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Analytical Report

Fluorochemical Characterization of Sediment, POTW Sludge, and

Landfill Leachate Samples

Decatur, AL (W1979)

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

Revision Date 3/28/01

Testing Laboratory

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1 Introduction

Results are reported for the analysis of a series of sediment, leachate, and sludge samples received by Centre Analytical Laboratories, Inc. (Centre) from the 3M Environmental Laboratory. The samples were collected from Decatur, Alabama and are part of 3M Project W1979. 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 Sulfonamide	PFOSA
Perfluorooctanoate	POAA

The analytical methods used for originally developed for water samples and were validated by Centre. The water sample validation protocol and results are on file with Centre. The methods were modified for the sediment and sludge samples. The procedures have not been fully validated for these matrices. 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. Seven individual sample containers were received. Samples were received on 7/28/00. 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 in water samples. Stability after this time period has not been validated. 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

Water samples (leachate and field blanks) 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.

For the sediment and sludge samples, a representative portion of sample (5 grams) was first extracted into 5 mL of methanol. The extracts were filtered and diluted to a final volume of 40 mL with Type I water. The diluted extracts were then treated in the same manner as the water samples, beginning with the solid phase extraction.

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 between 8/21/00 and 8/29/00 and analyzed by MS/MS between 8/24/00 and 8/30/00. The HPLC and MS/MS methods used for analysis and instrument parameters can be found in Attachments 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) for LC/MS/MS analysis. The instrument response 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 methods.

5.4 Matrix Spikes

Matrix spikes were prepared for every field sample using all compounds of interest. Matrix spike recoveries are given in Attachment B. No field spikes were made on these samples

5.5 Duplicates

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

5.6 Laboratory Control Samples

For LC/MS/MS analyses, MilliQ water was spiked with all compounds of interest at 25 and 250 ng/L during each extraction set. All recoveries for all compounds were between 70-130% in each LCS. Results are given along with the raw data in Attachments D.

5.7 Sample Related Comments

Empty containers were submitted as filed blanks. The containers were treated with 40 mL of MilliQ water and analyzed as per the method. There are no other sample related comments for this data set.

6 Data Summary

Please see Attachment A for a detailed listing of the analytical results. Field blank water and leachate results are reported in parts per trillion (ppt) (ng/L). Sediment and sludge sample results are reported in parts per billion (ppb) (ug/Kg) on both an as-received and dry-weight basis.

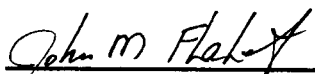
7 Data/Sample Retention

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
- 8.3 Attachment C: Chain of Custody
- 8.4 Attachment D: LC/MS/MS Raw Analytical Data

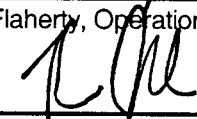
9 Signatures



John M. Flaherty, Operations Manager

3/28/01

Date



Kevin J Lloyd, Vice President

28 MARCH 2001

Date

Other Lab Members Contributing to Data

Karen Smith



Centre Analytical Laboratories, Inc.

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

Analytical Results W1974 Decatur, Alabama (GEN 014)

3M Sample Identification	PFOS (ng/L)	PFOSA (ng/L)	POAA (ng/L)
MC-213K Field Blank	ND	ND	ND
MC-214K Field Blank	ND	ND	ND
MC-235K Landfill Leachate	53100	258	48100
MC-235K Landfill Leachate dup	52300	249	46800
MC-236K Field Blank	ND	ND	ND
MC-245K Field Blank	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





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Analytical Results W1974 Decatur, Alabama (GEN 014)

3M Sample Identification	PFOS (ug/Kg) (as received)	PFOSA (ug/Kg) (as received)	POAA (ug/Kg) (as received)	% Solids
MC-210K Sediment Site 1	NQ	ND	ND	76.06
MC-210K Sediment Site 1 dup	NQ	NQ	NQ	76.06
MC-211K Sediment Site 2	0.253	NQ	NQ	85.79
MC-211K Sediment Site 2 dup	0.673	NQ	NQ	85.79
MC-212K Sediment Site 3	0.292	NQ	NQ	81.23
MC-212K Sediment Site 3 dup	0.299	NQ	NQ	81.23
MC-244K POTW Sludge	99.8	3.76	3.55	3.52
MC-244K POTW Sludge dup	110	3.44	8.57	3.52

3M Sample Identification	PFOS (ug/Kg) (dry weight)	PFOSA (ug/Kg) (dry weight)	POAA (ug/Kg) (dry weight)
MC-210K Sediment Site 1	NQ	ND	ND
MC-210K Sediment Site 1 dup	NQ	NQ	NQ
MC-211K Sediment Site 2	0.295	NQ	NQ
MC-211K Sediment Site 2 dup	0.785	NQ	NQ
MC-212K Sediment Site 3	0.36	NQ	NQ
MC-212K Sediment Site 3 dup	0.368	NQ	NQ
MC-244K POTW Sludge	2840	107	101
MC-244K POTW Sludge dup	3120	97.8	244

Limit of Detection (LOD) for the procedure is approximately 0.08 ug/Kg for all compounds (as received)

Limit of Quantitation (LOQ) for the procedure is 0.20 ug/Kg for all compounds (as received)

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-210K

Spiked Amount (ng/L): 250

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	18.2	221	81.1	PASS
PFOSA	0	213	85.2	PASS
POAA	0	218	87.2	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.

Concentrations represent amounts present in the sample extract

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: MC-211K

Spiked Amount (ng/L): 250

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	31.6	242	84.2	PASS
PFOSA	4.5	214	83.8	PASS
POAA	9.0	230	88.4	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.

Concentrations represent amounts present in the sample extract

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: MC-212K

Spiked Amount (ng/L): 250

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	36.5	216	71.8	PASS
PFOSA	4.0	203	79.6	PASS
POAA	16.1	218	80.8	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.

Concentrations represent amounts present in the sample extract

Attachment B: LC/MS/MS Laboratory Spike Recovery

Sample ID: MC-244K

Spiked Amount (ng/L): 250

	Sample Concentration (ng/L)	Matrix Spike Result (ng/L)	Matrix Spike Result (% Recovery)	Criteria (Pass / Fail)
PFOS	12500	7920	-1832.0	FAIL
PFOSA	470	362	-43.2	FAIL
POAA	444	446	0.8	FAIL

Lower Recovery Limit: 70

Upper Recovery Limit: 130

PFOS sample concentration exceeds the spiking amount by greater than 10X

Concentrations represent amounts present in the sample extract

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	53100	47000	-6100.0	FAIL
PFOSA	258	307	49.0	FAIL
POAA	48100	47300	-800.0	FAIL

Lower Recovery Limit:

Upper Recovery Limit:

PFOS and POAA sample concentration exceeds the spiking amount by greater than 10X

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

15093

3M Env. Lab Project #
For Internal Use Only

Project ID/Project Name	Gen-014 Decatur, AL
Template #	N/A
Project Lead	Kris J. Hansen
Dept. # (main)	4291
Final Report Due Date	
Internal Due Date	
Class/Job/Project #	0010525009

W1979

Report Results to:	Contact Name	USA Clemens	Date Available	072800
	Company	3M	Date Due	
	Mailing Address	Bldg. 2-3E-09 935 Bush Ave.	Contract Lab	Centre
	City, State, Zip	St. Paul, Minnesota		
	Telephone #	651 778 9568	FAX #	

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

Preservatives:

Analysis Requested:

Complete below. Attach any associated information.

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/Media	Preservatives:					Total Number of Containers	Analysis Requested:		
						HNO3	H2SO4	VOCs	None	Other		PFO5	PFO3A	PDA (PFOA)
1.	MC-210K-Site 1 - P/N/Total PFO5/LC Ion Pair HPLC-ESMS Sediment - Decatur, AL				Sediment						1	X	X	X
2.	MC-211K-Site 2 - P/N/Total PFO5/LC Ion Pair HPLC-ESMS Sediment - Decatur, AL										1	X	X	X
3.	MC-212K-Site 3 - P/N/Total PFO5/LC Ion Pair HPLC-ESMS Sediment - Decatur, AL										1	X	X	X
4.	MC-213K-Field Blank - P/N-Empty Ion Pair HPLC-ESMS Sediment - Decatur, AL										1	X	X	X
5.	MC-214K-Field Blank - Total PFO5-Empty Ion Pair HPLC-ESMS Sediment - Decatur, AL										1	X	X	X
6.	MC-235K-P/N/Total PFO5/LC Ion Pair HPLC-ESMS Landfill Leachate - Decatur, AL		07/27/00		Leachate						1	X	X	X
7.	MC-236K-Field Blank - P/N-Empty Ion Pair HPLC-ESMS Landfill Leachate - Decatur, AL										1	X	X	X
8.	MC-244-P/N/Total PFO5/LC Ion Pair HPLC-ESMS POTW Sludge - Decatur, AL				Sludge						1	X	X	X
9.	MC-245K-Field Blank - P/N-Empty Ion Pair HPLC-ESMS POTW Sludge - Decatur, AL										1	X	X	X
10.														

Chain of Custody	Collected by (print):	Sally Linda	Collector's signature:	S. Linda
	Item #	Relinquished by/Affiliation	Time	Date
	1-9	Alex A. Clemens / 3M		7/27/00
	Shipped Via:	Received by/Affiliation	Time	Date
		R. Keller	1100	7-28-00

Sample Condition Upon Receipt:	☒ Acceptable	☐ Other:	Comments:
Temperature:	°C	☒ Received on Ice	
Other Associated CoCs:	N/A	Copies to:	
		KJH, LAC	