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Analytical Report

Fluorochemical Characterization of Drinking Water Samples

Port St. Lucie, FL (W2363)

Centre Analytical Laboratory Report No. 023-007E (Revision 1)

Revision Date 3/20/01

Testing Laboratory

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3M Environmental Laboratory Contact

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Requester

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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 Port St. Lucie, 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

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 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 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 2/25/00 and analyzed by MS/MS on 2/26/00. 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 B.

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 B.

5.5 Duplicates

All samples 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.

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.

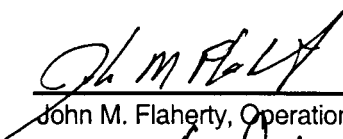
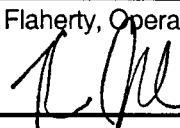
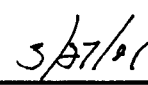
7 Data/Sample Retention

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	 _____ Kevin J Lloyd, Vice President	 _____ Date	 _____ Date
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Other Lab Members Contributing to Data

Enaksha Wickremesinha
Karen Smith



Centre Analytical Laboratories, Inc.

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

Analytical Results W2363 Port St. Lucie, FI

3M Sample Identification	Sample Description	PFOS (ng/L)	PFOSA (ng/L)	POAA (ng/L)
MC-615H	Intake-P/N	ND	ND	ND
MC-617H	Intake-P/N duplicate	ND	ND	ND
MC-620H	Intake-Field Blank Empty	ND	ND	ND
MC-621H	Outflow-P/N	ND	ND	ND
MC-623H	Outflow-P/N duplicate	ND	ND	ND
MC-626H	Site 1 P/N	ND	ND	ND
MC-628H	Site 1 P/N duplicate	ND	ND	ND
MC-631H	Site 2 P/N	ND	ND	ND
NA	Site 2 P/N duplicate	ND	ND	ND
MC-632H	Site 3 P/N	ND	ND	ND
NA	Site 3 P/N duplicate	ND	ND	ND
MC-633H	Field Blank-P/N Empty	ND	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 Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	87.1	87.1	PASS
PFOSA	0	90.4	90.4	PASS
POAA	0	96.2	96.2	PASS

Lower Recovery Limit:

Upper Recovery Limit:

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID:

Spiked Amount (ng/L):

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	83.6	83.6	PASS
PFOSA	0	89.5	89.5	PASS
POAA	0	93.2	93.2	PASS

Lower Recovery Limit:

Upper Recovery Limit:

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID:

Spiked Amount (ng/L):

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	87.4	87.4	PASS
PFOSA	0	81.0	81.0	PASS
POAA	0	98.3	98.3	PASS

Lower Recovery Limit:

Upper Recovery Limit:

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: MC-631H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	95.0	95.0	PASS
PFOSA	0	87.0	87.0	PASS
POAA	0	104	104.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID:

Spiked Amount (ng/L):

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	94.0	94.0	PASS
PFOSA	0	97.1	97.1	PASS
POAA	0	95.8	95.8	PASS

Lower Recovery Limit:

Upper Recovery Limit:

Attachment B: LC/MS/MS Field Spike Recovery

Sample ID: MC-618H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	125	125.0	PASS
PFOSA	0	128	128.0	PASS
POAA	0	125	125.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Attachment B: LC/MS/MS Field Spike Recovery

Sample ID: MC-624H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	128	128.0	PASS
PFOSA	0	109	109.0	PASS
POAA	0	126	126.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Attachment B: LC/MS/MS Field Spike Recovery

Sample ID: MC-629H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	130	130.0	PASS
PFOSA	0	107	107.0	PASS
POAA	0	126	126.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

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

Project ID/Project Name 023-017 MultiCity Tap Drinking Water Analysis

Template # _____ Final Report Due Date _____

Project Lead Kris Hansen Internal Due Date _____

Dept. # (main) 4291 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-019
W2363
Port St. Lucie, FL

Preservatives:					Total Number of Containers	Analysis Requested:														
HNO ₃	H ₂ SO ₄	VOCs	None	Other		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					Total Number of Containers	(Enter an 'X' in the box below to indicate request)									
						HNO ₃	H ₂ SO ₄	VOCs	None	Other											
1.	ML-605H-Intake-PIN	W2363	N/A	N/A	Water						1										
2.	ML-607H-Intake-Field/Lab										1										
3.	ML-618H-Intake-Field Spike-PIN										1										
4.	ML-619H-Intake-Lab Spike-PIN										1										
5.	ML-620H-Intake-Field Blank-PIN/empty										1										
6.	ML-621-Outflow-PIN										1										
7.	ML-623H-Outflow-Field/Lab Sp-P										1										
8.	ML-624H-Outflow-Field Spike-PIN										1										
9.	ML-625H-Outflow-Lab Spike-PIN										1										
10.	ML-626H-Site 1-PIN										1										

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>Veely Downer / 3M</u>	<u>-</u>	<u>2/14/02</u>		<u>[Signature]</u>	<u>1030</u>	<u>2-15-02</u>

Sample Condition Upon Receipt: ☒ Acceptable ☐ Other:	Comments:
Temperature: _____ °C ☒ Received on Ice	
Other Associated CoCs: <u>17120, 17121, 17122, 17123, 17125</u> <u>17127, 17128, 17129, 17130, 17131, 17134</u>	

Copies to: KJH, LAC, JF

Form 3877B - 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

Project ID/Project Name 023-007 multi-city Tap & Drinking Water Analysis

Template # _____ Final Report Due Date _____

Project Lead Kris Hansen Internal Due Date _____

Dept. # (main) 4291 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-019
W2363
Port St. Lucie, FL

Preservatives:

HNO3	H2SO4	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-628H-Site 1-Field/Lab Dup-P	W2363	N/A	N/A	Water															
2.	MC-629H-Site 1-Field Spike-PIN																			
3.	MC-630H-Site 1 Lab Spike-PIN																			
4.	MC-631H-Site 2-PIN																			
5.	MC-632H-Site 3-PIN																			
6.	MC-633H-Field blank-PIN empty																			
7.																				
8.																				
9.																				
10.																				

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
	1-6	Kelly Donwiler / 3M	-	2/14/00			1030	2-15-00

Sample Condition Upon Receipt: ☒ Acceptable ☐ Other:	Comments:
Temperature: 'C ☒ Received on Ice	
Other Associated CoCs: 17120, 17121, 17122, 17123, 17125 17127, 17128, 17129, 17130, 17131, 17133	

Copies to: KJH, LAG, JF