Fluorochemical Characterization of Drinking Water Samples. Cleveland, Tennessee (W1973)

Centre Analytical Laboratories, Inc.
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Analytical Report

Fluorochemical Characterization of Drinking Water Samples

Cleveland, Tennessee (W1973)

Centre Analytical Laboratory Report No. 023-007A

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
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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 Cleveland, Tennessee. 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 17 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. Seventeen 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.

Samples MC-127H and MC-134H were not analyzed as per client request. (Telephone call to Kris Hansen on 2/16/00).

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 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 2/17/00 and analyzed by MS/MS between 2/17/00 and 2/18/00. 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 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.

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.

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 — 2/28/00

John M. Flaherty, Manager-Industrial Services Group — Date

28-Feb-00

Kevin J Lloyd, Vice President — Date

Other Lab Members Contributing to Data

Enaksha Wickremesinhe
Karen Smith

3M Environmental Laboratory

Form 38778 - PWO

Chain of Custody /Reque. or Laboratory Analytical 17120

3M Env. Project # For Internal Use Only

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 #	CC105535009

Report Results to:

Contact Name: Kris Hansen
Company: 3M
Mailing Address: building 2-3E-09 PO Box 333331
City, State, Zip: St. Paul, MN 55133
Telephone #: (651) 778-6018 FAX #: (651) 778-6176

Date Available:
Date Due:
Contract Lab: CENTRE

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

GEN-013
W1973
Cleveland, TN

Preservatives: HNO_3 | H_2SO_4 | VOCs | None | Other

Total Number of Containers

Analysis Requested:
Complete below. Attach any associated information.
(Enter an 'X' in the box below to indicate request)

Enter the number of containers of each

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/ Media
1.	MC-115H-Intake-P/N	W1973	N/A	N/A	Water
2.	MC-117H-Intake-Field/Lab-Dup-P				
3.	MC-118H-Intake-Field Spike-P/N				
4.	MC-120H-Intake-Field Blank-P/N empty				
5.	MC-121H-Outflow-P/N				
6.	MC-123H-Outflow-Field/Lab-Dup-P				
7.	MC-124H-Outflow Field Spike-P/N				
8.	MC-125H-Outflow-Lab Spike-P/N				
9.	MC-126H-Site 1-P/N				
10.	MC-127H-Site 1-total PFOS				

STD 8/4/00

Chain of Custody

Collected by (print): N/A
Collector's signature: N/A

Item #	Relinquished by/Affiliation	Time	Date	Shipped Via:	Received by/Affiliation	Time	Date
1-10	Kelly Dowdicker	--	2/14/00	Fed Ex		1030	2-15-00

Sample Condition Upon Receipt: ● Acceptable O Other:
Temperature: °C ● Received on Ice
Other Associated CoCs: 17121, 17122, 17123, 17125, 17127, 17129, 17130, 17131, 17133, 17134
Copies to: KJH, LAC, JF

Comments:

Original - Accompanying Samples

3M Environmental Laboratory

Form 38776 - PWO

Chain of Custody /Reque. or Laboratory Analytical 17121

3M Env Project # For Internal Use Only

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) 41291
Class/Job/Project # 00105350009

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
Date Due
Contract Lab CENTREC

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

GEN-013
W1973
Cleveland, TN

Preservatives:

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/ Media	HNO_3	H_2SO_4	VOCs	None	Other	Total Number of Containers
	Enter the number of containers of each										
1.	MC-128H-Site1-Field/Lab Dup-P	W1973	N/A	N/A	Water				1		1
2.	MC-129H-Site1-Field Spike-P/N								1		1
3.	MC-130H-Site1-Lab Spike-P/N								1		1
4.	MC-131H-Site2-P/N								1		2
5.	MC-132H-Site3-P/N								1		2
6.	MC-133H-Field Blank-P/N-Empty								1		1
7.	MC-134H-Field Blank-Total PFOS-Empty								1		1
8.											
9.											
10.											

Analysis Requested:
Complete below. Attach any associated information.
(Enter an 'X' in the box below to indicate request)

Collected by (print): N/A
Collector's signature: N/A

Chain of Custody

Item #	Relinquished by/Affiliation	Time	Date	Shipped Via:	Received by/Affiliation	Time	Date
1-7	Kelly Donwerth/3M	—	2/14/00	FedEx		1030	2-15-00

Sample Condition Upon Receipt: ● Acceptable O Other:
Temperature: °C ● Received on Ice
Other Associated CoCs: 17120, 17122, 17123, 17125, 17127, 17128, 17129, 17130, 17131, 17133, 17134
Copies to: KJH, LAC, JF

Comments:

Original - Accompanying Samples Last Page - Originator

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

Analytical Results W1973 Cleveland, Tennesse

3M Sample Identification	Sample Description	PFOS (ng/L)	PFOSA (ng/L)	POAA (ng/L)
MC-115H	Intake-P/N	< 25	< 25	< 25
MC-117H	Intake-P/N duplicate	< 25	< 25	< 25
MC-120H	Intake-Field Blank	< 25	< 25	< 25
MC-121H	Outflow-P/N	< 25	< 25	< 25
MC-123H	Outflow-P/N duplicate	< 25	< 25	< 25
MC-126H	Site 1 P/N	< 25	< 25	< 25
MC-128H	Site 1 P/N duplicate	< 25	< 25	< 25
MC-131H	Site 2 P/N	< 25	< 25	< 25
NA	Site 2 P/N duplicate	< 25	< 25	< 25
MC-132H	Site 3 P/N	< 25	< 25	< 25
NA	Site 3 P/N duplicate	< 25	< 25	< 25
MC-133H	Field Blank P/N Empty	< 25	< 25	< 25