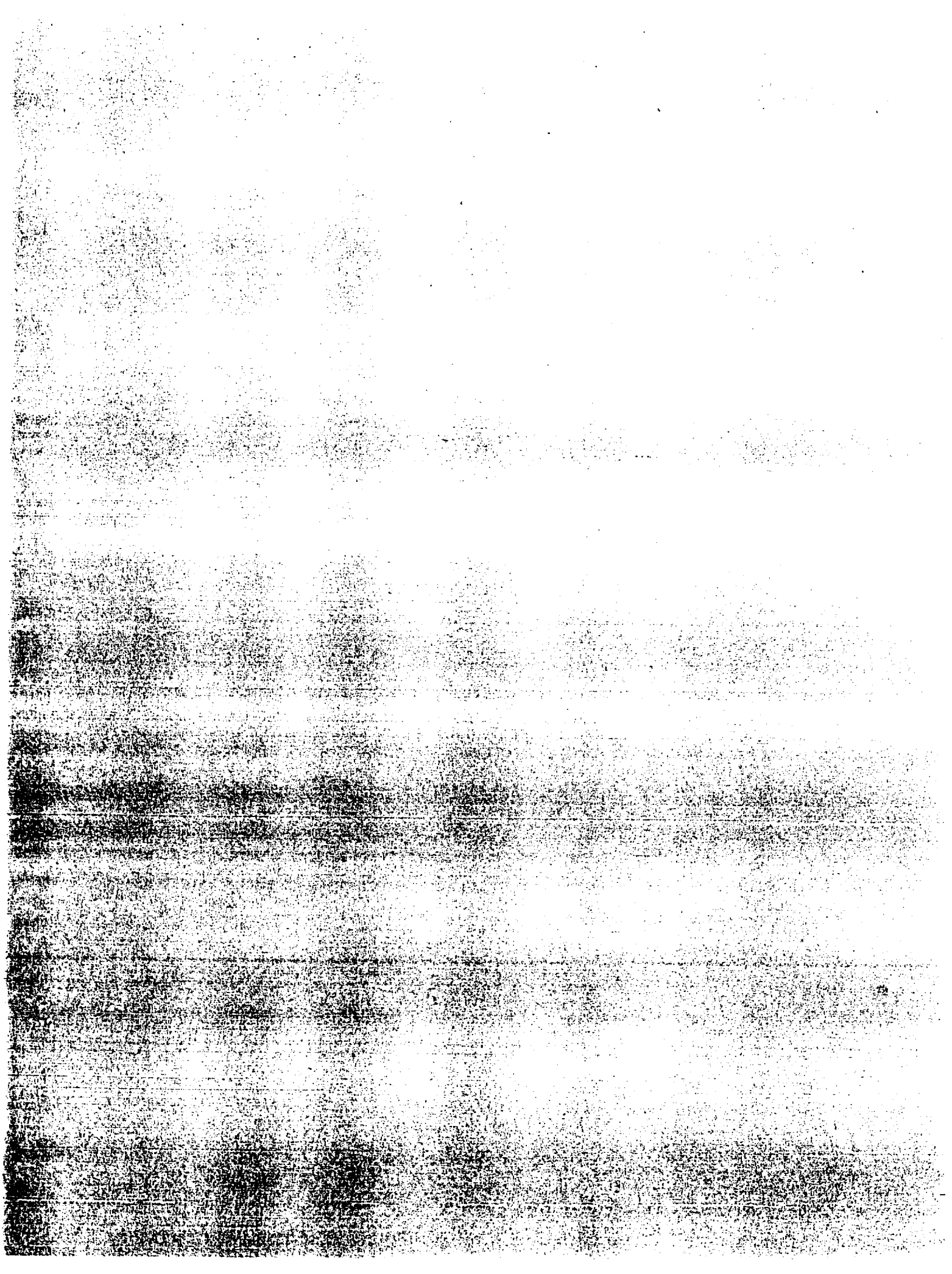
## 8 Attachments

**8.1** **Attachment A:** Table of LCMSMS Results for the Analysis of Eagle Plasma and Fish Tissue Samples
**8.2** **Attachment B:** Table of FACT acronyms and formulas
**8.3** **Attachment C:** Data associated with LCMSMS analysis
**8.4** **Attachment D:** LCMS report by P. Jones and K. Kannan

## 9 Signatures

Harold Johnson, Advanced Chemist — Date: 4/7/99

Kris Hansen, Ph.D., FACT Group Leader — Date: 4/7/99



| Sample number | Tissue: | Location: | ng PFOS / mL | PFHS? | PFDS? |
|---|---|---|---|---|---|
| WTEA-SWE-93-020 | Sea Eagle Plasma | Baltic | 125 | detected | |
| WTEA-SWE-93-017 | Sea Eagle Plasma | Baltic | 93 | detected | |
| WTEA-SWE-93-012 | Sea Eagle Plasma | Baltic | 215 | detected | |
| 629-38263 | Bald Eagle Plasma | N. America | 165 | | detected |
| 629-38264 | Bald Eagle Plasma | N. America | 198 | | detected |
| 629-07411 | Bald Eagle Plasma | N. America | 494 | | detected |
| 629-35547 | Bald Eagle Plasma | N. America | 1047 | | detected |
| 629-36389 | Bald Eagle Plasma | N. America | 226 | | detected |
| 629-37030 | Bald Eagle Plasma | N. America | 371 | | detected |
| 629-37021 | Bald Eagle Plasma | N. America | 374 | | detected |
| F980086 | Carp, whole body | Pine River, MI | BLD | | |
| F970312 | Lake Trout whole body | Siskiwit, MI | BLD | | |
| F970310 | Lake Trout whole body | Siskiwit, MI | BLD | | |
| 1998095 | Brown Trout muscle | Detroit River, MI | BLD | | |
| F97187 | Ciscowet muscle | Lake Superior | BLD | | |
| 1998095 | Brown Trout Liver | Detroit River, MI | BLQ | | |
| 1998064 | Channel Catfish egg | Lake St. Claire, MI | BLQ | | |
| 1998064 | Channel Catfish egg | Lake St. Claire, MI | BLQ | | |
| F980081 | Lake Trout, whole body | Pine River, MI | BLQ | | |
| F970181 | Lake Trout, whole body | Lake Superior | BLD | | |
| F97002 | Walleye, whole body | Detroit River, MI | BLD | | |
| 1998064 | Channel Catfish muscle | Lake St. Claire, MI | BLD | | |

BLQ = below limit of quantitation (10 ppb)
BLD = limit of detection (approximately 1 ppb)

**3M Environmental Laboratory- Fluorine Analytical Chemistry Team**
Kris Hansen - Sr. Analytical Chemist
Fluorine Analytical Chemistry Team
Building 2-3E-09
612-778-6018

## Screening of PFOS levels in Eagles and Albatross

**Summary:**

Ten samples of bird sera of bird sera, five from juvenile bald eagle and five from Layson albatross, we received at the Environmental Lab. The eagle sera was collected from birds in Northern Minnesota (1 sample), the Upper Peninsula of Michigan (2 samples), and the Lower Peninsula of Michigan (2 samples). All five albatross samples were obtained from birds found at the Officer Club plot on Sand Island within the Midway Atoll in the north central Pacific Ocean.

The sera samples were extracted; extracts were analyzed quantitatively for perfluorooctane sulfonate (PFOS) by high-pressure liquid chromatography-electrospray mass spectrometry (HPLC-ESMS). Analyte identification was verified by comparison of molecular ion-HPLC retention time of the extracted analyte and standard material. Samples were quantitatively evaluated against a five-point solvent curve.

The samples of eagle sera were determined to contain 30-77 ppb perfluorooctane sulfonate (PFOS); no PFOS was detected in the albatross above the limit of quantitation (10 ppb). Specific results and analytical parameters are attached to this report.

The presence of PFOS in samples of eagle sera was verified using HPLC-ESMSMS. This technique provides an additional degree of certainty to the analyte identification by specifically monitoring fragments daughter ions (m/z=80, 99, 130, 180, 230) characteristic of the PFOS primary ion (m/z = 499).

The presence of low levels of PFOSA, perfluorooctanesulfonylamide, was also confirmed in each of the samples of eagle sera.

**Experimental summary:**

*Sample preparation: Ion-pairing extraction*

In a pH controlled environment, an ion-pairing reagent, tetrabutyl ammonium sulfate (TBA), is used to extract the analyte from the matrix. Anionic compounds, like PFOS and perfluorooctanoate (POAA), are selectively targeted by the cationic reagent. Subsequent to the formation of the TBA-anion pair, the analyte is transferred to a non-polar organic solvent (ethyl acetate), dried, and reconstituted in methanol for HPLC-ESMS analysis.

*HPLC: Characteristic retention times for PFOS*

In HPLC, an aliquot of the extract is injected and passed through a chromatographic column. Based on the affinity of the analyte for the stationary-phase in the column relative to the liquid mobile-phase passing through the column, the analyte is retained for a characteristic amount of time. For example, in a standard solution, PFOS may elute at 10.5 minutes. Retention times between a standard PFOS solution and the analyte extracted from sera in this analysis were matched to within 1% on the HPLC system.

*ESMS: Detection and monitoring of the molecular ion*

Analysis of PFOS standards indicates that the primary ion characteristic of PFOS is m/z = 499 amu, corresponding to the mass of the anionic surfactant ($C_8F_{17}SO_3$-). This ion was monitored selectively to maximize sensitivity. A scan of m/z=100 to 1210 (negative only) was also collected.

*ES/MSMS: Confirmation of analyte identification*

Several samples in this set were analyzed by ESMSMS to verify the identity of the PFOS analyte ion. ES-MSMS is very similar to ESMS, except that it adds an additional dimension of certainty to compound identification. As in ESMS, a compound specific ion is selected. After selection, the selected ion is characterized further by smashing it apart with high-energy gas. As a result of the smashing, ionic fragments, characteristic of the molecule, are created and detected.

For example, for PFOS analysis, ion 499 is selected as the compound specific primary ion. This ion is smashed into other ions such as 80 amu (corresponding to $SO_3^-$), 99 amu (corresponding to $FSO_3^-$), 130 amu (corresponding to $CF_2SO_3^-$), 180 amu ($C_2F_4SO_3^-$), and 230 amu ($C_3F_6SO_3^-$). If PFOS is present in the samples, each of these secondary fragments is detected at the detector.

**Quality control summary:**

Due to sample size limitations, duplicate extraction and matrix spike analysis were not carried out on these samples. Quantitation of PFOS levels was determined relative to a standard solvent curve instead of an extracted matrix curve. As the extraction efficiency of PFOS is unknown, these results should be considered semi-quantitative and of screening quality. The standard curve was analyzed twice, before and after the samples. One 250-ppb calibration check was analyzed after five samples; recovery was acceptable at 87%. Minimal quality control was associated with this analysis. If more precision is required, the extracts may be re-analyzed.

*Limit of Quantitation*

The low standard analyzed with these samples was 10 ppb; this is the limit of quantitation associated with the analysis.

**Instrumental specifics:**

*HPLC system*

Hewlett Packard Series 1100 Liquid Chromatograph

Column: Keystone Betasil $C_{18}$
2 x 100 mm
5 µm particle size
Flow rate: 300 µL/min.
Solvent A: 2.0 mM Ammonium Acetate
Solvent B: Methanol
Solvent Gradient:
45 to 90 %B in 9.50 mins.
Hold at 90 %B for 3.50 mins.
Return to 45 %B in 1.50 mins.
Hold at 45 %B for 3.5 mins.
Injection volume: 10 µL
Injections / sample: 1

*Electrospray mass spectrometer*

Micromass Platform II API Mass Spectrometer, "Chick"

MassLynx 2.4 Software
Cone voltages: -60v
Mode: electrospray negative
Source temperature: 115 °C.
Analyzer vacuum pressures: 0.000043 mBar, 0.000079 mBar
Ions: 499, 413, 369
Electrode: cross-flow

*Electrospray MSMS*

Micromass Quattro II API Mass Spectrometer, "Madeline"

MassLynx 3.1 Software
Cone voltages: -60v
Collision gas energy: 45 V
Mode: electrospray negative
Source temperature: 115 °C.
Analyzer vacuum pressures: 0.000043 mBar, 0.000079 mBar
Primary Ion: 499; 498
Daughter ions: 80, 99, 130, 180, 230; 78
Electrode: Z-spray

# CONFIDENTIAL REPORT

## LOCATIONS AND DATA FROM AVIAN BLOOD PLASMA SAMPLES

TO:

3M Corporation
Minneapolis, Minnesota

BY:

John P. Giesy and Associates
Hilltop Place
2355 Bravender Road
Williamston, Michigan 48895

July 18, 1994

Avian blood plasma samples collected from two species, the bald eagle (*Haliaeetus leucocephalus*) and the Laysan albatross (*Diomedea immutabilis*) were sent to 3M for analysis for $C_8F_{17}SO_2$ and $C_8F_{17}CO_2$. Additional data for these blood samples follows:

*Laysan albatrosses*

All samples of Laysan albatrosses were collected from the Officers Club plot on Sand Island within the Midway Atoll in the north-central Pacific Ocean. Table 1 provides additional data on the birds sampled:

Table 1. Band number, year bird was banded, age at collection of blood sample, blood sample number, and date blood was collected for 5 Laysan albatrosses from Midway Atoll, north-central Pacific Ocean.

| Band Number | Year Banded | Collection Age | Sample Number | Date Collected |
|---|---|---|---|---|
| 1247-60080 | 1993 | 0 | LAC 08093 | 18 May 1993 |
| 1247-60095 | 1993 | 0 | LAC 09593 | 18 May 1993 |
| 1367-32659 | 1985 | 8 | LA 616 | 13 Dec 1992 |
| 1137-90880 | 1987 | 6 | LA 607 | 13 Dec 1992 |
| 1067-19156 | 1978 | 15 | LA 617 | 13 Dec 1992 |

No other data on organochlorine pesticides from these birds is currently available, however, a seperate split sample of each has been archived.

*Bald eagles*

All samples of bald eagles were collected within the Midwest, with one from Minnesota, and two each from the upper and lower peninsulas of Michigan (Figure 1). Table 2 provides additonal data on the birds sampled:

Table 2. Band number, location, county, region, date of blood sample collection, sex, weight, and age in days of 5 nestling bald eagles from Minnesota and Michigan.

| Band No. | Location | County | Region | Date Collected | Sex | Weight | Age in Days |
|---|---|---|---|---|---|---|---|
| 629-31740 | Devil's Lake | Menominee | U. Peninsula, MI | 1989 | Unknown | Unknown | Unknown |
| 629-31748 | Slate River Falls | Baraga | U. Peninsula, MI | 1989 | Unknown | Unknown | Unknown |
| 629-14593 | Steve's Island | St. Louis | Voyageurs NP, MN | 17 June 1992 | Male | 2.7 kg | 82 |
| 629-39332 | Caulkins Creek | Otsego | L. Peninsula, MI | 3 June 1993 | Female | 4.85 kg | 228 |
| 629-39339 | Pickerel Channel | Emmet | L. Peninsula, MI | 5 June 1993 | Female | 4.25 kg | 163 |

# Laboratory Report
# Analysis of Fluorochemicals in Wild Bird Livers

**Laboratory Report No. FACT-GEN-010**

**LR U2392**

*Testing Laboratory*

3M Environmental Technology & Safety Services
3M Environmental Laboratory
Fluorine Analytical Chemistry Team (FACT)
2-3E-09
935 Bush Avenue,
St. Paul, MN 55106

*Laboratory Contact*

Kris Hansen, Ph.D.
Bldg. 2-3E-09
P.O. Box 3331
St. Paul, MN 55133-3331
Phone: (651) 778-6018
FAX: (651) 778-6176

*Requester*

# 1 Introduction

Sixty liver samples collected from various species of birds were characterized with respect to specific fluorochemicals. The birds were classified according to specie and geographic location. Six species of birds are represented in this study, including Sandhill Cranes, Double-Crested Cormorants, White Pelicans, Brown Pelicans, Great Blue Herons, and Brandt's Cormorants. The bird were found at a variety of sites across the United States including Nebraska, New Mexico, Florida, Arizona, California, Louisiana, and Florida. In general, the cause of death to these birds is unknown. Details about the location each bird was found and about the cause of death (if known) are included in Appendix C of this report.

Liver samples were homogenized and extracted using an ion-pairing extraction procedure. The extracts were quantitatively analyzed for PFOS and PFOSA using high-pressure liquid chromatography/electrospray tandem mass spectrometry (HLPC/ESMSMS), and evaluated versus a standard curve extracted from rabbit liver. Analytical details are available in the study binder maintained by FACT in the 3M Environmental Lab. The presence or absence of other known fluorochemical contaminants and metabolites (PFOSAA, M556, M570, POAA, M513, PFHS, and PFDS) was determined by inspection of HPLC/ESMSMS analysis of 2 bird livers within each group. A full list of acronyms is attached in Appendix B.

With the exception of most of the Sandhill Cranes and a single Brown Pelican from California, the extracts from the livers of all of the birds were determined to contain PFOS above the method limit of quantitation (MDL =10 ppb). Extracts from the livers of three of 15 Sandhill Cranes showed some PFOS; two of these extracts contained PFOS levels very near the MDL.

Extracts from one Brandt's Cormorant from San Diego, CA and one White Pelican, from Calipatria, CA showed PFOS levels in great excess of those determined in the extracts from other birds within the same classification (>20x for the cormorant and nearly 10x for the pelican). If these two outlying points are removed and PFOS levels within a particular classification are averaged, some general observations based on the geographical location of the birds' habitat can be made.

With the exception of the Sandhill Cranes, in general, liver from birds collected from the western US (San Diego, CA, Calipatria, CA,) show relatively low levels of PFOS, with an average of less than 50 ppb. Livers from birds collected on the East Coast of the US (Miami, Naples, and Ft. Lauderdale, FL) show moderate PFOS levels, approximately 105 ppb. Extracts from bird livers collected from the Central US (LA and NV) were determined to contain the highest levels of PFOS, approximately 300 ppb.

It is unlikely that the differences associated with PFOS levels in the livers are purely a function of species. In more than one instance, the same species of bird living in a different environment showed significantly different PFOS levels. For example, livers collected from white pelicans in California were determined to contain an average PFOS concentration of 58 ppb, while livers collected from White Pelicans in Nevada showed average PFOS levels of over 350 ppb. Sandhill Cranes are an exception to this observation, as the livers from these birds were determined to be uniformly low in PFOS, regardless of the geographic location of the birds.

With the exception of the Sandhill Cranes, liver extracts from at least one of the two birds screened within each classification were determined to contain PFDS. Liver extracts from several species, including 1 Sandhill Crane, were determined to contain a small amount of PFHS. Nearly 20% of the liver extracts were determined to contain a small amount of PFOSA. No POAA, M513, M556, M570, or PFOSAA was detected in the liver extracts from any of the birds. Appendix A contains these results.

This data indicates that potential geographic trends associated with the environmental distribution of PFOS and other fluorochemicals may be traceable by characterization of low-level of these compounds in animal tissue samples.

Although rigorous quality control measures and 3M Environmental Laboratory Standard Operating Procedures were followed, the acquisition of this data was not necessarily collected according to applicable Good Laboratory Practices. This data is not suitable for distribution to any government agency in defense of product suitability or safety. Results should be considered to be semi-quantitative. Data presented here is the highest quality data available at this time.

## 2 Sample Receipt

On 5/5/98, Jean Burris from 3M Occupational Medicine delivered the bird liver samples to the Environmental Lab; samples were received frozen. The samples were originally sent to Geary Olsen (3M Occupational Medicine) by Scott P. Hansen of the National Wildlife Health Center in Madison, Wisconsin.

## 3 Holding Times

There is no holding time criteria associated with these samples for LC/ESMSMS analysis.

## 4 Methods - Analytical and Preparatory

**Analytical methods**

FACT-M-1.0, Extraction of Potassium Perfluorooctanesulfonate or Other Anionic Fluorochemical Surfactants from Liver for Analysis Using HPLC/ESMS

FACT-M-2.0, Analysis of Fluorochemicals in Liver Extract Using HPLC-Electrospray/Mass Spectrometry

**Sample preparation – aqueous samples, HPLC/ESMSMS: ion-pairing extraction**

Analyte is extracted from a sample matrix with ion pairing reagent [tetrabutyl ammonium hydrogen sulfate (TBA)] in a pH-controlled environment. The cationic reagent selectively targets anionic fluorochemicals. Once the anion-TBA pair is formed, the analyte is transferred into a non-polar organic solvent (ethyl acetate), dried, and reconstituted in methanol for MS analysis.

**HPLC/ESMS and HPLC/ESMSMS: for detailed qualitative work**

In HPLC, an aliquot of extract is injected and passed through a reverse phase liquid chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. For example, in a standard solution PFOS may elute at 8.0 minutes. Retention times between a standard PFOS solution and the analyte extracted from liver in this analysis were matched on the HPLC system to within 1%.

Following HPLC separation, ESMSMS provides a rapid and accurate means for analyzing a wide range of organic compounds, including fluorochemicals. Electrospray, an ionization technique used primarily for detection of molecular ions, is generally operated at relatively mild temperatures. Molecules are ionized, and a primary ion, characteristic of the analyte, is selected. This ion is bombarded with high-energy gas; subsequent collisions create smaller secondary ionic fragments unique to the primary ion, which are detected.

For example, for PFOS ($C_8F_{17}SO_3^-$) analysis, ion 499 is selected as the characteristic primary ion. This ion is fragmented into other ions such as 80 amu (corresponding to $SO_3^-$), and 230 amu ($C_3F_6SO_3^-$). Each of these secondary fragments is detected and can be used to differentiate PFOS from other compounds that might have the same characteristic 499 amu primary ion, but different chemical compositions and secondary ion fragmentation patterns.

# 5 Analysis

## 5.1 Calibration

A mid-level, extracted matrix calibration check standard is analyzed every 5-10 samples to monitor instrumental drift. Calibration check standards are compliant if instrumental response is within +/- 30% of the expected value.

Quantitation of the target analytes is based on linear regression analysis, weighted 1/x, of two extracted matrix curves bracketing each group of samples. Quantitation of each analyte is based in the response of a specific daughter ion using the multiple response-monitoring mode of the instrument. The second curve was not used for PFOS determination; bracketing calibration checks were all compliant, so this exclusion does not adversely affect the data quality.

## 5.2 Blanks

Two extraction blanks, utilizing water as surrogate matrix, are extracted with each batch of samples. Additionally, two samples of blank matrix, used for the extracted calibration curves, are extracted along with each batch of samples and curves.

Blanks are compliant if no target analyte is detected above the limit of detection for a specific analyte. In this study, all blanks were compliant.

## 5.3 Surrogates

Tetra-hydro perfluorooctane sulfonate is used as a surrogate in this analysis. Surrogate response is monitored to confirm gross instrumental failure, if necessary.

## 5.4 Matrix Spikes

Duplicate matrix spike analyses utilizing all target analytes were prepared and analyzed from Sandhill crane liver. Recoveries of PFOS and PFOSA were within the acceptable range of 70-130%.

## 5.5 Laboratory Control Samples

Laboratory Control Samples are not a component of this study.

## 5.6 Sample Related Comments

Matrix spikes were prepared for a single specie of bird. It is assumed that spike recoveries would be similar for all species. No standard material for PFDS is available. Compound identification is based on reasonable retention time and reasonable daughter ion spectrum. Until standard material is available, identification of this analyte should be considered to be preliminary.

Quantitation for PFOS was conducted by monitoring the transition 499 → 99. Some positive interference was observed when monitoring the 499 → 80 transition in the bird liver matrix. Transition parameters for the other target analytes are listed below.

**Table 1: Analyte Transitions for HPLC-ESMSMS Analysis**

| Compound | Primary Ion | Transition Monitored |
|---|---|---|
| PFOS | 499 | 99 |
| PFOSA | 498 | 78 |
| PFOSAA | 584 | 169 |
| M556 | 556 | 169 |
| M570 | 570 | 169 |
| POAA | 413 | 169 |
| M513 | 469 | 169 |
| PFHS | 399 | 99 |
| PFDS | 599 | 99 |

## 6 Data Summary

With the exception of most of the Sandhill Cranes and a single Brown Pelican from California, the extracts from the livers of all of the birds were determined to contain PFOS above the method limit of quantitation (MDL =10 ppb). Extracts from the livers of three of 15 Sandhill Cranes showed some PFOS; two of these extracts contained PFOS levels very near the MDL.

Extracts from one Brandt's Cormorant from San Diego, CA and one White Pelican, from Calipatria, CA showed PFOS levels in great excess of those determined in the extracts from other birds within the same classification (>20x for the cormorant and nearly 10x for the pelican). If these two outlying points are removed and PFOS levels within a particular classification are averaged, some general observations based on the geographical location of the birds' habitat can be made.

With the exception of the Sandhill Cranes, in general, liver from birds collected from the western US (San Diego, CA, Calipatria, CA,) show relatively low levels of PFOS, with an average of less than 50 ppb. Livers from birds collected on the East Coast of the US (Miami, Naples, and Ft. Lauderdale, FL) show moderate PFOS levels, approximately 105 ppb. Extracts from bird livers collected from the Central US (LA and NV) were determined to contain the highest levels of PFOS, approximately 300 ppb.

It is unlikely that the differences associated with PFOS levels in the livers are purely a function of species. In more than one instance, the same species of bird living in a different environment showed significantly different PFOS levels. For example, livers collected from white pelicans in California were determined to contain an average PFOS concentration of 58 ppb, while livers collected from White Pelicans in Nevada showed average PFOS levels of over 350 ppb. Sandhill Cranes are an exception to this observation, as the livers from these birds were determined to be uniformly low in PFOS, regardless of the geographic location of the birds.

With the exception of the Sandhill Cranes, liver extracts from at least one of the two birds screened within each classification were determined to contain PFDS. Liver extracts from several species, including 1 Sandhill Crane, were determined to contain a small amount of PFHS. Nearly 20% of the liver extracts were determined to contain a small amount of PFOSA. No POAA, M513, M556, M570, or PFOSAA was detected in the liver extracts from any of the birds.

This data indicates that potential geographic trends associated with the environmental distribution of PFOS and other fluorochemicals may be traceable by characterization of low-level of these compounds in animal tissue samples.

This report reflects the most accurate data available at this time. All numbers should be considered to be semi-quantitative.

## 7 Conclusion

This data indicates that potential geographic trends associated with the environmental distribution of PFOS may be traceable by characterization of low-level fluorochemicals in animal tissue samples.

## 8 Data / Sample Retention

Samples and extracts will be retained for future evaluation, if necessary.

## 9 Attachments

9.1 **Attachment A:** Tables of Qualitative and Quantitative Results from the Analysis of Liver Samples
9.2 **Attachment B:** Definitions of Fluorochemical Acronyms
9.3 **Attachment C:** Sample description and sample receipt data
9.4 **Attachment D:** Extraction and Analytical data

## 10 Signatures

Harold Johnson, Analytical Chemist — 4/28/99 Date

Kris Hansen, Ph.D., Principal Analytical Investigator — 4/28/99 Date

# Analysis of Wild Bird Livers.

# = not detected NA = not analyzed x = detected LOQ= Limit of Quantitation

| Sample No. | Species | Location | PFOS ppb | PFOSA ppb | PFHS | PFDS |
|---|---|---|---|---|---|---|
| 1 | Sandhill Crane | Kearney, NE | 41 | <LOQ | x | # |
| 2 | Sandhill Crane | Kearney, NE | <LOQ | <LOQ | NA | NA |
| 3 | Sandhill Crane | Kearney, NE | <LOQ | <LOQ | # | # |
| 4 | Sandhill Crane | Kearney, NE | <LOQ | <LOQ | NA | NA |
| 5 | Sandhill Crane | Kearney, NE | <LOQ | <LOQ | NA | NA |
| 6 | Sandhill Crane | Chochise Co., AZ | <LOQ | <LOQ | # | NA |
| 7 | Sandhill Crane | Chochise Co., AZ | <LOQ | <LOQ | # | NA |
| 8 | Sandhill Crane | Chochise Co., AZ | <LOQ | <LOQ | NA | NA |
| 9 | Sandhill Crane | Chochise Co., AZ | <LOQ | <LOQ | NA | NA |
| 10 | Sandhill Crane | Chochise Co., AZ | <LOQ | <LOQ | NA | NA |
| 11 | White Pelican | Calipatria, CA | 35 | <LOQ | x | x |
| 12 | White Pelican | Calipatria, CA | 1293 | <LOQ | x | x |
| 13 | White Pelican | Calipatria, CA | 29 | <LOQ | NA | NA |
| 14 | White Pelican | Calipatria, CA | 15 | <LOQ | NA | NA |
| 15 | White Pelican | Calipatria, CA | 153 | <LOQ | NA | NA |
| 16 | Brandt's Cormorant | San Diego, CA | 53 | 80 | # | x |
| 17 | Brandt's Cormorant | San Diego, CA | 46 | 31 | NA | NA |
| 18 | Brandt's Cormorant | San Diego, CA | 46 | 34 | NA | NA |
| 19 | Brandt's Cormorant | San Diego, CA | 80 | 13 | NA | NA |
| 20 | Brandt's Cormorant | San Diego, CA | 2055 | 94 | x | x |
| 21 | Dbl. Crested Cormorant | St. Martinville, LA | 59 | 20 | x | x |
| 22 | Dbl. Crested Cormorant | St. Martinville, LA | 145 | 7 | # | x |
| 23 | Dbl. Crested Cormorant | St. Martinville, LA | 333 | 23 | NA | NA |
| 24 | Dbl. Crested Cormorant | St. Martinville, LA | 76 | <LOQ | NA | NA |
| 25 | Dbl. Crested Cormorant | St. Martinville, LA | 170 | <LOQ | NA | NA |
| 26 | Brown Pelican | Miami, FL | 106 | 58 | # | x |
| 27 | Brown Pelican | Miami, FL | 134 | 11 | # | x |
| 28 | Brown Pelican | Miami, FL | 125 | 10 | NA | NA |
| 29 | Brown Pelican | Miami, FL | 159 | 47 | NA | NA |
| 30 | Brown Pelican | Miami, FL | 48 | 11 | NA | NA |
| 31 | Sandhill Crane | Valenica Co., NM | <LOQ | <LOQ | # | # |
| 32 | Sandhill Crane | Valenica Co., NM | <LOQ | <LOQ | # | # |
| 33 | Sandhill Crane | Socorro Co., NM | <LOQ | <LOQ | NA | NA |
| 34 | Sandhill Crane | Socorro Co., NM | <LOQ | <LOQ | NA | NA |
| 35 | Sandhill Crane | Valenica Co., NM | <LOQ | <LOQ | NA | NA |
| 36 | Dbl. Crested Cormorant | Naples, FL | 212 | 9 | # | x |
| 37 | Dbl. Crested Cormorant | Naples, FL | 10 | <LOQ | # | # |
| 38 | Dbl. Crested Cormorant | Naples, FL | 52 | <LOQ | NA | NA |
| 39 | Dbl. Crested Cormorant | Naples, FL | 100 | 35 | NA | NA |
| 40 | Dbl. Crested Cormorant | Naples, FL | 152 | 24 | NA | NA |
| 41 | Brown Pelican | Calipatria, CA | 16 | <LOQ | x | x |
| 42 | Brown Pelican | Calipatria, CA | 36 | <LOQ | # | NA |
| 43 | Brown Pelican | Calipatria, CA | <LOQ | <LOQ | NA | NA |
| 44 | Brown Pelican | Calipatria, CA | 6 | <LOQ | NA | NA |
| 45 | Brown Pelican | Calipatria, CA | 32 | <LOQ | NA | NA |
| 46 | Great Blue Heron | St. Martinville, LA | 188 | <LOQ | # | x |
| 47 | Great Blue Heron | St. Martinville, LA | 59 | <LOQ | # | x |
| 48 | Great Blue Heron | St. Martinville, LA | 1061 | 19 | NA | NA |
| 49 | Great Blue Heron | St. Martinville, LA | 261 | 8 | NA | NA |
| 50 | Great Blue Heron | St. Martinville, LA | 173 | 12 | NA | NA |
| 51 | White Pelican | Fallon, NV | 141 | <LOQ | # | x |
| 52 | White Pelican | Fallon, NV | 362 | <LOQ | x | x |
| 53 | White Pelican | Fallon, NV | 927 | <LOQ | NA | NA |
| 54 | White Pelican | Fallon, NV | 133 | <LOQ | NA | NA |
| 55 | White Pelican | Fallon, NV | 291 | <LOQ | NA | NA |
| 56 | Brown Pelican | Ft. Lauderdale, FL | 194 | 11 | x | x |
| 57 | Brown Pelican | Ft. Lauderdale, FL | 75 | 8 | # | x |
| 58 | Brown Pelican | Ft. Lauderdale, FL | 71 | <LOQ | NA | NA |
| 59 | Brown Pelican | Ft. Lauderdale, FL | 31 | <LOQ | NA | NA |
| 60 | Brown Pelican | Ft. Lauderdale, FL | 91 | 8 | NA | NA |

# 3M Environmental Lab - Fluorine Analytical Chemistry Team

**Semi-quantitative results of the analysis of wild bird livers**

| SAMPLE NUMBER | SPECIES | LOCATION | Liver [PFOS] ppb | Liver [PFOSA] ppb | Avg. [PFOS] ppb | Avg. [PFOS]ppb* |
|---|---|---|---|---|---|---|
| 4291001 | sandhill crane | Kearney, NE | 41 | <LOQ | | |
| 4291008 | sandhill crane | Kearney, NE | <LOQ | <LOQ | | |
| 4291009 | sandhill crane | Kearney, NE | <LOQ | <LOQ | | |
| 4291021 | sandhill crane | Kearney, NE | <LOQ | <LOQ | | |
| 4291058 | sandhill crane | Kearney, NE | <LOQ | <LOQ | <LOQ | <LOQ |
| 4296008 | sandhill crane | Cochise Co., AZ | <LOQ | <LOQ | | |
| 4296009 | sandhill crane | Cochise Co., AZ | <LOQ | <LOQ | | |
| 4296014 | sandhill crane | Cochise Co., AZ | <LOQ | <LOQ | | |
| 4296017 | sandhill crane | Cochise Co., AZ | <LOQ | <LOQ | | |
| 4296018 | sandhill crane | Cochise Co., AZ | <LOQ | <LOQ | <LOQ | <LOQ |
| 4337001 | white pelican | Calipatria, CA | 35 | <LOQ | | |
| 4337003 | white pelican | Calipatria, CA | 1293 | <LOQ | | |
| 4337004 | white pelican | Calipatria, CA | 29 | <LOQ | | |
| 4337017 | white pelican | Calipatria, CA | 15 | <LOQ | | |
| 4337018 | white pelican | Calipatria, CA | 155 | <LOQ | 305 | 58 |
| 4306020 | brandt's cormorant | San Diego, CA | 53 | 80 | | |
| 4306021 | brandt's cormorant | San Diego, CA | 46 | 31 | | |
| 4306022 | brandt's cormorant | San Diego, CA | 46 | 34 | | |
| 4306051 | brandt's cormorant | San Diego, CA | 80 | 13 | | |
| 4306056 | brandt's cormorant | San Diego, CA | 2055 | 94 | 456 | 56 |
| 4312001 | double-crested cormorant | St. Martinville, LA | 59 | 20 | | |
| 4312002 | double-crested cormorant | St. Martinville, LA | 145 | 7 | | |
| 4312005 | double-crested cormorant | St. Martinville, LA | 333 | 23 | | |
| 4312006 | double-crested cormorant | St. Martinville, LA | 76 | <LOQ | | |
| 4312007 | double-crested cormorant | St. Martinville, LA | 170 | <LOQ | 157 | 157 |
| 4360020 | brown pelican | Miami, FL | 106 | 58 | | |
| 4360021 | brown pelican | Miami, FL | 134 | 11 | | |
| 4360022 | brown pelican | Miami, FL | 125 | 10 | | |
| 4360023 | brown pelican | Miami, FL | 159 | 47 | | |
| 4360024 | brown pelican | Miami, FL | 48 | 11 | 115 | 115 |
| 4295007 | sandhill crane | Valenica Co., NM | <LOQ | <LOQ | | |
| 4295008 | sandhill crane | Valenica Co., NM | <LOQ | <LOQ | | |
| 4295014 | sandhill crane | Socorro Co., NM | <LOQ | <LOQ | | |
| 4295017 | sandhill crane | Socorro Co., NM | <LOQ | <LOQ | | |
| 4295018 | sandhill crane | Valenica Co., NM | <LOQ | <LOQ | <LOQ | <LOQ |
| 4392003 | double-crested cormorant | Naples, FL | 212 | 9 | | |
| 4392004 | double-crested cormorant | Naples, FL | 10 | <LOQ | | |
| 4392005 | double-crested cormorant | Naples, FL | 52 | <LOQ | | |
| 4392006 | double-crested cormorant | Naples, FL | 100 | 35 | | |
| 4392007 | double-crested cormorant | Naples, FL | 152 | 24 | 105 | 105 |
| 4336003 | brown pelican | Calipatria, CA | 16 | <LOQ | | |
| 4336006 | brown pelican | Calipatria, CA | 36 | <LOQ | | |
| 4336010 | brown pelican | Calipatria, CA | <LOQ | <LOQ | | |
| 4336012 | brown pelican | Calipatria, CA | 6 | <LOQ | | |
| 4336013 | brown pelican | Calipatria, CA | 32 | <LOQ | <22 | 22 |
| 4311003 | great blue heron | St. Martinville, LA | 188 | <LOQ | | |
| 4311005 | great blue heron | St. Martinville, LA | 59 | <LOQ | | |
| 4311006 | great blue heron | St. Martinville, LA | 1061 | 19 | | |
| 4311007 | great blue heron | St. Martinville, LA | 261 | 8 | | |
| 4311009 | great blue heron | St. Martinville, LA | 173 | 12 | 348 | 348 |
| 4328007 | white pelican | Fallon, NV | 141 | <LOQ | | |
| 4328053 | white pelican | Fallon, NV | 362 | <LOQ | | |
| 4328054 | white pelican | Fallon, NV | 927 | <LOQ | | |
| 4328056 | white pelican | Fallon, NV | 133 | <LOQ | | |
| 4326057 | white pelican | Fallon, NV | 291 | <LOQ | 371 | 371 |
| 4378005 | brown pelican | Ft. Lauderdale, FL | 194 | 11 | | |
| 4378011 | brown pelican | Ft. Lauderdale, FL | 75 | 8 | | |
| 4378013 | brown pelican | Ft. Lauderdale, FL | 71 | <LOQ | | |
| 4378014 | brown pelican | Ft. Lauderdale, FL | 31 | <LOQ | | |
| 4378016 | brown pelican | Ft. Lauderdale, FL | 91 | 8 | 92 | 92 |

* Average when outliers are excluded

LOQ = limit of quantitation (approx. 6 ppb)

| | | |
|---|---|---|
| MS-Sandhill Crane | 77% | 96% |
| MSD-Sandhill Crane | 64% | 64% |

**Qualitataive results of the analysis of wild bird livers**

| SAMPLE NUMBER | SPECIES | LOCATION | Liver [PFOS] ppb | PFHS? | PFDS? |
|---|---|---|---|---|---|
| 4291001 | sandhill crane | Kearney, NE | 41 | X | ** |
| 4291008 | sandhill crane | Kearney, NE | <LOQ | not measured | not measured |
| 4291009 | sandhill crane | Kearney, NE | <LOQ | ** | ** |
| 4291021 | sandhill crane | Kearney, NE | <LOQ | not measured | not measured |
| 4291058 | sandhill crane | Kearney, NE | <LOQ | not measured | not measured |
| 4296008 | sandhill crane | Cochise Co., AZ | <LOQ | ** | X |
| 4296009 | sandhill crane | Cochise Co., AZ | <LOQ | ** | X |
| 4296014 | sandhill crane | Cochise Co., AZ | <LOQ | not measured | not measured |
| 4296017 | sandhill crane | Cochise Co., AZ | <LOQ | not measured | not measured |
| 4296018 | sandhill crane | Cochise Co., AZ | <LOQ | not measured | not measured |
| 4337001 | white pelican | Calipatria, CA | 35 | X | X |
| 4337003 | white pelican | Calipatria, CA | 1358 | X | X |
| 4337004 | white pelican | Calipatria, CA | 30 | not measured | not measured |
| 4337017 | white pelican | Calipatria, CA | 17 | not measured | not measured |
| 4337018 | white pelican | Calipatria, CA | 167 | not measured | not measured |
| 4306020 | brandt's cormorant | San Diego, CA | 57 | ** | X |
| 4306021 | brandt's cormorant | San Diego, CA | 50 | not measured | not measured |
| 4306022 | brandt's cormorant | San Diego, CA | 46 | not measured | not measured |
| 4306051 | brandt's cormorant | San Diego, CA | 85 | not measured | not measured |
| 4306056 | brandt's cormorant | San Diego, CA | 2176 | X | X |
| 4312001 | double-crested cormorant | St. Martinville, LA | 61 | X | X |
| 4312002 | double-crested cormorant | St. Martinville, LA | 149 | ** | X |
| 4312005 | double-crested cormorant | St. Martinville, LA | 336 | not measured | not measured |
| 4312006 | double-crested cormorant | St. Martinville, LA | 78 | not measured | not measured |
| 4312007 | double-crested cormorant | St. Martinville, LA | 178 | not measured | not measured |
| 4360020 | brown pelican | Miami, FL | 115 | ** | X |
| 4360021 | brown pelican | Miami, FL | 149 | ** | X |
| 4360022 | brown pelican | Miami, FL | 126 | not measured | not measured |
| 4360023 | brown pelican | Miami, FL | 166 | not measured | not measured |
| 4360024 | brown pelican | Miami, FL | 49 | not measured | not measured |
| 4295007 | sandhill crane | Valenica Co., NM | 11 | ** | ** |
| 4295008 | sandhill crane | Valenica Co., NM | 9 | ** | ** |
| 4295017 | sandhill crane | Socorro Co., NM | <LOQ | not measured | not measured |
| 4295018 | sandhill crane | Valenica Co., NM | <LOQ | not measured | not measured |
| 4295014 | sandhill crane | Socorro Co., NM | <LOQ | not measured | not measured |
| 4392003 | double-crested cormorant | Naples, FL | 226 | ** | X |
| 4392004 | double-crested cormorant | Naples, FL | 11 | ** | ** |
| 4392005 | double-crested cormorant | Naples, FL | 54 | not measured | not measured |
| 4392006 | double-crested cormorant | Naples, FL | 103 | not measured | not measured |
| 4392007 | double-crested cormorant | Naples, FL | 154 | not measured | not measured |
| 4336003 | brown pelican | Calipatria, CA | 16 | X | X |
| 4336006 | brown pelican | Calipatria, CA | 37 | ** | X |
| 4336010 | brown pelican | Calipatria, CA | <LOQ | not measured | not measured |
| 4336012 | brown pelican | Calipatria, CA | 7 | not measured | not measured |
| 4336013 | brown pelican | Calipatria, CA | 33 | not measured | not measured |
| 4311003 | great blue heron | St. Martinville, LA | 202 | ** | X |
| 4311005 | great blue heron | St. Martinville, LA | 61 | ** | X |
| 4311006 | great blue heron | St. Martinville, LA | 1143 | not measured | not measured |
| 4311007 | great blue heron | St. Martinville, LA | 274 | not measured | not measured |
| 4311009 | great blue heron | St. Martinville, LA | 178 | not measured | not measured |
| 4328007 | white pelican | Fallon, NV | 146 | ** | X |
| 4328053 | white pelican | Fallon, NV | 368 | X | X |
| 4328054 | white pelican | Fallon, NV | 941 | not measured | not measured |
| 4328056 | white pelican | Fallon, NV | 141 | not measured | not measured |
| 4326057 | white pelican | Fallon, NV | 294 | not measured | not measured |
| 4378005 | brown pelican | Ft. Lauderdale, FL | 212 | X | X |
| 4378011 | brown pelican | Ft. Lauderdale, FL | 76 | ** | X |
| 4378013 | brown pelican | Ft. Lauderdale, FL | 72 | not measured | not measured |
| 4378014 | brown pelican | Ft. Lauderdale, FL | 32 | not measured | not measured |
| 4378016 | brown pelican | Ft. Lauderdale, FL | 94 | not measured | not measured |

X = detected ** = not detected

**PFOSAA, M556, M570, POAA, and M513 were not detected in the livers of any birds.**

## 3M Environmental Lab - Fluorine Analytical Chemistry Team

| PFS's | | |
|---|---|---|
| PFHS | perfluorohexanesulfonate | C6F13SO3- |
| PFOS | perfluorooctanesulfonate | C8F17SO3- |
| PFOSA | | C8F17SO2NH2 |
| PFOSAA | | C8F17SO2N((CH2CH3)(CH2COO))- |
| M556 | | C8F17SO2N((H)(CH2COO))- |
| M570 | | C8F17SO2N((CH3)(CH2COO))- |
| PFOSEA | perfluorooctanesulfonylethyl amide | C8F17SO2N((CH2CH3)(H)) |
| Et-FOSE-OH | | C8F17SO2N((CH2CH3)(CH2CH2OH)) |
| PFDS | perfluorodecanesulfonate | C10F21SO3-** |

| PFCA's | | |
|---|---|---|
| POAA | perfluorooctanoate | C7F15COO- |
| M513 | perfluorodecanoate | C9F19COO- |
| M613 | perfluorododecanoate | C11F23COO- |
| M463 | | C8F17COO- |
| M563 | | C10F21COO- |
| M363 | | C6F13COO- |

PFS = perfluorinated sulfoante
PFCA = perfluorinated carboxylic acid

** no standard material available for confirmation;