



Centre Analytical Laboratories, Inc.

3048 Research Drive
Phone: (814) 231-8032

State College, PA 16801
Fax: (814) 231-1253 or (814) 231-1580

AR226-0066

EM-4

Analytical Report

Fluorochemical Characterization of Drinking Water Samples

Pensacola, FI (W2176)

Centre Analytical Laboratory Report No. 023-007D

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 drinking 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 023-007.

Specific fluorochemical characterization by liquid chromatography / tandem mass spectrometry (LC/MS/MS) was requested for all samples. A total of 16 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

Data generated in support of this study was generated according to USEPA Good Laboratory Practices (GLPs). 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 02/15/00. The sample collection dates were not supplied. Chain-of-custody information is presented in Attachment A.

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.

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 μ 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 C₁₈ 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 2/22/00 and analyzed by MS/MS on 2/23/00. 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 sample at a concentration of 100 ng/L using all compounds of interest. Matrix spike recoveries are given in Attachment C.

Field spikes were also prepared on several samples at a concentration of 100 ng/L using all compounds of interest. Field spike recoveries are also given in Attachment C. The field spike results for sample MC-329H gave recoveries for all compounds of approximately 1000 %, indicating that this sample was possibly spiked at 1000 ppt instead of 100 ppt.

5.5 Duplicates

All samples were analyzed in duplicate. Results are given along with the sample results in Attachment B.

5.6 Laboratory Control Samples

Milliq water was spiked with all compound of interest at 25 and 250 ng/L. Initial analysis of the 25 ng/L LCS showed low recovery for PFOS. The standard was reinjected and was found to have acceptable recovery (70-130%). All other recoveries for all compounds were between 70-130% in each LCS.

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 B for a detailed listing of the analytical results.

7 Data/Sample Retention

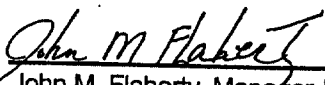
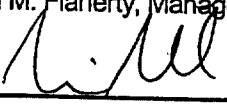
Samples are disposed of one month after the report is issued unless otherwise specified. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by Centre.

000325

8 Attachments

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

9 Signatures

 _____ John M. Flaherty, Manager-Industrial Services Group	<u>2/6/00</u> _____ Date
 _____ Kevin J Lloyd, Vice President	<u>28-Feb-00</u> _____ Date

Other Lab Members Contributing to Data

Enaksha Wickremesinhe
Karen Smith

3M Environmental Laboratory

Form 38778 - PWO

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

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

Chain of Custody / Requisition for Laboratory Analytical

17125

3M Env. Project #
For Internal Use Only

Project ID/Project Name 023-007 Multi-City Tap Drinking Water Analysis
Template #
Project Lead Kris Hansen
Dept. # (main) 4291
Final Report Due Date
Internal Due Date
Class/Job/Project # 0010535009

Report Results to:	Contact Name <u>Kris Hansen</u>	Date Available																
	Company <u>3M</u>	Date Due																
	Mailing Address <u>Building 2-3E-09 PO Box 33331</u>	Contract Lab																
	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.)

GEN-017
W2176
Pensacola, FL

Preservatives:					Total Number of Containers	Analysis Requested:													
HNO ₃	H ₂ SO ₄	VOCs	None	Other		Complete below. Attach any associated information.													
Enter the number of containers of each						(Enter an 'X' in the box below to indicate request)													

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/Media
1.	ML-315H-Intake-PIN	W2176	N/A	N/A	Water
2.	ML-317H-Intake-Field/Lab-Dup-P				
3.	ML-318H-Intake-Field Spike-PIN				
4.	ML-319H-Intake-Lab Spike-PIN				
5.	ML-320H-Intake-Field Blank-PIN				
6.	ML-321H-Outflow-PIN				
7.	ML-323H-Outflow-Field/Lab-Dup-P				
8.	ML-324H-Outflow-Field Spike-PIN				
9.	ML-325H-Outflow-Lab Spike-PIN				
10.	ML-326H-Site 1-PIN				

Chain of Custody	Collected by (print): <u>N/A</u>				Collector's signature: <u>N/A</u>			
	Item #	Relinquished by/Affiliation	Time	Date	Shipped Via:	Received by/Affiliation	Time	Date
	1-10	<u>Kelly Donawick</u>	-	<u>2/14/00</u>	<u>Fed Ex</u>	<u>[Signature]</u>	<u>1030</u>	<u>2-15-00</u>

Sample Condition Upon Receipt: ☒ Acceptable ☐ Other:

Temperature: 'C ☒ Received on Ice

Other Associated CoCs: 17120, 17121, 17122, 17123, 17127, 17128, 17124, 17130, 17131, 17133, 17134

Copies to:

KJH, LAC, JF

Comments:

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 /Reque. or Laboratory Analytical

17127

3M Env. Project #
For Internal Use Only

Project ID/Project Name	023-007 multi-City Tap & Drinking water Analysis
Template #	Final Report Due Date
Project Lead	Internal Due Date
Dept. # (main)	Class/Job/Project # 0010535009

Report Results to:	Contact Name	Kris Hansen
	Company	3M
	Mailing Address	Building 2-3E-09 PO BOX 33331
	City, State, Zip	St. Paul, MN 55331
	Telephone #	(651) 728-6018
	FAX #	(651) 728-6018

Date Available

Date Due

Contract Lab

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

GEN-017

W2176

Pensacola, FL

Preservatives:

Analysis Requested:

Complete below. Attach any associated information.

Final Number of Containers

 HNO_3

H₂SO₄

vocs

None

Other

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/Media
1.	ML-328H-Site1-Field/Lab Dup-P	W276	N/A	N/A	Water
2.	ML-329H-Site1-Field Spike-P/N	↓	↓	↓	↓
3.	ML-330H-Site1-Lab Spike-P/N	↓	↓	↓	↓
4.	ML-331H-Site2-P/N	↓	↓	↓	↓
5.	ML-332H-Site3-P/N	↓	↓	↓	↓
6.	ML-333H-Field Blank-P/N	↓	↓	↓	↓
7.					
8.	KTD 2/14/20	KTD 2/14/20	KTD 2/14/20	KTD 2/14/20	KTD 2/14/20
9.					
10.					

Enter the number of containers of each

(Enter an 'X' in the box below to indicate request)

Collector's signature: N/A

Chain of Custody	Collected by (print):		Time	Date
	Item #	Relinquished by/Affiliation		
	1-6	Kelly Donwiler 13M	-	2/14/00

Shipped Via:	Received by/Affiliation	Time	Date
FedEx		1930 2-15-00	2-15-00

Sample Condition Upon Receipt: ☒ Acceptable ☐ Other:

Temperature: 'C ● Received on Ice

Other Associated CoCs:	17120, 17121, 17122, 17123, 17125	Copies to:
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17128, 17129, 17130, 17131, 17133, 17134

Copies to:

KJH, LAC, JF

Comments:

000328



Centre Analytical Laboratories, Inc.

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

Analytical Results W2171 Pensacola, FI

3M Sample Identification	Sample Description	PFOS (ng/L)	PFOSA (ng/L)	POAA (ng/L)
MC-315H	Intake-P/N	< 25	< 25	< 25
MC-317H	Intake-P/N duplicate	< 25	< 25	< 25
MC-320H	Intake-Field Blank Empty	< 25	< 25	< 25
MC-321H	Outflow-P/N	< 25	< 25	< 25
MC-323H	Outflow-P/N duplicate	< 25	< 25	< 25
MC-326H	Site 1 P/N	< 25	< 25	< 25
MC-328H	Site 1 P/N duplicate	< 25	< 25	< 25
MC-331H	Site 2 P/N	< 25	< 25	< 25
NA	Site 2 P/N duplicate	< 25	< 25	< 25
MC-332H	Site 3 P/N	46.6	< 25	< 25
NA	Site 3 P/N duplicate	42.3	< 25	< 25
MC-333H	Field Blank-P/N Empty	< 25	< 25	< 25

000329

