



## **Centre Analytical Laboratories, Inc.**

3048 Research Drive  
Phone: (814) 231-8032

State College, PA 16801

www.centrelab.com

Fax: (814) 231-1253 or (814) 231-1580

---

# **Analytical Report**

**Fluorochemical Characterization of Surface Water Samples**

**Pensacola, FI (W2176)**

**Centre Analytical Laboratory Report No. 023-014C (Revision 1)**

**Revision Data 3/21/01**

---

### ***Testing Laboratory***

Centre Analytical Laboratory, Inc.  
3048 Research Drive  
State College, PA 16801

---

### ***3M Environmental Laboratory Contact***

Kent R. Lindstrom  
Bldg. 2-3E-09  
P.O. Box 33331  
St. Paul, MN 55133-3331  
Phone: (651) 778-5352

---

### ***Requester***

Kris J. Hansen, Ph.D.  
3M Environmental Technology & Safety Services  
Bldg. 2-3E-09  
P.O. Box 33331  
St. Paul, MN 55133-3331

## **1 Introduction**

Results are reported for the analysis of a series of surface water samples received by Centre Analytical Laboratories, Inc. (Centre) from the 3M Environmental Laboratory. The samples were collected from Pensacola, FL. The Centre study number assigned to the project is 023-014.

Specific fluorochemical characterization by liquid chromatography / tandem mass spectrometry (LC/MS/MS) was requested for all samples. A total of 8 samples were received for analysis.

The samples were prepared and analyzed by LC/MS/MS for the following list of fluorochemicals:

- Table 1: Target Analysis

| Compound Name                 | Acronym |
|-------------------------------|---------|
| Perfluorooctane Sulfonate     | PFOS    |
| Perfluorooctane Sulfonylamide | PFOSA   |
| Perfluorooctanoate            | POAA    |

The analytical method used was validated by Centre. The validation protocol and results are on file with Centre. Data presented here is the highest quality data available at this time.

## **2 Sample Receipt**

The samples were submitted in individual plastic containers and were not preserved. Sixteen individual sample containers were received. Samples were received on 05/03/00. The sample collection dates were not supplied. Chain-of-custody information is presented in Attachment C.

## **3 Holding Times**

The analytical method used was validated against a maximum holding time of 14 days. The stability of the analytes of interest for longer periods has not been determined. However, it should be noted that field fortifications in water and other matrices have shown acceptable recoveries at 100 and 1000 ng/L for periods longer than 14 days.

## **4 Methods - Analytical and Preparatory**

### **4.1 LC/MS/MS**

#### **4.1.1 Sample Preparation for LC/MS/MS Analysis**

Samples were initially treated with 200  $\mu$ L of 250 mg/L sodium thiosulfate solution to remove residual chlorine. Solid phase extraction (SPE) was used to prepare the samples for LC/MS/MS analysis. A forty-milliliter portion of sample was transferred to a C<sub>18</sub> SPE cartridge. The cartridge was first eluted with 5 mL of 40% methanol in water solution. The eluate was discarded and the SPE column was then eluted with 100% methanol. A 5 ml portion of methanol was collected for analysis by LC/MS/MS. This treatment resulted in an eight-fold concentration of the samples prior to analysis.

#### **4.1.2 Sample Analysis by LC/MS/MS**

In HPLC, an aliquot of extract is injected and passed through a liquid-phase 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. Following HPLC separation, ES/MS provides a rapid and accurate means for analyzing a wide range of organic compounds, including fluorochemicals. Electrospray is generally operated at relatively mild temperatures; molecules are ionized, fragmented, and detected. Ions characteristic of known fluorochemicals are observed and quantitated against standards.

A Hewlett-Packard HP1100 HPLC system coupled to a Micromass Ultima MS/MS was used to analyze the sample extracts. Analysis was performed using selected reaction monitoring (SRM). Samples were extracted on 5/17/00 and analyzed by MS/MS on 5/19/00. The HPLC and MS/MS methods used for analysis and instrument parameters can be found in attachment D.

## **5 Analysis**

### **5.1 Calibration**

A 7-point calibration curve was analyzed at the beginning and end of the analytical sequence for the compounds of interest. The calibration points were prepared at 0, 25, 50, 100, 250, 500, and 1000 ng/L (ppt). The response of the quantitation ion versus the concentration was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and correlation coefficient ( $r$ ) and coefficient of determination ( $r^2$ ) were determined. A calibration curve is acceptable if  $r \geq 0.985$  ( $r^2 \geq 0.970$ ).

Calibration standards are prepared using the same SPE procedure used for samples.

Calibration check standards were analyzed periodically (every three to five sample injections) throughout the analysis sequence. Compliance is obtained if the standard analyte concentrations are within  $\pm 20\%$  of the actual value.

For the results reported here, calibration criteria were met.

## **5.2 Blanks**

Extraction blanks were prepared and analyzed with every extraction batch of samples. The extraction blanks should not have any target analytes present at or above the concentration of the low-level calibration standard. For these samples, the extraction blanks were compliant.

Instrument blanks in the form of clean methanol solvent were also analyzed after every high-level calibration standard, and after known high-level samples. Again, the blanks should not have any target analytes present at or above the low-level calibration standard. For the samples presented here the instrument blanks are compliant.

## **5.3 Surrogates**

Surrogate spikes are not a component of the LC/MS/MS analytical method.

## **5.4 Matrix Spikes**

Matrix spikes were prepared for every field sample (excluding blanks) at a concentration of 100 ng/L using all compounds of interest. Matrix spike recoveries are given in Attachment B. All laboratory matrix spikes showed recoveries of all compounds between 70-130%.

Field spikes were prepared on sample MC-301H at a concentration of 100 ng/L using all compounds of interest. The field spike is identified as sample MC-304H. Field spike recoveries are also given in Attachment B. All field spikes showed recoveries of all compounds between 70-130%.

## **5.5 Duplicates**

All field samples (excluding blanks) were analyzed in duplicate. Results are given along with the sample results in Attachment A.

## **5.6 Laboratory Control Samples**

MilliQ water was spiked with all compound of interest at 25 and 250 ng/L. All recoveries for all compounds were between 70-130% in each LCS. Recoveries are given with the raw data in Attachment D.

## **5.7 Sample Related Comments**

Field blank samples consisted of empty containers. Forty milliliters of type I water filtered through a hypercarb cartridge was added to the empty container and analyzed in the same manner as the other samples.

# **6 Data Summary**

Please see Attachment A for a detailed listing of the analytical results.

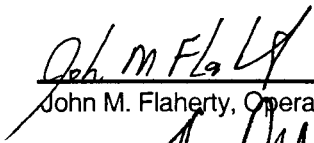
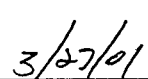
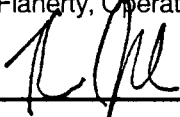
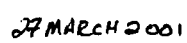
# **7 Data/Sample Retention**

Samples are disposed of one month after the report is issued unless otherwise specified. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by Centre.

## 8 Attachments

- 8.1 Attachment A: Results
- 8.2 Attachment B: Matrix Spike Recoveries (Field and Laboratory Spikes)
- 8.3 Attachment C: Chain of Custody
- 8.4 Attachment D: LC/MS/MS Raw Analytical Data

## 9 Signatures

|                                                                                   |                                                                                     |
|-----------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|
|  |  |
| John M. Flaherty, Operations Manager                                              | Date                                                                                |
|  |  |
| Kevin J Lloyd, Vice President                                                     | Date                                                                                |

Other Lab Members Contributing to Data

Enaksha Wickremesinhe  
Karen Smith  
David Bell



# Centre Analytical Laboratories, Inc.

3048 Research Drive, State College PA 16801 814-231-8032 FAX 814-231-1253

## Analytical Results W2176 Pensacola, FI

| 3M Sample Identification | Sample Description                 | PFOS (ng/L) | PFOSA (ng/L) | POAA (ng/L) |
|--------------------------|------------------------------------|-------------|--------------|-------------|
| MC-301H                  | Site 1 P/N Surface Water           | NQ          | ND           | ND          |
| MC-303H                  | Site 1 P/N Surface Water Duplicate | NQ          | ND           | ND          |
| MC-306H                  | Site 2 P/N Surface Water           | 28.5        | ND           | ND          |
| NA                       | Site 2 P/N Surface Water Duplicate | NQ          | ND           | ND          |
| MC-307H                  | Site 3 P/N Surface Water           | NQ          | ND           | ND          |
| NA                       | Site 3 P/N Surface Water Duplicate | NQ          | ND           | ND          |
| MC-308H                  | Field Blank-P/N Empty              | ND          | ND           | ND          |
| MC-384H                  | Quiet Surface Water                | NQ          | ND           | ND          |
| NA                       | Quiet Surface Water Duplicate      | NQ          | ND           | ND          |

Limit of Detection (LOD) for the procedure is approximately 2.5 ng/L for PFOS and PFOSA and 7.5 ng/L for POAA

Limit of Quantitation (LOQ) for the procedure is 25 ng/L for all compounds

ND - Compound not detected

NQ - Compound detected at a level between the LOD and LOQ. Result is not quantifiable.

ND < LOD < NQ < LOQ



## Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID:

Spiked Amount (ng/L):

|       | Sample<br>Concentration<br>(ng/L) | Matrix Spike<br>Result<br>(ng/L) | Matrix Spike<br>Result<br>(% Recovery) | Criteria<br>(Pass / Fail) |
|-------|-----------------------------------|----------------------------------|----------------------------------------|---------------------------|
| PFOS  | 20.8                              | 102                              | 81.2                                   | PASS                      |
| PFOSA | 0                                 | 105                              | 105.0                                  | PASS                      |
| POAA  | 0                                 | 111                              | 111.0                                  | PASS                      |

Lower Recovery Limit:

Upper Recovery Limit:

Note: Sample results less than 25 ng/L are reported as NQ in the results section as they are below the limit of quantitation. Results are given in this table for recovery calculations only.

Also note that sample MC-305H is a laboratory spike of sample MC-301H

## Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: MC-306H

Spiked Amount (ng/L): 100

|       | Sample<br>Concentration<br>(ng/L) | Matrix Spike<br>Result<br>(ng/L) | Matrix Spike<br>Result<br>(% Recovery) | Criteria<br>(Pass / Fail) |
|-------|-----------------------------------|----------------------------------|----------------------------------------|---------------------------|
| PFOS  | 28.5                              | 124                              | 95.5                                   | PASS                      |
| PFOSA | 0                                 | 98.5                             | 98.5                                   | PASS                      |
| POAA  | 0                                 | 101                              | 101.0                                  | PASS                      |

Lower Recovery Limit: 70

Upper Recovery Limit: 130



## Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID:

Spiked Amount (ng/L):

|       | Sample<br>Concentration<br>(ng/L) | Matrix Spike<br>Result<br>(ng/L) | Matrix Spike<br>Result<br>(% Recovery) | Criteria<br>(Pass / Fail) |
|-------|-----------------------------------|----------------------------------|----------------------------------------|---------------------------|
| PFOS  | 20.9                              | 109                              | 88.1                                   | PASS                      |
| PFOSA | 0                                 | 98.2                             | 98.2                                   | PASS                      |
| POAA  | 0                                 | 102                              | 102.0                                  | PASS                      |

Lower Recovery Limit:

Upper Recovery Limit:

Note: Sample results less than 25 ng/L are reported as NQ in the results section as they are below the limit of quantitation. Results are given in this table for recovery calculations only.

## Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: MC-384H

Spiked Amount (ng/L): 100

|       | Sample<br>Concentration<br>(ng/L) | Matrix Spike<br>Result<br>(ng/L) | Matrix Spike<br>Result<br>(% Recovery) | Criteria<br>(Pass / Fail) |
|-------|-----------------------------------|----------------------------------|----------------------------------------|---------------------------|
| PFOS  | 15.7                              | 107                              | 91.3                                   | PASS                      |
| PFOSA | 0                                 | 88.2                             | 88.2                                   | PASS                      |
| POAA  | 0                                 | 94.0                             | 94.0                                   | PASS                      |

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Note: Sample results less than 25 ng/L are reported as NQ in the results section as they are below the limit of quantitation. Results are given in this table for recovery calculations only.

## Attachment B: LC/MS/MS Field Spike Recovery

Sample ID:

Spiked Amount (ng/L):

|       | Sample<br>Concentration<br>(ng/L) | Matrix Spike<br>Result<br>(ng/L) | Matrix Spike<br>Result<br>(% Recovery) | Criteria<br>(Pass / Fail) |
|-------|-----------------------------------|----------------------------------|----------------------------------------|---------------------------|
| PFOS  | 20.8                              | 93.6                             | 72.8                                   | PASS                      |
| PFOSA | 0                                 | 80.2                             | 80.2                                   | PASS                      |
| POAA  | 0                                 | 96.9                             | 96.9                                   | PASS                      |

Lower Recovery Limit:

Upper Recovery Limit:

Note: Sample results less than 25 ng/L are reported as NQ in the results section as they are below the limit of quantitation. Results are given in this table for recovery calculations only.

Also note that sample MC-304H is a field spike of sample MC-301H

# 3M Environmental Laboratory

Form 38778 - PWO

Shipping Address:  
3M Bldg 2-3E-09  
935 Bush Avenue  
St Paul, MN 55106

Telephone:  
Sample Receiving: (651) 778-4948  
Alternate: (651) 778-6753  
FAX: (651) 778-6176

## Chain of Custody /Request for Laboratory Analytical

14268

3M Env. Lab Project #  
For Internal Use Only

Project ID/Project Name GE0017/Water Samples taken at Pensacola, FL sent from Battelle  
Template #  
Project Lead KJH  
Dept. # (main) 4291  
Final Report Due Date  
Internal Due Date  
Class/Job/Project # 0010535009

wa176

|                    |                  |                                      |                |                       |
|--------------------|------------------|--------------------------------------|----------------|-----------------------|
| Report Results to: | Contact Name     | <u>Kris Hansen</u>                   | Date Available | <u>050300</u>         |
|                    | Company          | <u>3m</u>                            | Date Due       |                       |
|                    | Mailing Address  | <u>Building 2-3E-09 PO Box 33331</u> | Contract Lab   | <u>CENTRE</u>         |
|                    | City, State, Zip | <u>St. Paul, MN 55133</u>            |                |                       |
|                    | Telephone #      | <u>(651) 778-6018</u>                | FAX #          | <u>(651) 778-6176</u> |

Special Instructions and/or Specific Regulatory Requirements:  
(method, limit of detection, reporting units, etc.)

### Preservatives:

| HNO <sub>3</sub> | H <sub>2</sub> SO <sub>4</sub> | VOCs | None | Other |
|------------------|--------------------------------|------|------|-------|
|                  |                                |      |      |       |

Total Number of Containers

### Analysis Requested:

Complete below. Attach any associated information.

| Item # | Client Sample Identification                                                      | 3M LIMS#     | Date Sampled | Time Sampled | Matrix/Media | Enter the number of containers of each | (Enter an 'X' in the box below to indicate request) |
|--------|-----------------------------------------------------------------------------------|--------------|--------------|--------------|--------------|----------------------------------------|-----------------------------------------------------|
| 1.     | MC-301H-Site 1-PIU Ion Pair HPLC-ESMS Surface water - Pensacola, FL               | <u>wa176</u> | <u>N/A</u>   | <u>N/A</u>   | <u>Water</u> |                                        |                                                     |
| 2.     | MC-303H-Site 1-Field Lab Dup-P Ion Pair HPLC-ESMS - Surface water - Pensacola, FL |              |              |              |              |                                        |                                                     |
| 3.     | MC-304H-Site 1-Field Spike-PIU Ion Pair HPLC-ESMS - Surface water - Pensacola, FL |              |              |              |              |                                        |                                                     |
| 4.     | MC-305H-Site 1-Lab Spike-PIU Ion Pair HPLC-ESMS Surface water - Pensacola, FL     |              |              |              |              |                                        |                                                     |
| 5.     | MC-306H-Site 2-PIU Ion Pair HPLC-ESMS Surface water - Pensacola, FL               |              |              |              |              |                                        |                                                     |
| 6.     | MC-307H-Site 3-PIU Ion Pair HPLC-ESMS Surface water - Pensacola, FL               |              |              |              |              |                                        |                                                     |
| 7.     | MC-308H-Field Blank-PIU Ion Pair HPLC-ESMS Surface water - Pensacola, FL          |              |              |              |              |                                        |                                                     |
| 8.     | MC-304H-Quick Ion Pair HPLC-ESMS Surface water - Pensacola, FL                    |              |              |              |              |                                        |                                                     |
| 9.     | <u>KTD 5/2/00</u>                                                                 |              |              |              |              |                                        |                                                     |
| 10.    |                                                                                   |              |              |              |              |                                        |                                                     |

|                  |                                |                             |       |        |                            |                         |       |        |
|------------------|--------------------------------|-----------------------------|-------|--------|----------------------------|-------------------------|-------|--------|
| Chain of Custody | Collected by (print): Battelle |                             |       |        | Collector's signature: N/A |                         |       |        |
|                  | Item #                         | Relinquished by/Affiliation | Time  | Date   | Shipped Via:               | Received by/Affiliation | Time  | Date   |
|                  | 1-8                            | Kelly Dowdler/3M            | 14:00 | 5/2/00 | Fed Ex                     | A. Keller               | 10:30 | 5-3-00 |
|                  |                                |                             |       |        |                            |                         |       |        |
|                  |                                |                             |       |        |                            |                         |       |        |

Sample Condition Upon Receipt: ☒ Acceptable ☐ Other:

Temperature: 1°C ☒ Received on Ice

Other Associated CoCs: 14265, 14266, 14267, 14269, 14270

Copies to: KJH, LAC, DMA

Comments: