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Analytical Report

3M Company

Analysis of PFOA and PFOS In Water Samples from Oakdale Municipality

E05-0243

Exygen Report No. L0004933

Testing Laboratory

Exygen Research
3058 Research Drive
State College, PA 16801

Requester

Gary A. Hohenstein
Manager, Environmental Operations
EHS Operations
3M Company
Building 42-2E-27
St. Paul, MN 55106

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1 Introduction

Results are reported for the analysis of perfluorooctanoate (PFOA) and perfluorooctanesulfonate (PFOS) in water samples received at Exygen as requested by Gary Hohenstein of 3M Company. The Exygen project number assigned to the samples is L0004933. Table I lists the target analytes quantitated for the samples.

Table I. Target Analytes for Quantitation

<u>Parameter</u>	<u>Acronym</u>	<u>Formula</u>
Perfluorooctanoate	PFOA or C8 acid	$C_7F_{15}COO^-$
Perfluorooctanesulfonate	PFOS or C8 sulfonate	$C_8F_{17}SO_3^-$

2 Sample Receipt

Twenty-one samples were received at Exygen in 500 mL white plastic narrow-mouth bottles. At the request of 3M, all samples were analyzed. A copy of all sample log-in information is presented in Attachment A.

The samples were received on 03/30/05. The samples were shipped ambient via UPS. The samples were stored refrigerated from time of receipt until analysis.

3 Methods - Analytical and Preparatory

The Exygen method V0001780 was used to prepare and analyze the samples. A summary of the sample preparation can be found in Section 3.1 and a summary of the analysis conditions can be found in Section 3.2.

3.1 Water Sample Preparation

Solid phase extraction (SPE) was used to prepare the water samples for LC/MS/MS analysis. Forty milliliters of the water sample was transferred to a C_{18} SPE cartridge. The cartridge was eluted with 5 mL of 100% methanol. A portion of the extract was transferred to autosampler vials and analyzed using electrospray LC/MS/MS.

3.2 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (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, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against extracted standards.

An HP1100 system interfaced to an Applied Biosystems API 4000 was used to analyze the sample extracts for quantitation.

Isomer separation was obtained using a gradient elution through a Thermo Fluophase RP, 60 x 2.1 mm x 5µm column. The sum of the isomers is reported as a single value for each of the analytes.

The following gradient was performed:

Mobile Phase (A): 2mM Ammonium Acetate in Water
Mobile Phase (B): Methanol

Time	%A	%B
0.0	65	35
1.0	65	35
8.0	25	75
10.0	25	75
11.0	65	35
18.0	65	35

The following parameters were used for operation of the mass spectrometer:

Parameter	Setting
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	413->369 (PFOA), 499->80 (PFOS), 415->370 (¹³ C-PFOA, surrogate spike)
Gas Temperature	350°C
Drying Gas (N2)	7.0 L/min

4 Analysis

4.1 Calibration

A 7-point calibration curve was analyzed initially and throughout the analytical sequence for PFOA, PFOS and ¹³C PFOA. The calibration points were prepared at 0, 25, 50, 100, 250, 500, and 1000 ng/L (ppt) for LC/MS/MS analysis. Calibration standards are prepared using the same SPE procedure used for samples. The instrument response versus the concentration was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept

and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r^2 \geq 0.985$.

For the results reported here, calibration criteria were met. The calibration curves are included in the raw data in Attachment C.

4.2 Surrogates

^{13}C PFOA ($\text{C}_8\text{F}_{15}[^{13}\text{C}]_2\text{F}_2\text{OOH}$) is used as a surrogate for the water samples. ^{13}C PFOA was added to the empty sample containers before the bottles were sent to the field for sampling. In the field, all sample and quality control bottles were filled to a volumetric fill line with the sample.

^{13}C PFOA recoveries can be found in Attachment B.

4.3 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared by adding a known concentration of PFOA, ^{13}C PFOA, and PFOS to laboratory reagents. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range.

4.4 Field Matrix Spikes

One matrix spike was prepared for every water sample in the field. Compounds were added to the empty sample containers before the bottles were sent to the field for sampling. Matrix spikes are used to assess method accuracy in the matrix. The matrix spikes should show recoveries between 70-130%. For the results reported here, the matrix spikes were within the acceptable range.

4.5 Sample Related Comments

All water samples were collected in duplicate. Duplicate sample results are reported along with the sample results in Attachment B.

5 Data Summary

Please see Attachment B for a detailed listing of the analytical results. The results are reported in parts per billion (ng/mL) for PFOA and PFOS.

6 Statement of Accuracy

Based on results of laboratory control spikes, field matrix spikes, and surrogate spikes, the analytical accuracy for PFOS and PFOA results for all samples is $\pm 30\%$. All laboratory control spikes, surrogate spikes and field matrix spikes showed recoveries within these ranges.

Specific sample recoveries are given in Attachment B.

7 Data/Sample Retention

Samples will be returned to 3M 60 days after final reporting. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by Exygen. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.

8 Attachments

- 8.1 Attachment A: Analytical Results
- 8.2 Attachment B: Spike Recovery Data
- 8.3 Attachment C: Chain of Custody Forms
- 8.4 Attachment D: Raw Analytical Data

9 Signatures



Karen Risha, Principal Investigator

04/12/05
Date



John M. Flaherty, Vice President

4/12/05
Date

Section A

Summary of PFOA and PFOS in Water Samples E05-0243

Oxygen ID	Sample ID	Analyte Found (ng/mL)	
		C8 Acid PFOA Perfluorooctanoate	C8 Sulfonate PFOS Perfluorooctanesulfonate
L4933-1	O-W5-127287	0.779	1.14
L4933-2	O-W5-127287 Dup	0.710	0.941
L4933-4	O-W1-208462	0.0887	0.0619
L4933-5	O-W1-208462 Dup	0.0939	0.0609
L4933-7	O-W2-208463	0.0823	NQ
L4933-8	O-W2-208463 Dup	0.0817	NQ
L4933-10	O-W8-572608	0.503	0.704
L4933-11	O-W8-572608 Dup	0.457	0.551
L4933-13	O-W9-151575	0.813	0.580
L4933-14	O-W9-151575 Dup	0.763	0.502
L4933-16	O-W3-208454	ND	ND
L4933-17	O-W3-208454 Dup	ND	ND
L4933-19	Field Blank	ND	ND

ND = Not detected at or above 0.025 ng/mL.

NQ = Not quantifiable = Measured concentration between 0.025 ng/mL and the Limit of Quantitation (LOQ) which is 0.050 ng/mL.

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Section B

Field Matrix Spike Recovery Summary for PFOA and PFOS In Water E05-0243

Sample Description	C8 Acid PFOA				C8 Sulfonate PFOS		
	Amount Spiked (ng/mL)	Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
O-W5-127287 Spike (L4933-3, 1 ppb Spike)	1	0.779	1.74	96	1.140	2.11	97
O-W1-208462 Spike (L4933-8, 0.1 ppb Spike)	0.1	0.0887	0.174	85	0.0619	0.134	72
O-W2-208463 Spike (L4933-9, 0.1 ppb Spike)	0.1	0.0823	0.178	96	0.0296	0.124	94
O-W8-572608 Spike (L4933-12, 0.5 ppb Spike)	0.5	0.503	1.05	109	0.704	1.26	111
O-W9-151575 Spike (L4933-15, 1 ppb Spike)	1	0.813	1.89	108	0.580	1.53	95
O-W3-208454 Spike (L4933-18, 0.1 ppb Spike)	0.1	ND	0.106	106	ND	0.111	111
Field Blank Low Spike (L4933-20, 0.1 ppb Spike)	0.1	ND	0.0987	99	ND	0.0983	98
Field Blank High Spike (L4933-21, 1 ppb Spike)	1	ND	0.953	95	ND	0.839	84

*Sample residue exceeds the spiking level significantly; therefore, an accurate recovery value cannot be calculated

ND = Not detected at or above 0.025 ng/mL.

NQ = Not quantifiable = Measured concentration between 0.025 ng/mL and the Limit of Quantitation (LOQ) which is 0.050 ng/mL.

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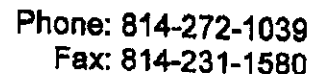
Surrogate Spike Recovery Summary for ¹³C-PFOA in Water E05-0243

Oxygen ID	Sample Description	¹³ C-PFOA		
		Amount Spiked (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
L4933-1	O-W5-127287	0.5	0.430	86
L4933-2	O-W5-127287 Dup	0.5	0.411	82
L4933-3	O-W5-127287 1.0 ppb Spk	1.0	1.03	103
L4933-4	O-W1-208462	0.5	0.460	92
L4933-5	O-W1-208462 Dup	0.5	0.494	99
L4933-6	O-W1-208462 0.1 ppb Spk	0.1	0.0966	97
L4933-7	O-W2-208463	0.5	0.436	87
L4933-8	O-W2-208463 Dup	0.5	0.422	84
L4933-9	O-W2-208463 0.1 ppb Spk	0.1	0.0991	99
L4933-10	O-W8-572608	0.5	0.424	85
L4933-11	O-W8-572608 Dup	0.5	0.416	83
L4933-12	O-W8-572608 0.5 ppb Spk	0.5	0.476	95
L4933-13	O-W9-151575	0.5	0.460	92
L4933-14	O-W9-151575 Dup	0.5	0.394	79
L4933-15	O-W9-151575 1.0 ppb Spk	1.0	1.02	102
L4933-16	O-W3-208454	0.5	0.450	90
L4933-17	O-W3-208454 Dup	0.5	0.437	87
L4933-18	O-W3-208454 0.1 ppb Spk	0.1	0.104	104
L4933-19	Field Blank	0.5	0.489	98
L4933-20	Field Blank 0.1 ppb Spk	0.1	0.0991	99
L4933-21	Field Blank 1.0 ppb Spk	1.0	0.964	96

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Section C



Login Group: L0004933

```
Conform COC Sample: True
Conform COC: True
Conform Sample: True
Conform Request: True
```

1. **THEORY OF THE CASE**

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Login

Login Group: L0004933

Login #:	5043	Conform COC Sample:	True
Project:	P0000990	Conform COC:	True
Company Name:	3M	Conform Sample:	True
Submitted By:	Kent Lindstrom	Conform Request:	True
Login Type:	Immediate Receipt of Samples		
Started:	True		
Date Start:	03/30/2005		
Due Date:	04/09/2005		
Received Date:	03/30/2005		
Received By:	Ammerman, Mark		
Spread Sample:			
Label:			
Oxygen SD/PI:	Risha, Karen		
Project Title/Type:	Analysis of Fluorochemicals by LCMSMS for NRG Sampling of Oakdale Site / ROUTINE		
Login Notes:			
Conform Notes:			

Packages / Containers

Package	Carton	Date / Condition	Shipper / ID	Temp. Control/Temp.	Direction / Handled By
PK0005816		Received Date: 3/30/2005 12:00:00AM Package & Contents Uncompromised	UPS 1257955E0141403628	None 23.2	RECEIVED Ammerman, Mark

Container #	Gross Weight	pH	Container Type	Preservative	Mfg. Lot	Mfg. ID
C0066687	248.70 g		500 ml Clear Plastic Narrow	NONE		
C0066688	267.80 g		500 ml Clear Plastic Narrow	NONE		
C0066689	243.50 g		500 ml Clear Plastic Narrow	NONE		
C0066690	252.80 g		500 ml Clear Plastic Narrow	NONE		
C0066691	255.30 g		500 ml Clear Plastic Narrow	NONE		
C0066692	257.20 g		500 ml Clear Plastic Narrow	NONE		
C0066693	257.50 g		500 ml Clear Plastic Narrow	NONE		

Login

Samples

Sample ID	Container	Matrix	Fraction	Sample	Date Sampled	Date Due
L0004933-0001	C0066673	LIQUID	Water	O-W5-127287	03/29/2005	04/09/2005
L0004933-0002	C0066674	LIQUID	Water	O-W5-127287 Dup	03/29/2005	04/09/2005
L0004933-0003	C0066675	LIQUID	Water	O-W5-127287 1.0 spike	03/29/2005	04/09/2005
L0004933-0004	C0066676	LIQUID	Water	O-W1-208462	03/29/2005	04/09/2005
L0004933-0005	C0066677	LIQUID	Water	O-W1-208462 Dup	03/29/2005	04/09/2005
L0004933-0006	C0066678	LIQUID	Water	O-W1-208462 0.1 spike	03/29/2005	04/09/2005
L0004933-0007	C0066679	LIQUID	Water	O-W2-208463	03/29/2005	04/09/2005
L0004933-0008	C0066680	LIQUID	Water	O-W2-208463 Dup	03/29/2005	04/09/2005
L0004933-0009	C0066681	LIQUID	Water	O-W2-208463 0.1 spike	03/29/2005	04/09/2005
L0004933-0010	C0066682	LIQUID	Water	O-W8-572608	03/29/2005	04/09/2005
L0004933-0011	C0066683	LIQUID	Water	O-W8-572608 Dup	03/29/2005	04/09/2005
L0004933-0012	C0066684	LIQUID	Water	O-W8-572608 0.5 ppb spike	03/29/2005	04/09/2005
L0004933-0013	C0066685	LIQUID	Water	O-W9-151575	03/29/2005	04/09/2005
L0004933-0014	C0066686	LIQUID	Water	O-W9-151575 Dup	03/29/2005	04/09/2005
L0004933-0015	C0066687	LIQUID	Water	O-W9-151575 1.0 ppb spike	03/29/2005	04/09/2005
L0004933-0016	C0066688	LIQUID	Water	O-W3-208454	03/29/2005	04/09/2005
L0004933-0017	C0066689	LIQUID	Water	O-W3-208454 Dup	03/29/2005	04/09/2005
L0004933-0018	C0066690	LIQUID	Water	O-W3-208454 0.1 ppb spike	03/29/2005	04/09/2005
L0004933-0019	C0066691	LIQUID	Water	Field Blank		04/09/2005
L0004933-0020	C0066692	LIQUID	Water	Field Blank 0.1 ppb spike		04/09/2005
L0004933-0021	C0066693	LIQUID	Water	Field Blank 1.0 ppb spike		04/09/2005

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Login

Login Reviewed By:

SLA

Date/Time:

3/30/2005 1735

3M Environmental Laboratory

Form 38778 - PWO

Shipping Address:

3M Bldg. 2-3E-09
935 Bush Avenue
St. Paul, MN 55105

Telephone:

Sample Receiving: (651) 778-4948
Attn: (651) 778-4753
FAX: (651) 778-5176

Chain of Custody / Request for Laboratory Analytical

08914

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

Project ID/Project Name: OKING Municipal Wells PC
Template #: Final Report Due Date
Project Lead: Kent Lindstrom
Dept. # (main): 530711
Class/Job/Project #: 0022634449

Internal Due Date
Contract Lab

EOS-0243

Contact Name: Gary Heberstein

Company: 3M

Mailing Address

City, State, Zip

Telephone #

FAX #

Date Available

Date Due

Contract Lab

3/30

4/20

EXP 3 en

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

Preservatives:

HNO₃

H₂SO₄

VOA

None

Other

Total Number of Containers

Analysis Requested:

Complete below. Attach any associated information.

See Attached Lab Request
EOS-0243

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

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/Media
1.	O-W5-127287	81112	3/29/05	0834	W
2.	O-W5-127287 Dup	81113	3/29/05	0836	
3.	O-W5-127287 1.0 spike	81114	3/29/05	0836	
4.	O-W1-208462	81103	3/29/05	0900	
5.	O-W1-208462 Dup	81104	3/29/05	0900	
6.	O-W1-208462 - 0.1 spike	81105	3/29/05	0900	
7.	O-W2-208463	81106	3/29/05	0915	
8.	O-W2-208463 Dup	81107	3/29/05	0915	
9.	O-W2-208463 0.1 spike	81108	3/29/05	0915	
10.	O-W8-572608	81118	3/29/05	0930	

Collected by (print): Brad Jacobson-Pace

Collector's signature: Brad Jacobson

Item #

Relinquished to/Affiliation

Time

Date

Shipped Via

Received by/Affiliation

Time

Date

1-10 Brad Jacobson

1121 3/29/05 Self

1600 3/29/05 UPS AIR

1600 3/29/05 1600 7/29/05

Sample Condition Upon Receipt: ☒ Acceptable ☐ Other: ICE NOT RETURNED

Temperature: 17 °C ☐ Received on ice

Other Associated Co's: 08915 08912

Copies to: KPL ✓

Comments:

ALL ITEMS PUT IN FRIDGE 12-1 W/IN
RECEIPT @ 3M ENV. LAB. (AE) PC 3/29/05

* TO 0246EN

3M Environmental Laboratory

Form 36778 - PWO

Shipping Address:
3M Bldg. 2-3E-09
333 Bush Avenue
St. Paul, MN 55105
Telephone: (612) 778-6848
Sample Receipts: (612) 778-6848
Altitude: (612) 778-6848
Fax: (612) 778-6178

Chain of Custody / Request for Laboratory Analytical 08915

3M Env. Lab Project #
For Internal Use Only

EOS-0243

Project ID/Project Name	DATEABLE Municipal	EC'S IV
Template #		
Project Lead	West Lindstrom	
Dept. # (main)	530711	
Class/Job/Project #	002267849	
Contract Name	Gary Holmstrom	
Company	3M	
Mailing Address		
City, State, Zip		
Telephone #		
FAX #		

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

Item #	Client Sample Identification	3M LIMS#	Date Sampled	Time Sampled	Matrix/Media	Preservatives:				Total Number of Containers	Analysts Requested:
						HNO3	H2SO4	VO3	None		
1.	Q-WB-57208 Dup	81117	3/29/05	0930	W					1	Complete below. Attach any associated information. See Attached Lab Request EOS-0243
2.	Q-WB-57208 0.5 ppb spike	81120		0930						1	
3.	Q-WB-151575	81121		1006						1	
4.	Q-WB-151575 Dup	81122		1006						1	
5.	Q-WB-151575 1.0 ppb spike	81123		1006						1	
6.	Q-WB-208454	81107		0950						1	
7.	Q-WB-208454 Dup	81110		0950						1	
8.	Q-WB-208454 0.1 ppb spike	81111		0950						1	
9.	Field Blank	81127	Expt	0950						1	
10.	Field blank 0.1 ppb spike	81128	Expt	0950						1	

Enter the number of bottles of each

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

Collector's signature: Brad Jacobson

Collected by (print): Brad Jacobson - Rec

Item #	Quantity	Date	Time	Shipping Via	Received by/initials	Date	Time
1-10	Brad Jacobson Rec	11/21	3:45 PM	DE HP	Brad Jacobson	11/20	3:27 PM
11-10	Brad Jacobson Rec	1/6/05	3:00 PM	UPS AIR			

Sample Condition Upon Receipt:	Acceptable	Other:	ICE NOT REQUIRED
Temperature: 121 °C		O Received on ice	
Other Associated Costs:		Expire to:	
08914 08912		KAL	

Comments: ① Exposed-filled Blanks and shipped w/ the bottle order. Saw 3/29/05

All items per in 12065 R-1 upon receipt @ 3M ENVIRO LAB - (AE) JLF 3/29/05

* TO

3/29/2005

Page 1 of 3

**3M ENVIRONMENTAL LABORATORY
CONTRACT LABORATORY WORK ORDER BY SAMPLE**

Project: E05-0243

Contract Lab(s): EXYGEN

Requester: Hohenstein, Gary A (0042-02E-27)

Completion Date:

Department: 530711

Project Lead: Kent Lindstrom

Project Number: 0022674449

Phone Number: 651-778-5352

Date Received: 3/28/2005

Email Address: krlindstrom@mmm.com

Project Description: Oakdale Municipal Well FC's IV

Ship Date:

3/29/05

Comments:

Pace field sampling.

3M Sample Number	Sampled Date	Sample Description	
E05-0243-81103	3/29/2005	O-W1-208462	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81104	3/29/2005	O-W1-208462 Dup	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81105	3/29/2005	O-W1-208462 0.1 PPB Spike	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81106	3/29/2005	O-W2-208463	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81107	3/29/2005	O-W2-208463 Dup	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81108	3/29/2005	O-W2-208463 0.1 PPB Spike	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005

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3/29/2005

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**3M ENVIRONMENTAL LABORATORY
CONTRACT LABORATORY WORK ORDER BY SAMPLE**

Project: E05-0243 (cont.)

Contract Lab(s): EXYGEN

3M Sample Number	Sampled Date	Sample Description	
E05-0243-81109	3/29/2005	O-W3-208454	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81110	3/29/2005	O-W3-208454 Dup	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81111	3/29/2005	O-W3-208454 0.1 PPB Spike	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81112	3/29/2005	O-W5-127287	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81113	3/29/2005	O-W5-127287 Dup	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81114	3/29/2005	O-W5-127287 1.0 PPB Spike	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81118	3/29/2005	O-W8-572608	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81119	3/29/2005	O-W8-572608 Dup	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81120	3/29/2005	O-W8-572608 0.5 PPB Spike	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005

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3/29/2005

Page 3 of 3

3M ENVIRONMENTAL LABORATORY
CONTRACT LABORATORY WORK ORDER BY SAMPLE

Project: E05-0243 (cont.)

Contract Lab(s): EXYGEN

3M Sample Number	Sampled Date	Sample Description	
E05-0243-81121	3/29/2005	O-W9-151575	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81122	3/29/2005	O-W9-151575 Dup	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81123	3/29/2005	O-W9-151575 1.0 PPB Spike	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81127	3/29/2005	Field Blank	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81128	3/29/2005	Field Blank 0.1 PPB Spike	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005
E05-0243-81129	3/29/2005	Field Blank 1.0 PPB Spike	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	4/20/2005
PFOS	PFOS by ESMS	PFOS	4/20/2005

0018

45

PAT FERRETTI
(651) 778-8753
3A ENVIRONMENTAL LAB
835 BUSH AVENUE
SAINT PAUL MN 55106

25 LBS

1 OF 1

SHIP TO:

JOHN FLAHERTY/SAMPLE CHECK-IN
(814) 231-8032

OXYGEN RESEARCH
117 RESEARCH DR

TATE COLLEGE PA 16801-2752



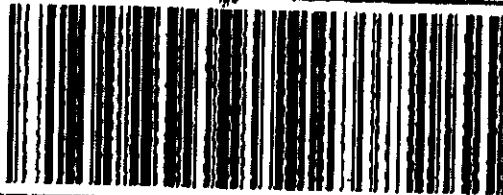
PA 168 0-10



UPS NEXT DAY AIR

TRACKING #: 1Z 579 55E 00 140 3528

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BILLING: P/P

Dept No.: 530711; 0622574448
REF 2: E05-0243

UOM 5.9.27 UPS Thermal 2 25.0A 01/2000



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Analytical Report

3M Company

Analysis of PFOA and PFOS In Water Samples from Hastings Municipality

E05-0186

Oxygen Report No. L0004709

Testing Laboratory

Oxygen Research
3058 Research Drive
State College, PA 16801

Requester

Gary A. Hohenstein
Manager, Environmental Operations
EHS Operations
3M Company
Building 42-2E-27
St. Paul, MN 55106

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1 Introduction

Results are reported for the analysis of perfluorooctanoate (PFOA) and perfluorooctanesulfonate (PFOS) in water samples received at Exygen as requested by Gary Hohenstein of 3M Company.

The Exygen project number assigned to the samples is L0004708. Table I lists the target analytes quantitated for the samples.

Table I. Target Analytes for Quantitation

<u>Parameter</u>	<u>Acronym</u>	<u>Formula</u>
Perfluorooctanoate	PFOA or C8 acid	$C_7F_{15}COO^-$
Perfluorooctanesulfonate	PFOS or C8 sulfonate	$C_8F_{17}SO_3^-$

2 Sample Receipt

Four samples were received at Exygen in 500 mL white plastic narrow-mouth bottles. At the request of 3M, all samples were analyzed. A copy of all sample log-in information is presented in Attachment A.

The samples were received on 03/01/05. The samples were shipped ambient via UPS. The samples were stored refrigerated from time of receipt until analysis.

3 Methods - Analytical and Preparatory

The Exygen method entitled, "Method of Analysis for the Determination of Perfluorooctane sulfonate (PFOS), Perfluorooctane sulfonylamide (PFOSA), and Perfluorooctanoate (POAA) in Water," modified for isomer separation, was used to prepare and analyze the samples. A summary of the sample preparation can be found in Section 3.1 and the modified analysis conditions for isomer separation can be found in Section 3.2.

3.1 Water Sample Preparation

Solid phase extraction (SPE) was used to prepare the water samples for LC/MS/MS analysis. Forty milliliters of the water sample was transferred to a C_{18} SPE cartridge. The cartridge was washed with 5 mL of a 40% methanol: 60% water wash. The cartridge was eluted with 5 mL of 100% methanol. A portion of the extract was transferred to autosampler vials and analyzed using electrospray LC/MS/MS.

3.2 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (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, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against extracted standards.

An HP1100 system interfaced to an Applied Biosystems API 4000 was used to analyze the sample extracts for quantitation.

Isomer separation was obtained using a gradient elution through a Thermo Fluophase RP, 50 x 2.1 mm x 5µm column. The sum of the isomers is reported as a single value for each of the analytes.

The following gradient was performed:

Mobile Phase (A): 2mM Ammonium Acetate in Water
Mobile Phase (B): Methanol

Time	%A	%B
0.0	65	35
1.0	65	35
8.0	25	75
10.0	25	75
11.0	65	35
18.0	65	35

The following parameters were used for operation of the mass spectrometer:

Parameter	Setting
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	413->369 (PFOA), 499->80 (PFOS), 415->370 (¹³ C-PFOA, surrogate spike)
Gas Temperature	350°C
Drying Gas (N2)	7.0 L/min

4 Analysis

4.1 Calibration

A 7-point calibration curve was analyzed initially and throughout the analytical sequence for PFOA, PFOS and ¹³C PFOA. The calibration points were prepared at 0, 25, 50, 100, 250, 500, and 1000 ng/L (ppt) for LC/MS/MS analysis. Calibration standards are prepared using the same SPE procedure used for samples. The instrument response versus the concentration was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept

and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r^2 \geq 0.985$.

For the results reported here, calibration criteria were met. The calibration curves are included in the raw data in Attachment C.

4.2 Surrogates

^{13}C PFOA ($\text{C}_8\text{F}_{15}[^{13}\text{C}]\text{F}_2[^{13}\text{C}]\text{OOH}$) is used as a surrogate for the water samples. ^{13}C PFOA was added to the empty sample containers before the bottles were sent to the field for sampling. In the field, all sample and quality control bottles were filled to a volumetric fill line with the sample.

^{13}C PFOA recoveries can be found in Attachment B.

4.3 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared by adding a known concentration of PFOA, ^{13}C PFOA, and PFOS to laboratory reagents. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range.

4.4 Field Matrix Spikes

A low and a high matrix spike were prepared with the sample and sample duplicate in the field. Compounds were added to the empty sample containers before the bottles were sent to the field for sampling. Matrix spikes are used to assess method accuracy in the matrix. The matrix spikes should show recoveries between 70-130%. For the results reported here, the matrix spikes were within the acceptable range.

4.5 Sample Related Comments

The water samples were collected in duplicate. Duplicate sample results are reported in Attachment B.

5 Data Summary

Please see Attachment B for a detailed listing of the analytical results. The results are reported in parts per billion (ng/mL) for PFOA and PFOS.

6 Statement of Accuracy

Based on results of laboratory control spikes, field matrix spikes, and surrogate spikes, the analytical accuracy for PFOS and PFOA results is $\pm 30\%$. Laboratory control spikes, surrogate spikes, and field matrix spikes showed recoveries within this range.

Specific sample recoveries are given in Attachment B.

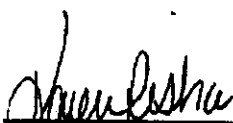
7 Data/Sample Retention

Samples will be returned to 3M 60 days after final reporting. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by Exygen. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.

8 Attachments

- 7.1 Attachment A: Analytical Results
- 7.2 Attachment B: Spike Recovery Data
- 7.3 Attachment C: Chain of Custody Forms
- 7.4 Attachment D: Raw Analytical Data

9 Signatures


Karen Risha, Principal Investigator

03/14/05
Date


John M. Flaherty, Vice President

3/14/05
Date

SECTION A

Summary of PFOA and PFOS in Water Samples E05-0186

Oxygen ID	Sample ID	Analyte Found (ng/mL)	
		C8 Acid PFOA Perfluorooctanoate	C8 Sulfonate PFOS Perfluorooctanesulfonate
L4709-1	H-W6-207643	ND	ND
L4709-2	H-W6-207643 Dup	ND	ND

ND = Not detected at or above 0.025 ng/mL.

NQ = Not quantifiable = Measured concentration between 0.025 ng/mL and the Limit of Quantitation (LOQ) which is 0.050 ng/mL.

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SECTION B

Field Matrix Spike Recovery Summary for PFOA and PFOS in Water E05-0186

Sample Description	Amount Spiked (ng/mL)	C8 Acid PFOA			C8 Sulfonate PFOS		
		Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
H-W6-207643 Low Spike (L4146-3, 0.1 ppb Spike)	0.1	ND	0.128	128	ND	0.117	117
H-W6-207643 High Spike (L4146-4, 1 ppb Spike)	1	ND	1.04	104	ND	1.03	103

ND = Not detected at or above 0.025 ng/mL

NQ = Not quantifiable = Measured concentration between 0.025 ng/mL and the Limit of Quantitation (LOQ) which is 0.050 ng/mL.

Surrogate Spike Recovery Summary for ¹³C-PFOA In Water E05-0186

Exygen ID	Sample Description	¹³ C-PFOA		
		Amount Spiked (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
L4709-1	H-W6-207643	0.5	0.491	98
L4709-2	H-W6-207643 Dup	0.5	0.522	104
L4709-3	H-W6-207643 Low Spk	0.5	0.546	109
L4709-4	H-W6-207643 High Spk	0.5	0.540	108

0007

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State College, PA 16801, USA
T: 800.281.3219
F: 814.272.1019
exygen.com

SECTION C



3058 Research Drive
State College, PA 16801

Phone: 814-272-1039
Fax: 814-231-1580

Login

Login Group: L0004709

Login #: 4819
Project: P0001275
Company Name: 3M
Submitted By: KENT LINDSTROM
Login Type: Immediate Receipt of Samples
Started: True
Date Start: 03/01/2005
Due Date: 03/11/2005
Received Date: 03/01/2005
Received By: Ammerman, Mark
Spread Sample:
Label:
Exygen SD/PI: Risha, Karen
Project Title/Type: Cottage Grove Residential Water Sampling / ROUTINE
Login Notes:
Conform Notes:

Conform COC Sample: True
Conform COC: True
Conform Sample: True
Conform Request: True

Packages / Containers

<u>Package</u>	<u>Carton</u>	<u>Date / Condition</u>	<u>Shipper / ID</u>	<u>Temp. Control/Temp.</u>	<u>Direction / Handled By</u>	
PK0005560		Login Date: 3/1/2005 11:31:46AM Package & Contents Uncompromised	UPS 1Z57955E0142913994	None 22.3	RECEIVED Ammernan, Mark	
<u>Container #</u>	<u>Gross Weight</u>	<u>pH</u>	<u>Container Type</u>	<u>Preservative</u>	<u>Mfg. Lot</u>	<u>Mfg. ID</u>
C0063087	253.90 g		500 ml Clear Plastic Narrow	NONE		
C0063088	259.00 g		500 ml Clear Plastic Narrow	NONE		
PK0005561		Login Date: 3/1/2005 11:34:38AM Package & Contents Uncompromised	UPS 1Z57955E0140875806	None 22.3	RECEIVED Ammernan, Mark	
<u>Container #</u>	<u>Gross Weight</u>	<u>pH</u>	<u>Container Type</u>	<u>Preservative</u>	<u>Mfg. Lot</u>	<u>Mfg. ID</u>
C0063089	252.90 g		500 ml Clear Plastic Narrow	NONE		
C0063090	244.40 g		500 ml Clear Plastic Narrow	NONE		

Samples

Sample ID	Container	Matrix	Fraction	Sample	Date Sampled	Date Loaded In	Date Due
L0004709-0001	C0063087	LIQUID	Water	HW6-207643	02/28/2005	03/01/2005	03/11/2005
L0004709-0002	C0063089	LIQUID	Water	HW6-207643 Dup	02/28/2005	03/01/2005	03/11/2005
L0004709-0003	C0063088	LIQUID	Water	HW6-207643 Low Spike	02/28/2005	03/01/2005	03/11/2005
L0004709-0004	C0063090	LIQUID	Water	HW6-207643 High Spike	02/28/2005	03/01/2005	03/11/2005

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3/1/2005
Login.rpt

Report Version: Feb 22 2005 9:53AM
Page 1 of 2

Instance: R0149340

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100 101 102 103 104 105 106 107 108 109 110 111 112 113 114 115 116 117 118 119 120 121 122 123 124 125 126 127 128 129 130 131 132 133 134 135 136 137 138 139 140 141 142 143 144 145 146 147 148 149 150 151 152 153 154 155 156 157 158 159 160 161 162 163 164 165 166 167 168 169 170 171 172 173 174 175 176 177 178 179 180 181 182 183 184 185 186 187 188 189 190 191 192 193 194 195 196 197 198 199 200 201 202 203 204 205 206 207 208 209 210 211 212 213 214 215 216 217 218 219 220 221 222 223 224 225 226 227 228 229 230 231 232 233 234 235 236 237 238 239 240 241 242 243 244 245 246 247 248 249 250 251 252 253 254 255 256 257 258 259 260 261 262 263 264 265 266 267 268 269 270 271 272 273 274 275 276 277 278 279 280 281 282 283 284 285 286 287 288 289 290 291 292 293 294 295 296 297 298 299 300 301 302 303 304 305 306 307 308 309 310 311 312 313 314 315 316 317 318 319 320 321 322 323 324 325 326 327 328 329 330 331 332 333 334 335 336 337 338 339 340 341 342 343 344 345 346 347 348 349 350 351 352 353 354 355 356 357 358 359 360 361 362 363 364 365 366 367 368 369 370 371 372 373 374 375 376 377 378 379 380 381 382 383 384 385 386 387 388 389 390 391 392 393 394 395 396 397 398 399 400 401 402 403 404 405 406 407 408 409 410 411 412 413 414 415 416 417 418 419 420 421 422 423 424 425 426 427 428 429 430 431 432 433 434 435 436 437 438 439 440 441 442 443 444 445 446 447 448 449 450 451 452 453 454 455 456 457 458 459 460 461 462 463 464 465 466 467 468 469 470 471 472 473 474 475 476 477 478 479 480 481 482 483 484 485 486 487 488 489 490 491 492 493 494 495 496 497 498 499 500 501 502 503 504 505 506 507 508 509 510 511 512 513 514 515 516 517 518 519 520 521 522 523 524 525 526 527 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2222 2223 2224 2225 2226 2227 2228 2229 2230 2231 2232 2233 2234 2235 2236 2237 2238 2239 2240 2241 2242 2243 2244 2245 2246 2247 2248 2249 2250 2251 2252 2253 2254 2255 2256 2257 2258 2259 2260 2261 2262 2263 2264 2265 2266 2267 2268 2269 2270 2271 2272 2273 2274 2275 2276 2277 2278 2279 2280 2281 2282 2283 2284 2285 2286 2287 2288 2289 2290 2291 2292 2293 2294 2295 2296 2297 2298 2299 2300 2301 2302 2303 2304 2305 2306 2307 2308 2309 2310 2311 2312 2313 2314 2315 2

Login

Login Reviewed By: gc Date/Time: 3-1-05 1155



3M Environmental Laboratory

Form 38776 - PWS

Shipping Address:
3M Bldg. 2-35-09
330 Bush Avenue
St. Paul, MN 55108
Telephone:
Sample Receiving: (651) 779-4848
Alternate: (651) 779-4753
FAX: (651) 779-6178

Chain of Custody / Request for Laboratory Analytical

08547

Project ID/Project Name: Hastings Municipal Well Field
Template #: FE-3.11
Project Lead: Kent Lindstrom
Dept. # (main): 530711
Class/Job/Project #: 002267449

EOS-0186

Contact Name: Gary Hohenstein
Company: 3M
Mailing Address:
City, State, Zip:
Telephone #:
FAX #:

Date Available: 7/1
Date Due: 7/3
Contract Lab: Exygen

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

* Note: Reference Trip Blank on EOS-0184
EOS 2/24/05

Analysis Requested:
Complete below. Attach any associated information.

Preservatives:
HNO3
H2SO4
VOCs
None
Other

Total Number of Containers

See Attached Lab Request
EOS-0186

Enter the number of containers of each

Method/
Media

Date Sampled

Time Sampled

3M
LIMS#

Client Sample Identification

Item #

1. HW6-207643

2. HW6-207643 Dup

3. HW6-207643 Low Spike

4. HW6-207643 High Spike

5.

6.

7.

8.

9.

10.

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

Collected by (print): Brad Jacobson

Item #

1-4

1-4

1-4

1-4

1-4

1-4

1-4

1-4

1-4

Date

Time

Received by/Initiation

1252

2/24/05

Self

1530

2/28/05

UPS AM

1320

2/28/05

1320

2/28/05

1320

2/28/05

1320

2/28/05

1320

2/28/05

1320

2/28/05

1320

2/28/05

1320

Sample Condition Upon Receipt: ☒ Acceptable ☐ Other:

Temperature: 1 °C ☐ Recooled on Ice

Other Associated CoCs: KPL

Comments:

* TO EXYGEN

2/28/2005

Page 1 of 1

3M ENVIRONMENTAL LABORATORY
CONTRACT LABORATORY WORK ORDER BY SAMPLE

Project: E05-0186

Contract Lab(s): EXYGEN

Requester: Hohenstein, Gary A (0042-02E-27)

Department: 530711

Project Number: 0022674449

Date Received: 2/25/2005

Project Description: Hastings Municipal Well FC's 11

Completion Date:

Project Lead: Kent Lindstrom

Phone Number: 651-778-5352

Email Address: krlindstrom@mmm.com

Ship Date:

2/28/05

Comments:

Pace field sampling.

Note: Reference trip blank on E05-0184. BGJ 2/28/05

E05-0186 samples traveled with E05-0184 samples. AC, PLF, 2/28/2005.

3M Sample Number	Sampled Date	Sample Description	
E05-0186-79868	2/28/2005	H-W6-207643	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	3/15/2005
PFOS	PFOS by ESMS	PFOS	3/15/2005
E05-0186-79869	2/28/2005	H-W6-207643 Dup	
<u>Analysis Code</u>	<u>Analytical Method</u>	<u>Components</u>	<u>Analysis Due Date</u>
PFOA	PFOA	PFOA	3/15/2005
PFOS	PFOS by ESMS	PFOS	3/15/2005

0011

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