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Analytical Report

Fluorochemical Characterization of Surface Water Samples

Decatur, Alabama (W1979)

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

Revision Date 3/21/01

Testing Laboratory

Centre Analytical Laboratory, Inc.
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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 surface water samples received by Centre Analytical Laboratories, Inc. (Centre) from the 3M Environmental Laboratory. The samples were collected from Decatur, Alabama. 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 9 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. Nine individual sample containers were received. Sample MC-202H was not analyzed as per client request. (E-mail communication from Kris Hansen on 5/16/00). 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 uL 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_{18} 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 5/16/00 and analyzed by MS/MS between 5/19/00 and 5/20/00. 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 +/-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-201H at a concentration of 100 ng/L using all compounds of interest. The field spike is identified as MC-204H. Field spike recoveries are also given in Attachment B. PFOS showed high recovery in the field spike. All other compounds showed recoveries 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, Manager-Operations Manager — Date 3/27/01

Kevin J Lloyd, Vice President — Date 27 MARCH 2001

Other Lab Members Contributing to Data

Enaksha Wickremesinhe
Karen Smith
David Bell

ANALYTICAL REPORT

Centre Analytical Laboratories, Inc.
3048 Research Drive, State College PA 16801 814-231-8032 FAX 814-231-1253

Analytical Results W1979 Decatur, Alabama

3M Sample Identification	Sample Description	PFOS (ng/L)	PFOSA (ng/L)	POAA (ng/L)
MC-201H	Site 1 P/N Surface Water	NQ	ND	NQ
MC-203H	Site 1 P/N Surface Water Duplicate	NQ	ND	ND
MC-206H	Site 2 P/N Surface Water	NQ	ND	ND
NA	Site 2 P/N Surface Water Duplicate	NQ	ND	NQ
MC-207H	Site 3 P/N Surface Water	ND	ND	ND
NA	Site 3 P/N Surface Water Duplicate	ND	ND	ND
MC-208H	Field Blank P/N Empty	ND	ND	ND
MC-284H	Quiet P/N Surface Water	108	NQ	63.1
NA	Quiet P/N Surface Water Duplicate	114	NQ	57.3

Limit of Detection (LOD) for the procedure is appoximately 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: MC-205H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	23.1	128	104.9	PASS
PFOSA	0	107	107.0	PASS
POAA	9.2	106	96.8	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.

Also note that sample MC205-H is a laboratory spike of sample MC-201H

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: MC-206H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	14.5	117	102.5	PASS
PFOSA	0	103	103.0	PASS
POAA	0	113	113.0	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 Laboratory Spike Recovery

Sample ID: MC-207H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	0	100	100.0	PASS
PFOSA	0	101	101.0	PASS
POAA	0	105	105.0	PASS

Lower Recovery Limit: 70

Upper Recovery Limit: 130

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: MC-284H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	108	200	92.0	PASS
PFOSA	18.9	110	91.1	PASS
POAA	63.1	161	97.9	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: MC-204H

Spiked Amount (ng/L): 100

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	23.1	169	145.9	FAIL
PFOSA	0	85.6	85.6	PASS
POAA	9.2	111	101.8	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.

Also note that sample MC-204H is a field spike of sample MC-201H

BACK TO MAIN

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 14266

Project ID/Project Name	BEN014/Water Supply Study at Decatur, AL Sent to Battelle
Template #	Final Report Due Date
Project Lead KJH	Internal Due Date
Dept. # (main) 4291	Class/Job/Project # 00105350009

3M Env. Lab Project # For Internal Use Only: W1979

Report Results to:

Contact Name	Kris Hansen
Company	3M
Mailing Address	Building 2-3E-09 PO Box 33331
City, State, Zip	St. Paul, MN 55133
Telephone #	(651) 778-6018 FAX # (651) 778-6176

Date Available	05/03/00
Date Due	
Contract Lab	CENTRE

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

Preservatives: HNO_3 | H_2SO_4 | VOCs | None | Other

Total Number of Containers

Analysis Requested:
Complete below. Attach any associated information.

KJD 5/2/00

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)
1.	MC-201H-Site1-PIN Ion Pair HPLC-ESMS-Surface Water, Decatur, AL	W1979	NA	N/A	Water		1	
2.	MC-202H-Site1-Total DFOS Ion Pair HPLC-ESMS Surface Water - Decatur, AL						1	
3.	MC-203H-Site1-Field Lab Dup-P Ion Pair HPLC-ESMS-Surface Water-Decatur, AL						1	
4.	MC-204H-Site1-Field Spike-PIN Ion Pair HPLC-ESMS-Surface Water - Decatur, AL						1	
5.	MC-205H-Site1-Lab Spike-PIN Ion Pair HPLC-ESMS-Surface Water-Decatur, AL						1	
6.	MC-206H-Site2-PIN Ion Pair HPLC-ESMS Surface Water - Decatur, AL						1	
7.	MC-207H-Site3-PIN Ion Pair HPLC-ESMS Surface Water - Decatur, AL						1	
8.	MC-208H-Field Blank-PIN-Empty Ion Pair HPLC-ESMS-Surface Water-Decatur, AL						1	
9.	MC-284H-Quiet-PIN Ion Pair HPLC-ESMS Surface Water - Decatur, AL						1	
10.							9	

KJD 5/2/00

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
1-9	Kelly Dawsilar / 3M	11:00	5/2/00	FedEx	R. Fellen	10:30	5-3-00

Sample Condition Upon Receipt: ✓ Acceptable ○ Other:

Temperature: °C ✓ Received on Ice

Other Associated CoCs: 14265, 14267, 14268, 14269, 14270

Copies to: KJH, LAL, DMA

Comments:

Original - Accompanying Samples | Last Page - Originator | See Reverse Side for Instructions