



May 5, 2015

UPS Next Day

Mr. Gary Krueger
Remediation Division
Minnesota Pollution Control Agency
520 Lafayette Road North
St. Paul, MN 55155

Re: 3M Cottage Grove Site (Site)
2014 Annual Perfluorochemical (PFC) Groundwater, Pore Water and Surface Water Report
And Response to Comments on the 2013 Annual Groundwater Monitoring Report

Dear Mr. Krueger:

Enclosed please find three (3) electronic copies and three (3) hard copies of the 2014 Annual PFC Groundwater, Pore Water and Surface Water Report for the 3M Cottage Grove Site.

In addition, this letter provides a response to AECOM comments on the 2013 Annual PFC Groundwater, Pore Water and Surface Water Report, which was transmitted to 3M via email dated October 13, 2014, from the MPCA. The AECOM comments on the 2013 annual report were primarily in the form of summary discussions. Specific items in their discussions are identified and a response is referenced by page number and topic from their September 8, 2014, memorandum to MPCA. For ease of reference, comments or discussion items provided below are followed by 3M's response. Clarifying information regarding these comments was included, where possible and as appropriate, in the attached 2014 annual report.

AECOM Comments

Item 1 – (page 1, Groundwater Elevations, 1st paragraph): There is no discussion or interpretation of results that presents findings on the production well's ability to provide groundwater plume containment.

3M Response to Comment: *In accordance with the MPCA approved Groundwater Sampling Plan for the Site, a Performance Evaluation Report will be submitted to the MPCA on an annual basis after the complete groundwater interception system has been installed and is fully operating. The Performance Evaluation Report will be submitted in addition to an Annual Monitoring Report that provides a summary of the groundwater quality data for the Site.*

As discussed in the Groundwater Sampling Plan for the Site, the Performance Evaluation Report will include the following information:

- *An evaluation of site-wide hydrogeologic and remediation systems.*
- *An assessment of PFC migration in groundwater.*

**Exhibit
3630**

State of Minnesota v. 3M Co.,
Court File No. 27-CV-10-28862

May 5, 2015

- *An evaluation of the groundwater remediation systems with respect to:*
 - *The effectiveness of plume capture by the existing system(s).*
 - *Modifications to pumping, if necessary, including the reduction or increase in pumping.*
 - *The effect from any of the removal actions that have been completed.*
- *A discussion of the effects of remediation including sediment removal from the East Cove and groundwater extraction from PW-09, PW-10 and existing production wells.*
- *Documentation that treated groundwater is discharged in accordance with state requirements as stated in the RD/RA Plan.*

*An extended pilot pump test program was performed at the Site from February 2013 to February 2014 to determine the number, and flow rates, of wells necessary to fulfill 3M's commitment under the selected groundwater alternative for the Site (Alternative GW-1). The results of the extended pilot pump test program and recommended groundwater interception system were discussed during a presentation made to the MPCA in June and November 2014, and are summarized in a document titled *Extended Groundwater Pilot Pump Test and Design of Groundwater Recovery System, Cottage Grove Site*, provided to MPCA on March 17, 2015.*

Item 2 – (page 2, Groundwater Quality, 1st paragraph): By identifying each of the Areas on one figure (currently these Areas are identified using different maps symbols on three separate figures) using shading or outlining, there would be a simplified identification system and provide a greater degree of consistency between text and figures.

3M Response to Comment: *A figure will be added to future reports to show the approximate location of each area referred to in the text.*

Item 3 – (page 2, Groundwater quality, 4th paragraph) – Groundwater quality tables should provide a reference to Minnesota Department of Health (MDH) Risk Limits (HRLs).

3M Response to Comment: *As presented in previous responses to comments, comparing the Site groundwater data to MDH HRLs is not appropriate for the Site since the groundwater is not being used as drinking water.*

Item 3 – (page 2, Groundwater quality, 5th paragraph) – The hydraulic gradient value presented in Section 3.1 does not include units representing the numerical value.

3M Response to Comment: *The units feet per foot (ft/ft) will be added to the text; however, this is a dimensionless parameter and units are not always reported.*

Item 4 – (page 2, Pore Water/Surface Water Quality, 1st paragraph, 3rd sentence) – Including an additional sentence to the last paragraph of Section 3.3 indicating annual pore water and surface water sampling will continue in 2015 would provide clarification on the intent to perform additional annual sampling.

3M Response to Comment: *As presented in previous responses to comments, and in meetings held with the MPCA, 3M's position is that the effectiveness of the Site remedial activities that have been completed, and are continuing, can be adequately assessed using groundwater elevation and groundwater PFC monitoring data. 3M does not agree that additional pore water and surface water sampling will provide a technically sound basis to evaluate the remedial alternatives selected for the Site. As presented to the MPCA in the past, with the completion of the September 2014 sampling round, 3M has met all their obligations regarding pore water/surface water sampling, as specified in Section 4.2 of the MPCA-approved Sampling Plan for the Site.*

Item 5 – (page 2, Pore Water/Surface Water Quality, 2nd paragraph, 1st sentence) – Mississippi River Pore Water and Surface Water Sample Results does not include the units presented by the numeric values included in the table.

3M Response to Comment: *Comment noted, a revised table is attached and presents the values of the numeric values as parts per billion (ppb).*

Item 6 – (page 2, Findings/Future Course of Action, 1st paragraph, 1st sentence) – The Report indicates that baseline groundwater, pore water and surface water samples were collected in October 2012. Although this is accurate for groundwater samples, pore water and surface water baseline samples were collected in 2006 (with the exception of 1W-25d and f) with a complete sampling event performed in 2011.

3M Response to Comment: *The report uses the term "baseline" as the sampling event performed in October 2012 prior to the startup of the extended pilot pump test program in February 2013.*

May 5, 2015

Item 7 – (page 2, Findings/Future Course of Action, 1st paragraph, 3rd sentence) – AECOM suggest that following completion and presentation of the extended pump test results to MPCA, that future annual reports include a discussion of production well pumping rates and the ability of the production wells to effectively provide plume containment. Future annual reports should also include, as previously requested by the MPCA, a summary of annual pore water and surface water sample results.

3M Response to Comment: *See responses to Item 1 and Item 4.*

Item 8 – (page 3, General Report Comments, 1st paragraph, 3rd sentence) – Although the results of the extended pump test will be discussed under separate cover, a brief discussion of the extended pumping test would provide context for the reviewer as it is discussed throughout the remainder of the report

3M Response to Comment: *The report that was submitted in 2014 to the MPCA was for the 2013 monitoring program and the extended pilot pump test had not been completed as of the end of 2013. The extended pilot pump test was completed in September 2014 with the collection of the final pore water and surface water sampling. Results of the pump test are summarized in the Extended Groundwater Pilot Pump Test and Design of Groundwater Recovery System report submitted to MPCA via cover letter dated March 17, 2015.*

If you have any questions regarding this information, please call me at (651) 737-3477.

Sincerely,



Karie Blomquist, P.E.
Environmental Engineering Specialist
Corporate Environmental Programs
Building 224-5W-17

Enclosures

Cc: Mr. Sandeep Burman – MPCA
Mr. Fred Campbell – MPCA
Mr. Gerald Stahnke – MPCA
Ms. Virginia Yingling – MDH

3M_MN01595953

bcc: G.A. Hohenstein – 224-5W-03
J. Kesari – Weston Solutions (cover only)
M.A. Nash – 220-9E-02
F.A. Tayab – Bickel & Brewer
M.C. Yeager – 220-9E-02
J.R. Kotsmith – 224-5W-17



Table 3-4
Summary of Pore Water and Surface Water Sample Results
September/October 2006 through August 2013
3M Cottage Grove Site

LOCATION	Approximate distance from shoreline	ANALYTE	FOREWATER (µg/L; ppb)				SURFACE WATER (µg/L; ppb)				
			Sept/Oct 06	Jun-11	Oct-12	Aug-13	Sept/Oct 06	Jun-11	Oct-12	Aug-13	
			IW	IW	IW	IW	Shallow	Deep	Composite	Composite	Composite
IW-09b	50 feet	PFBA	1.35	67.6	1.67	30.3	<0.100	<0.050	<0.100	0.553	<0.025
		PFOA	0.585	43.2	2.03	109	<0.050	0.530	<0.025	0.187	<0.024
		PFOS	0.114	45.5	1.27	2.84	<0.050	<0.050	<0.050	0.124	<0.0232
IW-09	160 feet	PFBA	5.01	2.32	3.65	8.10	1.24	NR	<0.100	0.300	<0.025
		PFOA	2.69	1.25	1.41	5.74	0.192	0.167	<0.025	0.139	<0.024
		PFOS	1.09	0.415	0.958	0.822	0.162	0.183	<0.050	0.088	<0.0232
IW-09d	360 feet	PFBA	0.112	83.3	11.8	0.057	NR	<0.200	<0.100	0.158	<0.025
		PFOA	<0.050	48.5	0.355	<0.0240	0.065	NR	<0.025	0.064	<0.024
		PFOS	<0.050	0.533	0.141	<0.0232	<0.050	<0.050	<0.050	0.043	<0.0232
IW-09f	500 feet	PFBA	NR	2.98	<0.0500	30.7	0.405	0.477	<0.100	0.264	<0.025
		PFOA	0.054	0.158	<0.0240	2.83	<0.050	0.054	<0.025	0.085	<0.024
		PFOS	<0.050	0.090	<0.0232	0.061	<0.050	<0.050	<0.050	0.051	<0.0232
IW-14b	50 feet	PFBA	695	79.9	24.9	32.1	NR	0.540	<0.100	0.543	0.0415
		PFOA	436	467	123	221	0.195	0.156	<0.025	1.36	0.051
		PFOS	12.2	64.9	40.9	25.5	0.063	0.054	<0.050	0.780	<0.0232
IW-14	100 feet	PFBA	281	38.8	142	192	0.414	NR	<0.100	0.0618	<0.025
		PFOA	300	205	529	517	0.053	0.057	<0.025	<0.024	<0.024
		PFOS	12.4	28.3	18.3	4.22	NR	<0.050	<0.050	<0.0232	<0.0232
IW-14d	300 feet	PFBA	0.282	0.318	0.747	1.14	0.329	0.318	<0.100	<0.050	<0.025
		PFOA	<0.050	0.054	0.257	0.115	<0.100	<0.100	<0.025	<0.024	<0.024
		PFOS	<0.050	0.089	0.947	0.032	<0.050	<0.050	<0.050	<0.0232	<0.0232
IW-14f	500 feet	PFBA	0.178	33.3	78.4	39.1	<0.050	<0.050	<0.100	<0.050	<0.025
		PFOA	<0.050	0.813	59.3	0.926	<0.050	<0.050	<0.025	<0.024	<0.024
		PFOS	<0.025	0.086	1.23	0.05	<0.050	<0.050	<0.050	<0.0232	<0.0232

ppb - parts per billion
µg/L - micrograms per liter
IW - Interstitial Water (Pore water)
NR - Not reported due to quality control issues
NS - Not sampled

Notes: In September/October 2006 two surface water samples were collected at discrete depth intervals at each location (i.e., shallow and deep, 0.2 and 0.8 of the total depth of the water column). In June 2011, October 2012, August 2013, and October 2014 one composite sample was collected at each location, composited from the same depth intervals.

Table 3-4 Pore Water Results

3M_MN01595955



Table 3-4 (cont'd)
Summary of Pore Water and Surface Water Sample Results
September/October 2006 through August 2013
3M Cottage Grove Site

LOCATION	Approximate distance from shoreline	ANALYTE	POREWATER (µg/L; ppb)				SURFACE WATER (µg/L; ppb)				
			Sept/Oct 06	Jun-11	Oct-12	Aug-13	Sept/Oct 06	Jun-11	Oct-12	Aug-13	
			IW	IW	IW	IW	Shallow	Deep	Composite	Composite	Composite
IW-19b	50 feet	PFBA	NR	198	154	157	2.81	1.96	<0.100	0.223	0.115
		PFOA	118.3	34.4	10.3	6.96	0.154	0.129	<0.025	0.357	0.434
		PFOS	53.1	43.2	95.6	40.4	0.127	0.089	<0.050	0.100	0.412
IW-19	100 feet	PFBA	86.6	107	112	18.3	NR	NR	<0.100	0.089	0.037
		PFOA	28.9	257	843	1.61	0.119	0.126	<0.025	0.041	0.108
		PFOS	NR	30.3	81.1	0.591	0.105	0.097	<0.050	<0.0232	<0.0232
IW-19d	300 feet	PFBA	1.40	0.143	4.07	8.00	0.095	0.117	<0.100	0.069	<0.025
		PFOA	0.184	0.097	0.206	0.206	<0.050	<0.050	<0.025	<0.024	<0.024
		PFOS	0.081	0.118	0.246	0.207	<0.050	<0.050	<0.050	<0.0232	<0.0232
IW-19f	500 feet	PFBA	118	0.57	141	47.1	0.245	0.516	<0.100	0.051	<0.025
		PFOA	6.84	<0.0250	15.3	183	<0.050	NR	<0.025	0.028	<0.024
		PFOS	1.71	<0.0500	1.26	10.9	<0.050	<0.050	<0.050	<0.0232	<0.0232
IW-25b	50 feet	PFBA	NS	.56	1.47	37.2	NS	NS	12.5	0.686	0.397
		PFOA	NS	97.3	1.3	169	NS	NS	1.01	0.202	0.328
		PFOS	NS	595	2.41	86	NS	NS	1.10	0.151	0.132
IW-25	100 feet	PFBA	23.1	13.6	34.7	35.5	NR	NR	6.25	0.819	3.64
		PFOA	129	110	74.7	9.98	0.163	0.146	0.583	0.575	1.26
		PFOS	206	82.4	0.916	3.28	0.695	0.102	0.586	0.422	0.311
IW-25d	300 feet	PFBA	NS	25	15.9	38.6	NS	NS	<0.100	0.389	0.043
		PFOA	NS	16.6	2.04	19.5	NS	NS	<0.025	0.335	<0.024
		PFOS	NS	0.138	19.1	0.141	NS	NS	<0.050	0.236	<0.0232
IW-25f	500 feet	PFBA	NS	2.02	1.23	6.05	NS	NS	<0.100	0.760	<0.025
		PFOA	NS	1.25	0.482	5.13	NS	NS	<0.025	0.163	<0.024
		PFOS	NS	2.64	0.201	2.18	NS	NS	<0.050	0.087	<0.0232

ppb - parts per billion
µg/L - micrograms per liter
IW - Interstitial Water (Pore water)
NR - Not reported due to quality control issues
NS - Not sampled

Note: In September/October 2006 two surface water samples were collected at discrete depth intervals at each location (i.e., shallow and deep, 0.2 and 0.8 of the total depth of the water column). In June 2011, October 2012, August 2013, and October 2014 one composite sample was collected at each location, composited from the same depth intervals.

Table 3-2.PWf SW f0306a.xlsx

3M_MN01595956



**2014 ANNUAL PERFLUOROCHEMICALS (PFCs)
GROUNDWATER, PORE WATER AND SURFACE WATER
REPORT FOR THE 3M COTTAGE GROVE SITE**

COTTAGE GROVE, MN

APRIL 2015

Prepared for:

3M Company

Prepared by:

**WESTON SOLUTIONS, INC.
West Chester, Pennsylvania 19380**

W.O. #02181.002.197

3M_MN01595957

3630.0008



TABLE OF CONTENTS

Section	Page
1. INTRODUCTION.....	1-1
1.1 PURPOSE AND OBJECTIVES OF THE SAMPLING PLAN	1-3
2. SAMPLING PROGRAM.....	2-1
2.1 GROUNDWATER	2-1
2.2 PORE WATER AND SURFACE WATER	2-3
3. WATER MONITORING AND RESULTS.....	3-1
3.1 GROUNDWATER ELEVATIONS	3-1
3.2 EXTENDED PILOT PUMP TEST	3-2
3.3 GROUNDWATER QUALITY	3-3
3.3.1 Background.....	3-4
3.3.2 D1/D2 Area.....	3-5
3.3.3 D9 Area.....	3-6
3.3.4 WWTP Area.....	3-6
3.3.5 D5 Area.....	3-7
3.3.6 D8 Area.....	3-7
3.3.7 East Cove Area	3-8
3.4 PORE WATER/SURFACE WATER QUALITY.....	3-8
4. FINDINGS.....	4-1
4.1 GROUNDWATER ANALYTICAL DATA	4-1
4.2 SURFACE WATER / PORE WATER ANALYTICAL DATA.....	4-2
4.3 SITE HYDROGEOLOGIC CONDITIONS	4-2
5. FUTURE COURSE OF ACTION.....	5-1
6. REFERENCES.....	6-1



FIGURES

Figure		Page
Figure 1-1	Site Location Map.....	1-4
Figure 2-1	Groundwater Monitoring Network	2-5
Figure 2-2	Pore Water and Surface Water Sampling Locations – 3M Cottage Grove Site ..	2-6
Figure 3-1	Well Locations and Site Geologic Map	3-10
Figure 3-2	Groundwater Elevation Contour Map, January 2014	3-11
Figure 3-3	Groundwater Elevation Contour Map, April 2014	3-12
Figure 3-4	Groundwater Elevation Contour Map, August 2014	3-13
Figure 3-5	Groundwater Elevation Contour Map, December 2014	3-14



TABLES

Table	Page
Table 2-1 PFCs Groundwater Monitoring Plan	2-7
Table 3-1 Depth-to-Groundwater and Groundwater Elevation Data - October 2012 – December 2014	3-15
Table 3-2 Summary of Groundwater PFC Analytical Data - October 2012 through October 2014.....	3-16
Table 3-3 Mann-Kendall Trend Test Summary	3-19
Table 3-4 Summary of Pore Water and Surface Water PFC Analytical Data	3-20

ATTACHMENTS

ATTACHMENT A	SUMMARY OF WELL CONSTRUCTION INFORMATION
ATTACHMENT B	PORE WATER AND SURFACE WATER SAMPLING SHEETS SEPTEMBER/OCTOBER 2014 SAMPLING EVENT
ATTACHMENT C	HYDROGRAPHS FOR SITE MONITORING WELLS
ATTACHMENT D	LABORATORY ANALYTICAL PACKAGES AND CHAIN-OF-CUSTODY DOCUMENTATION FOR GROUNDWATER SAMPLING EVENTS
ATTACHMENT E	PFBA, PFOA AND PFOS TREND GRAPHS
ATTACHMENT F	LABORATORY ANALYTICAL PACKAGES FOR PORE WATER AND SURFACE WATER SAMPLING – SEPTEMBER/OCTOBER 2014



1. INTRODUCTION

This annual 2014 report has been prepared by Weston Solutions, Inc. (WESTON®) for 3M Company (3M), and includes an assessment of perfluorochemicals (PFCs) in groundwater, pore water, and surface water at the 3M Cottage Grove, Minnesota facility (Site). The Site has been in operation since 1947, and currently manufactures a range of products including adhesive products, specialty paper, industrial polymers, abrasives, and reflective road sign materials. Research and development of a proprietary nature are also performed at the facility. The Site is located approximately 3 miles southeast of the City of Cottage Grove, Minnesota, and is approximately 1,700 acres in size. The industrial, developed portion of the site is approximately 200 acres as shown in Figure 1-1.

Since the early 1980s, 3M has worked cooperatively with the Minnesota Pollution Control Agency (MPCA) in conducting investigations to characterize environmental media at the Site. More recently, as early as December 2004, 3M had worked with the MPCA to voluntarily assess the presence of PFCs at the Site. This included field activities to characterize site soil and groundwater quality as well as sediment and surface water quality at the East Cove. It also included field activities to characterize sediment, surface water, and pore water quality in the Mississippi River. Subsequently, 3M entered into a Settlement Agreement and Consent Order (the Agreement) in May 2007 for the purpose of performing the remedial investigations and response actions to address PFCs at the Site.

In September and October 2008, two groundwater production wells (PW-09 and PW-10) were installed at the Site and a 48-hour pumping test was performed on each. The results of the short-term pump tests were utilized to develop groundwater remedial Alternative GW-1, which consists of an expanded groundwater extraction network to prevent off-site migration of groundwater beneath the Eastern Disposal Areas (Areas D1, D2 and D9) and the East Cove, as well as the eastern portion of the main plant area. These areas are shown on Figure 1-2. The extracted groundwater would be routed through a new granular activated carbon (GAC) treatment building and subsequently used in manufacturing operations prior to discharge.



As described in the MPCA-approved Feasibility Study (FS) Report (WESTON, 2008) and the Remedial Design/Response Action (RD/RA) Plan (WESTON, 2009) for the Site, 3M and MPCA agreed that the remedial Alternative GW-1 would be implemented in a phased manner. The phased implementation of the remedial Alternative GW-1 involves the performance of an extended pilot pump test on the two new production wells (PW-09 and PW-10), to refine and optimize the long-term pumping system operational parameters, and to identify if additional production wells are needed.

Soil and East Cove sediment response actions also have been completed in accordance with the Agreement and MPCA-approved RD/RA Plan to address the PFCs found in these media. In June 2011, prior to the implementation of remedial measures in the East Cove, pore water (interstitial water (IW)) and surface water sampling was performed in the Mississippi River at a subset of the locations previously sampled in 2006. PFBA, PFOA, and PFOS concentrations in pore water and surface water were obtained at four transect locations to characterize pre-remediation conditions for comparison to future PFC concentrations detected in these media.

At a meeting with the MPCA on March 15, 2012, 3M agreed to prepare a monitoring plan for PFCs in groundwater at the Site, which also would include pore water and surface water sampling in the Mississippi River prior to, during, and after the extended pump test. The *Groundwater, Pore Water and Surface Water Sampling Plan for Perfluorochemicals (PFCs) – 3M Cottage Grove Site* (Sampling Plan) was submitted to the MPCA on May 4, 2012.

After resolution of MPCA and AECOM comments on the Sampling Plan, the PFCs baseline (i.e. prior to the extended pump test) groundwater, pore water, and surface water sampling was performed in October 2012. The results of the October 2012 sampling event were provided to MPCA in the June 2013 in the *Baseline Monitoring Report for Perfluorochemicals (PFCs) in Groundwater, Pore Water, and Surface Water, 3M Cottage Grove Site, Cottage Grove, MN* (WESTON, 2013).

The extended pilot pump test was initiated in February 2013 with the startup of production wells PW-09 and PW-10. Groundwater extracted from PW-09 and PW-10 was routed to



the new GAC treatment facility. The field data collection period of the extended pilot pump test continued until February 2014. The details of the extended pilot pump test were submitted to MPCA in March 2015 in a separate report. In accordance with the RD/RA Plan, PW-09 and PW-10 continue to operate in conjunction with existing Site production wells to intercept groundwater migrating toward the Mississippi River.

1.1 PURPOSE AND OBJECTIVES OF THE SAMPLING PLAN

In addition to the baseline groundwater sampling, the Sampling Plan includes a requirement for quarterly (for four years after completion of the extended pilot pump test) and annual groundwater sampling for PFCs. The monitoring for PFCs is being implemented in accordance with the provisions of the MPCA-approved RD/RA Plan to provide groundwater quality data to assess the impact of the completed PFCs soil and sediment remediation activities, and the effectiveness of on-going groundwater remediation activities. The Sampling Plan includes Mississippi River pore water and surface water sampling for PFCs prior to, during, and after the extended pilot pump test to assess whether any trends are apparent for these environmental media.

The components of the Sampling Plan include:

- Identification of sample type and locations, monitoring frequency, and monitoring rationale,
- Procedures for sample collection,
- Procedures for documentation and quality assurance/quality control (QA/QC),
- Sample analysis and analytical parameters, and
- Reporting.

This is the second annual monitoring report since baseline sampling activities were conducted in October 2012. It contains a description of sampling methodologies and the results of groundwater, pore water and surface water sampling performed in 2014 in Sections 2 and 3. A summary of the findings of the groundwater, pore water and surface water investigations is provided in Section 4 and future course of action is presented in Section 5.



Legend
□ Property Lines

Aerial Source: ESRI Imagery
Mapping Service. 2013

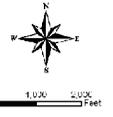


Figure I-1
Site Location Map
Cottage Grove Site

R:\VIC\cottage_grove\moss\site\layout_049_01.mxd, 3/14/2014 11:22:55 AM, print

3M_MN01595964



2. SAMPLING PROGRAM

The PFC Sampling Plan contains the monitoring program for the Site. As indicated in the Sampling Plan, 3M implemented baseline groundwater monitoring for PFCs in October 2012, prior to the startup of the extended pilot pump test. These baseline monitoring results are used as a reference point for comparison to future monitoring. The 2014 PFC sampling program that was conducted to assess groundwater, pore water, and surface water conditions at the Site is described in the following sections.

2.1 GROUNDWATER

The current groundwater monitoring network at the Site consists of 33 monitoring wells and one piezometer. In addition, there are six existing production wells (PW-01 through PW-05/PW-06), one infrequently used production well (PW-07), and two new production wells (PW-09 and PW-10). Production wells PW-02 and PW-05 are currently the primary production wells used for water supply (not used for drinking water) at the Cottage Grove Plant. Production wells PW-01, PW-03, PW-04 and PW-05/PW-06 are not routinely pumped, but are available for use on an as-needed basis. Former production well PW-08 was abandoned in April 2012 as it was no longer used.

The monitoring wells and production wells that are included in the annual and quarterly sampling network for PFCs are presented in Table 2-1 and shown on Figure 2-1. A table summarizing available well construction and other information for the Site wells is included in Attachment A.

In accordance with the Sampling Plan, the annual groundwater sampling event was performed in April 2014 and included the analysis of the following 12 PFCs:

- Perfluorooctanoic acid (PFOA)
- Perfluorooctane sulfonate (PFOS)
- Perfluorobutane sulfonate (PFBS)
- Perfluorohexane sulfonate (PFHS)
- Perfluorobutanoic acid (PFBA)
- Perfluoropentanoic acid (PFPeA)
- Perfluorohexanoic acid (PFHxA)



- Perfluoroheptanoic acid (PFHpA)
- Perfluorononanoic acid (PFNA)
- Perfluorodecanoic acid (PFDA)
- Perfluoroundecanoic acid (PFUnA)
- Perfluorododecanoic acid (PFDoA)

Quarterly groundwater sampling was performed in January, July and October 2014, and included analysis for: PFBA, PFBS, PFHS, PFOS and PFOA.

As noted in Table 2-1, a few modifications to the Site monitoring well network have occurred since the implementation of the Groundwater Sampling Plan. A summary of these modifications are provided in the following paragraphs.

Monitoring wells MW-06 and MW-14 were abandoned in May 2013 in accordance with Minnesota Department of Health (MDH) regulations. Attempts to rehabilitate both monitoring wells were made prior to abandonment. The screened interval of monitoring well MW-14 was clogged and could not be restored to a useable condition. An obstruction was present in monitoring well MW-06 at approximately 85 feet below ground surface (ft bgs) that could not be removed. A new monitoring well to replace MW-06 was not installed because MW-06 was an upgradient monitoring well and another upgradient monitoring well MW-05 is being used to assess upgradient conditions.

New monitoring wells MW-04R, MW-14R and MW-112 were installed in May 2013. Monitoring well MW-04R was installed as a replacement well for adjacent monitoring well MW-04. The open borehole interval of monitoring well MW-04 had partially collapsed limiting the flow of groundwater into this well. This well could not be abandoned due to overhead power lines that prevent access to the location by a drilling rig. Monitoring well MW-14R was installed as a replacement for monitoring well MW-14 to provide groundwater quality and elevation data in the vicinity of production wells PW-05 and PW-05/PW-06. Per MPCA approval received by 3M in January 2014, monitoring well MW-14R will replace production well PW-05/PW-06, which is not routinely pumped, as a groundwater monitoring point in the sampling program. Finally, monitoring well MW-112 was installed in the eastern Site area to provide an additional groundwater monitoring point between the East Cove and the former D1/D2 Area. Groundwater samples were collected



in July 2013 from monitoring wells MW-04R, MW-14R and MW-112 to provide baseline PFC groundwater quality data at these locations.

2.2 PORE WATER AND SURFACE WATER

Paired pore water and surface water samples were collected from the Mississippi River during the week of September 29, 2014. This sampling event was performed after the extended pilot pumping test was completed at production wells PW-09 and PW-10. This pore water and surface water sampling event could not be performed until after water levels in the Mississippi River had subsided due to above normal precipitation that caused high water conditions in the river.

Samples were collected from the same locations as the September/October 2006, June 2011 (pre-East Cove remediation), October 2012 (pre-extended pilot pump test), and August 2013 (mid-point of extended pilot pump test) sampling events. The samples were collected from the four transect locations in the Mississippi River perpendicular to the shoreline to assess the current distribution of PFCs in surface water and groundwater beneath the river (Figure 2-2). As shown in Figure 2-2, four samples were collected from each of the four transects (IW-09, IW-14, IW-19 and IW-25). The sampling locations were approximately 50 feet (ft), 100 ft, 300 ft and 500 ft from the shoreline.

These transects were selected based on their proximity to the following Site areas:

- IW-09 – transect located near the D5 Area;
- IW-14 - transect near the wastewater treatment plant (WWTP) Area;
- IW-19 - transect near the D1/D2/D9 Areas;
- IW-25 – transect near the East Cove.

Pore water and surface water sampling protocols and standard operating procedures are contained in the Sampling Plan. Modifications to the pore water and surface water sampling protocols were detailed in a response to MPCA comments dated August 21, 2012. A boat was used to gain access to each of the sampling points. Sampling was performed from downstream to upstream transect locations. A GPS with submeter accuracy was used to establish each sampling location. As agreed with MPCA, surface water samples were collected first. Based on similarities in the 2006 surface water results



from discrete depths corresponding to 20 and 80 percent of the water column, the surface water samples collected during the September/October 2014 sampling event consisted of a single composite sample of equal proportions from these same depths (20 and 80 percent) to represent the average water column concentration.

Following the collection of the surface water sample, a co-located pore water sample was collected. The pore water samples were collected using a decontaminated stainless steel sampling probe with 0.5 ft of 0.01-inch slotted screen and sufficient riser to extend above the surface of the water. The sampling probe was driven approximately one foot into the bottom sediment so that the screen was placed at an approximate depth of 0.5 ft to 1.0 ft below the top of sediment. Groundwater within the probe was purged using a peristaltic pump and disposable polyethylene tubing. Samples were collected from the pore water probes after either 3 well volumes were removed, or after the water level had sufficiently recharged after pumping dry. When possible, the pore water sampling probes were allowed to recover a minimum of 24 hours prior to sampling; however, due to high winds and the forecast for heavy precipitation that would potentially compromise sample integrity, some of the pore water locations were sampled slightly less than 24 hours after purging. Collected samples were analyzed for PFBA, PFOS and PFOA by the 3M Environmental Laboratory in St. Paul, Minnesota. Pore water and surface water sampling field sheets are provided in Attachment B.



Legend:
 ○ Monitoring Well/Recovery
 ● Protection Well
 MW-12 PFC Sampling Location



0 500 1,000
 Feet

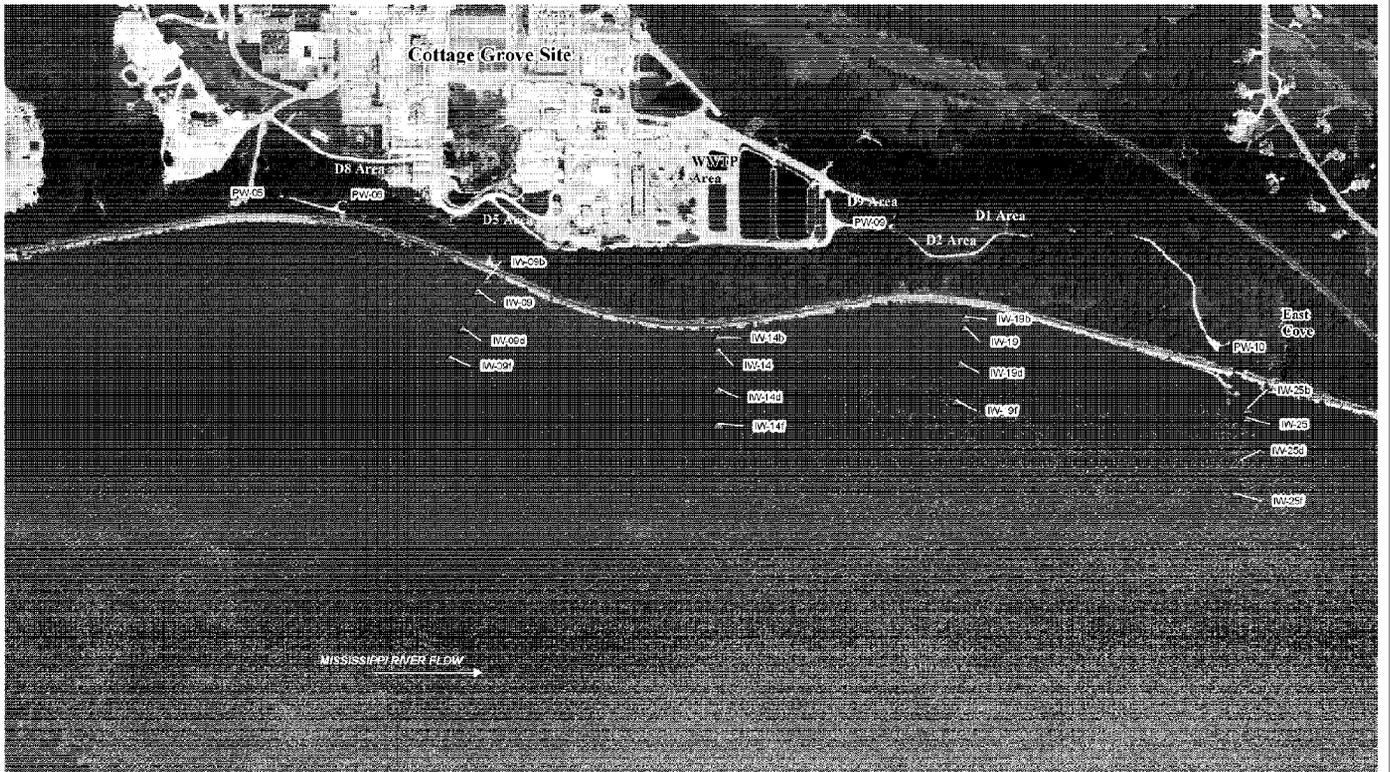


Map Source:
 U.S. Department of Agriculture, Farm Services Agency, Aerial Photography Field Office,
 National Agricultural Imagery Program (NAIP) Digital Orthorectified Images (DOQ), Minnesota, 2002

Figure 2-1
 Groundwater Monitoring Network
 Cottage Grove Site

File: \\C:\Cottage_Groves\Brow_Brothers_Sampling_While_Updated.mxd, 8/8/2014 9:42:27 AM, view

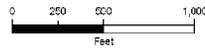
3M_MN01595970



Legend
 ▲ Pore Water Sampling Location
 ● Protection Wall

Note:
 Sampling location along transect IW-6, IW-14 and IW-15 outside with the
 sampling locations from 2008. ©Weston, 2/07

File: \\Cottage_Grove\Projects\03-12-08_001\proj\001_Updated.mxd, 09/26/11, 9:47:47, .tcm:0



Map Source:
 ESRI Imagery Mapping Services 2013

Figure 2-2
 Pore Water and Surface Water
 Sampling Locations
 Cottage Grove Site

3M_MN01595971



**Table 2-1
PFCs Groundwater Monitoring Plan
3M Cottage Grove Site
Cottage Grove, MN**

Well ID	October 2012 (Baseline)	Quarterly Sampling until 4 Years after Completion of Extended Pump Test				Rationale for Sampling after Baseline
		1Q	2Q ⁽¹⁾	3Q	4Q	
Monitoring Wells						
MW-01	WL	WL	WL	WL	WL	
MW-02	WL	WL	WL	WL	WL	
MW-03	WL	WL	WL	WL	WL	
MW-04	WL	WL	WL	WL	WL	
MW-04R ⁽²⁾	--	WL	WL	WL	WL	
MW-05	WL	WL	WL	WL	WL	
MW-06 ⁽²⁾	--	--	--	--	--	
MW-07	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	Upgradient well
MW-08	WL	WL	WL	WL	WL	
MW-09	WL	WL	WL	WL	WL	
MW-10	WL	WL	WL	WL	WL	
MW-11	WL	WL	WL	WL	WL	
MW-12	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	D5 Area (RD/RA)
MW-13	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	D9 Area (RD/RA)
MW-14 ⁽²⁾	WL	--	--	--	--	
MW-14R	--	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	D8 Area, replaced PW-06
MW-15	WL	WL	WL	WL	WL	
MW-16	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	D5 Area
MW-17	WL	WL	WL	WL	WL	
MW-18	WL	WL	WL	WL	WL	
MW-19	WL	WL	WL	WL	WL	
MW-101	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	D1 Area (RD/RA)
MW-102	WL, 12 PFCs	WL	WL, 12 PFCs	WL	WL	D1 Area (RD/RA)
MW-103	WL, 12 PFCs	WL	WL, 12 PFCs	WL	WL	D2 Area (RD/RA)
MW-104	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	D2 Area (RD/RA)
MW-105	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	D9 Area (RD/RA)
MW-108	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	WWTP Area (RD/RA)
MW-109	WL	WL	WL	WL	WL	
MW-110	WL, 12 PFCs	WL, 5 PFCs	WL, 12 PFCs	WL, 5 PFCs	WL, 5 PFCs	D5 Area (RD/RA)
MW-112	WL	WL	WL	WL	WL	
ECPZ-01	WL	WL	WL	WL	WL	East Cove Area
ECPZ-02R ⁽³⁾	WL	WL	WL	WL	WL	East Cove Area
ECPZ-03R ⁽³⁾	WL	WL	WL	WL	WL	East Cove Area
INF-PZ01 ⁽³⁾	WL	WL	WL	WL	WL	
Piezometer						
PZ-14	WL	WL	WL	WL	WL	
Production Wells						
PW-06 ⁽⁴⁾	WL ⁽⁵⁾ , 12 PFCs	WL ⁽⁵⁾	WL ⁽⁵⁾	WL ⁽⁵⁾	WL ⁽⁵⁾	D8 Area (RD/RA)
PW-09	WL ⁽⁵⁾ , 12 PFCs	WL ⁽⁵⁾ , 5 PFCs	WL ⁽⁵⁾ , 12 PFCs	WL ⁽⁵⁾ , 5 PFCs	WL ⁽⁵⁾ , 5 PFCs	D9 Area
PW-10	WL ⁽⁵⁾ , 12 PFCs	WL ⁽⁵⁾ , 5 PFCs	WL ⁽⁵⁾ , 12 PFCs	WL ⁽⁵⁾ , 5 PFCs	WL ⁽⁵⁾ , 5 PFCs	East Cove Area

Notes:

WL - Water level measurement.

WWTP - Wastewater Treatment Plant

12 PFCs - PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoA, PFBS, PFHS, and PFOS.

5 PFCs - PFBA, PFBS, PFHS, PFOS, and PFOA.

(1) The annual groundwater sampling event will be performed in 2Q sampling event.

(2) In May 2013, monitoring wells MW-06 and MW-14 were abandoned, and monitoring wells MW-04R, MW-14R, and MW-112 were installed.

(3) Re-classified as monitoring wells since they are registered with the Minnesota Department of Health (MDH) with unique well IDs.

(4) PW-06 is currently not in use; therefore, a sample cannot be collected from this well. Nearby monitoring well MW-14R has been added to the sampling program to replace PW-06.

(5) Water level measurement will be recorded if pump is not operating.



3. WATER MONITORING AND RESULTS

3.1 GROUNDWATER ELEVATIONS

Figure 3-1 shows the locations of the production wells, monitoring wells and piezometer on the Minnesota Geologic Survey (MGS) geologic map for the Site. Beneath the main plant area, the uppermost water-bearing unit is the Oneota Dolomite. Where the Oneota Dolomite is absent, the uppermost water-bearing unit occurs within the unconsolidated glaciofluvial sediments. As shown in Figure 3-1, the Oneota Dolomite (blue) is eroded in a significant area to the south and east of the Main Plant area where a bedrock valley (yellow and green) is present. Site production wells PW-05 and PW-05/PW-06, in addition to new production wells PW-09 and PW-10, are all screened within the unconsolidated sediments within the limits of the bedrock valley.

Depth-to-groundwater measurements were measured on a quarterly basis at the monitoring well and piezometer locations shown in Figure 2-1. Water level measurements recorded in groundwater monitoring wells are presented in Table 3-1. Hydrographs were constructed for select monitoring points, and are included in Attachment C. As discussed previously in Section 2.1, monitoring wells MW-04R, MW-14R and MW-112 were installed in May 2013, while monitoring wells MW-06 and MW-14 were deemed unusable due to well integrity issues and abandoned at the same time in accordance with MDH regulations.

Efforts were also completed in May 2013 to rehabilitate monitoring well MW-12 as the screened interval of this well is clogged. The rehabilitation efforts could not completely clear the screened interval of this well as it continues to pump dry during sampling events; however, the water level in this monitoring well recovers sufficiently within 24 hours to allow the collection of a representative groundwater sample. Therefore, monitoring well MW-12 continues to be included in the quarterly groundwater sampling program for the Site, but the water level recorded in this well is not used to construct groundwater elevation contours.

As shown in Table 3-1, the water levels in monitoring wells more distant from the Mississippi River (e.g. monitoring wells MW-01, MW-02, MW-03, MW-09, MW-17 and



MW-18) exhibited greater change in water level than monitoring wells closer to the river. These fluctuations in water levels are also shown in the hydrographs presented in Attachment C. Increases in water levels occur in response to significant precipitation events, and after the spring melt. Groundwater levels subsequently decline in response to lower recharge during the winter months.

The groundwater elevation data collected from the network of wells shown in Table 3-1 were used to prepare groundwater elevation contour maps. Figures 3-2 through 3-5 present groundwater elevation contour maps constructed for January, April, August, and December 2014. As shown in Figures 3-2 through 3-5, the direction of groundwater flow is consistently to the south toward the Mississippi River. A moderate hydraulic gradient of 0.009 (ft/ft) is calculated along the interpreted direction of groundwater flow from monitoring well MW-01 to the Mississippi River.

Areas of depression in the groundwater surface induced by the pumping of production wells PW-02, PW-05, PW-09 and PW-10 are evident in all of the groundwater elevation contour maps. As part of the extended pilot pump test, production well PW-09 was operating at 900 gpm on January 22, 2014. As shown in Figure 3-2, the effect of production well PW-09 operating at this high flow rate creates a larger cone of depression in the groundwater surface compared to the groundwater elevation contour maps for the other dates; however, the effect of continuous pumping of production wells PW-09 and PW-10 is also evident in the groundwater elevation maps constructed for April, August and December 2014.

3.2 EXTENDED PILOT PUMP TEST

The extended pilot pump test was performed at the Site from February 2013 through February 2014. The primary purpose of the pump test was to provide information to complete the design of a groundwater interception system in accordance with the approved RD/RA Plan and the Minnesota Decision Document (MDD) for the Site.

The current groundwater recovery network consists of Site production wells PW-05, PW-09 and PW-10. The extended pilot pump test consisted of continuing to pump production



well PW-05, and varying the flow rates at production wells PW-09 and PW-10 while collecting water level data at nearby monitoring wells. The water level data were used to calculate the area of groundwater capture zones induced by production wells PW-09 and PW-10 at various flow rates. The pump test was conducted in accordance with the approved pump test work plan (WESTON, 2013) and continued until sufficient operational data were collected to allow completion of the final design of the groundwater interception system with respect to the number, spacing and pumping rates for the production (extraction) well network.

The proposed final design of the groundwater interception system was determined using a combination of groundwater elevation data collected during the extended pilot pump test and performing predictive simulations using a groundwater flow model calibrated to Site hydrogeologic conditions. The results of this analysis indicated that the recommended groundwater interception system would consist of production well PW05/PW-06 (or a replacement), PW-09, PW-10 and one additional new well between production wells PW-5/PW-06 and PW-09. Details of this analysis were provided in a separate document that was submitted to the MPCA in March 2015 (WESTON, 2015).

3.3 GROUNDWATER QUALITY

Quarterly groundwater sampling events were performed in January, July and October 2014. The annual groundwater sampling event was performed in April 2014. Table 3-2 contains a summary of groundwater PFC analytical data for the groundwater sampling performed since the October 2012 baseline sampling event. Laboratory analytical packages and chain-of-custody documentation for the 2014 sampling events are included in Attachment D.

PFBA, PFOA and PFOS concentrations for select wells are plotted on trend graphs and provided in Attachment E. The PFC analytical data were evaluated by applying the Mann-Kendall trend test (at an $\alpha = 0.05$ significance level) to PFBA, PFOA, PFOS and PFHS concentrations for those Site monitoring wells where sufficient historical groundwater analytical data are available (i.e. $n \geq 5$). The Mann-Kendall trend test is a non-parametric statistical procedure that is used for analyzing trends in data over time (Gilbert, 1987).



Nonparametric methods require no assumptions regarding the underlying statistical distribution of the data. The outcome of the procedure depends on the ranking of individual data points and not the overall magnitude of the data points. The Mann-Kendall procedure can be used for data sets that include irregular sampling intervals, data below the detection limit, and trace or missing data. The method may be applied to track data trends for purpose of groundwater compliance monitoring, site assessment, and monitoring of the performance of groundwater corrective actions (USEPA, 2009). Mann-Kendall test outcomes consist of the identification of statistically significant increasing trends, no statistically significant trend or a statistically significant decreasing trend at the specified significance level.

Table 3-3 provides a summary of the results of the Mann-Kendall analysis, and a discussion of the groundwater analytical results is provided in the following subsections. The discussion of groundwater analytical results is organized by the location of wells by Site area and focused primarily on the PFBA, PFOS and PFOA results. However, the analytical results for other PFCs are discussed where pertinent.

3.3.1 Background

Table 3-2 provides a summary of background groundwater analytical results collected from monitoring well MW-07. As shown on Figure 2-1, monitoring well MW-07 is located to the northeast of the Cottage Grove Plant near Highway 61.

The groundwater analytical data for the period October 2012 through October 2014 indicates PFBA, PFOA and PFOS are consistently detected in the samples collected from monitoring well MW-07. PFBS, PFHA, PFHpA, PFHS and PFPeA were also detected. The Mann-Kendall results presented in Table 3-3 indicate a statistically significant increasing trend for PFOS, PFOA, PFBS and PFHS, while no statistically significant trend was identified for PFBA in the period October 2012 through October 2014. Groundwater elevation data for the Site have consistently shown that monitoring well MW-07 is hydraulically upgradient of the plant and the former disposal areas on Site (see Figures 3-2



through 3-5). 3M is not aware of the source for PFCs in groundwater at this upgradient location.

3.3.2 D1/D2 Area

Soil excavation activities were performed from December 2009 through May 2010 in the D1/D2 Area that included the removal, and off-site disposal at a permitted facility, of approximately 15,000 cubic yards (yd³) of PFC-containing soil. Wells MW-101, MW-102, MW-103 and MW-104 monitor groundwater conditions near the D1/D2 Area. Monitoring well MW-112 was installed to the east of the D1/D2 Area in May 2013. Although not included in the list of monitoring wells in the long-term sampling program for the Site, a groundwater sample was collected from newly installed monitoring well MW-112 during the July 2013 sampling event to provide baseline PFCs groundwater quality data.

As shown in Table 3-2, PFBA, PFBS, PFOA, PFHA, PFHpA, PFHS, PFNA, PFOA, PFOS and PFPeA are consistently detected in groundwater samples collected from monitoring wells MW-101, MW-102, MW-103 and MW-104. In accordance with the Groundwater Sampling Plan, quarterly groundwater samples are collected from monitoring wells MW-101 and MW-104, while monitoring wells MW-102 and MW-103 are sampled annually. Detected PFC concentrations are generally highest in MW-101. PFBA and PFHS concentrations are higher in MW-101 and MW-102 (D1 Area) compared to the results for monitoring wells MW-103 and MW-104 (D2 Area). PFOA concentrations in the period October 2012 through October 2014 ranged from 35.1 µg/L (MW-104) to 147 µg/L (MW-101). PFOS concentrations are consistently lowest in monitoring well MW-103 (1.55 to 7.82 µg/L), while the PFOS results for monitoring wells MW-101, MW-102 and MW-104 were comparable (107 to 250 µg/L).

The Mann-Kendall trend test indicated no statistically significant trend for PFOS, PFOA, PFBA or PFHS for monitoring well MW-104 (Table 3-3). A statistically significant decreasing PFOS, PFOA, PFBA and PFHS trend was identified in monitoring well MW-101, while a statistically significant decreasing PFBS trend was determined for monitoring well MW-104. A statistically significant increasing PFBS trend was determined for



monitoring well MW-101, although PFBS concentrations have fluctuated in a relatively narrow range of 19.5 to 37.8 µg/L. Insufficient data is currently available to establish a trend applying the Mann-Kendall test to PFCs detected in monitoring wells MW-102 and MW-103. A cursory review of the results in Table 3-2 for monitoring wells MW-102 and MW-103 indicates that the PFC concentrations fluctuate with no discernible trend.

3.3.3 D9 Area

In the D9 Area, soil excavation activities were performed from May through August 2010 with the removal, and off-site disposal at a permitted facility, of approximately 8,400 yd³ of soil. Wells MW-105 and MW-13 monitor groundwater quality conditions near the D9 Area. The continued pumping of production well PW-09 is remediating PFCs in groundwater in the D9 Area (and a portion of the WWTP and D1/D2 Areas).

A comparison of the PFC analytical data summarized in Table 3-2 for monitoring wells MW-13 and MW-105 indicates higher PFC concentrations in monitoring well MW-105 compared to MW-13. Monitoring well MW-105 is immediately downgradient of the D9 Area while monitoring well MW-13 is slightly to the west of the limits of the D9 Area.

A visual inspection of the PFC results for production well PW-09 in Table 3-2 suggests an increase in PFC concentrations at this pumping center, and the Mann-Kendall trend test identifies a statistically significant increasing trend in PFOS and PFOA concentration for production well PW-09. As shown in Table 3-3, a statistically significant decreasing trend was identified for PFOA and PFBS for monitoring well MW-105. No statistically significant trend was identified for the remaining PFCs for the Mann-Kendall analysis performed for monitoring wells MW-105 and production well PW-09. Further, no statistically significant trend in PFCs was identified for monitoring well MW-13.

3.3.4 WWTP Area

Monitoring well MW-108 is included in the quarterly groundwater sampling program to provide groundwater quality data downgradient of the Site waste water treatment plant (WWTP) area. As shown in the PFC analytical data provided in Table 3-2 and trend graph presented in Attachment E, PFC concentrations continue to fluctuate in monitoring well



MW-108 with a temporary increase in PFCs in July 2014. Prior to July 2014, PFC concentrations showed a general decline over time. The Mann-Kendall trend analysis for monitoring well MW-108 indicated a statistically significant decreasing trend in PFHS and no statistically significant trend for the remaining PFCs analyzed.

3.3.5 D5 Area

Monitoring wells MW-110, MW-12 and MW-16 provide groundwater quality and elevation data for the D5 Area. Monitoring well MW-12 is located within the limits of the D5 Area, while monitoring wells MW-110 and MW-16 are located immediately south to southeast (downgradient) of the D5 Area (see Figure 2-1). As shown in Table 3-2, prior to 2014, PFBA, PFOS and PFOA concentrations for monitoring well MW-12 are higher compared to monitoring wells MW-110 and MW-16. Comparing the PFC results for 2014 to previous PFC data for D5 area monitoring wells, PFC concentrations have decreased for monitoring well MW-12 while an increase in some PFCs (e.g. PFOA) were apparent in monitoring wells MW-110 and MW-16.

The Mann-Kendall trend test provides the following results:

- Monitoring well MW-12: Statistically significant decreasing trend for PFOS, PFOA, PFBA, PFBS and PFHS.
- Monitoring well MW-110: Statistically significant decreasing trend for PFOS. Statistically significant increasing trend in PFOA, PFBA, PFBS and PFHS.
- Monitoring well MW-16: No statistically significant trend for PFOS or PFBA. A statistically significant increasing trend for PFOA, PFBS and PFHS.

In the final design of the groundwater interception system for the Site, discussed in subsection 3.2, an additional production (recovery) well is recommended to be installed between existing production wells PW-05/PW-06 and PW-09.

3.3.6 D8 Area

As discussed in Section 2.1, quarterly groundwater samples were to be collected from production well PW-05/PW-06. However, due to a declining yield, the use of production well PW-05/PW-06 was scaled back in March 2013 to intermittent use. To compensate for



the reduction in flow of production well PW-06, the plant water supply demands were met by pumping nearby production well PW-05 at a higher average flow rate. In 2014, production well PW-05 operated at an average flow rate of 916 gpm, which represents the highest flow rate for all the Site production wells. Monitoring well MW-14R is located between production wells PW-05 and PW-06 to the southwest (hydraulically downgradient) of the D8 Area. Following MPCA approval in January 2014, quarterly groundwater samples were collected from monitoring well MW-14R, as a replacement monitoring point for production well PW-06.

A review of the PFC analytical data provided in Table 3-2, and trend graphs provided in Attachment E, for monitoring well MW-14R visually indicates an increasing trend in PFC concentrations. However, as shown in Table 3-3, the Mann-Kendall analysis identified no statistically significant trend for PFCs except for a statistically significant increasing trend for PFBS.

3.3.7 East Cove Area

Remedial activities in the East Cove area included the removal, and off-site disposal at a permitted landfill, of approximately 16,500 yd³ of sediment. These activities were performed from August 2011 through January 2012. Production well PW-10 was installed to contain and remove groundwater potentially impacted with PFCs in the East Cove area. Table 3-2, and the trend graphs provided in Attachment E, present the groundwater analytical results through October 2014 for production well PW-10. A cursory review of the PFC analytical results for PW-10 indicates generally stable concentrations. This observation is confirmed by the Mann-Kendall trend test that indicated no statistically significant trends for the PFCs analyzed except for PFHS where a statistically significant decreasing trend was identified.

3.4 PORE WATER/SURFACE WATER QUALITY

As described in the *Groundwater, Pore Water, and Surface Water Sampling Plan for Perfluorochemicals (PFCs)* (Sampling Plan) submitted to the MPCA in April 2012, three pore water/surface water sampling events were conducted at the same locations where



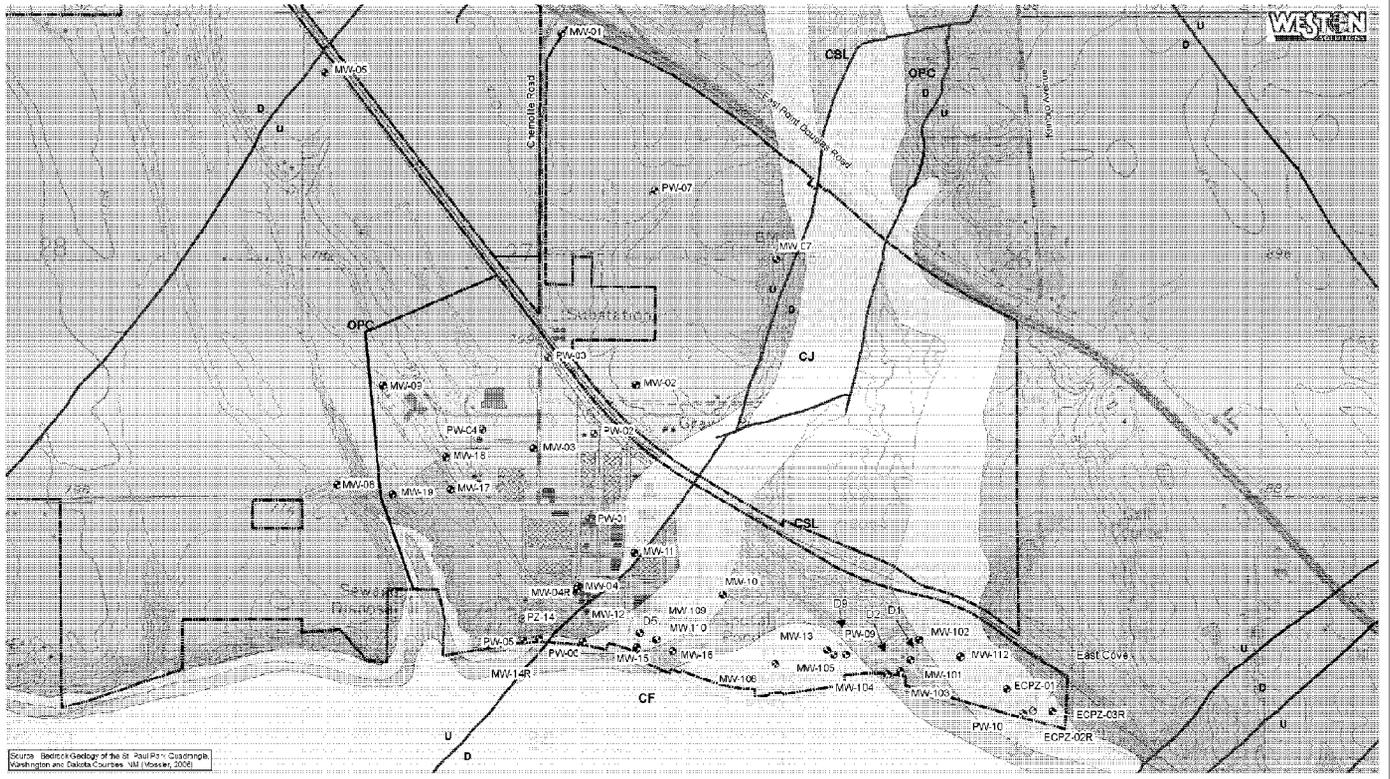
sampling was performed previously in September/October 2006 and June 2011. The additional three sampling events were conducted prior to (October 2012), during (August 2013), and after (September/October 2014) the extended pilot pump test. These pore water sampling events complete the pore water and surface sampling commitment made to MPCA in the Sampling Plan.

The laboratory PFC analytical data package for the September/October 2014 sampling event is provided in Attachment F. A summary of the pore water and surface water analytical results from the pre-, mid-, and post extended pilot pump test sampling events are provided in Table 3-4. The PFC analytical results for the September/October 2006 and June 2011 sampling events for the same locations are also provided in Table 3-4 for comparison purposes. The pore water and surface water sampling locations are provided in Figure 2-2. As shown in Figure 2-2, the pore water and surface water transects are located downgradient of the following Site areas:

- Transect IW-09: downgradient of the former D5 Area
- Transect IW-14: downgradient of the WWTP Area
- Transect IW-19: downgradient of the former D1/D2 Area
- Transect IW-25: downgradient of the East Cove Area.

The following observations for the pore water and surface water data presented in Table 3-4 are provided:

- PFC concentrations in pore water are higher than surface water PFC concentrations.
- Locations closer to the shoreline generally had higher pore water concentrations than locations more distant from shore.
- Pore water PFC concentrations fluctuate over time with no discernible trend.
- Where detected, PFCs in surface water have fluctuated but have generally declined in the reporting period provided in Table 3-4.



Source: Bedrock Geology of the St. Paul River Drainage, Washington and Idaho Counties, Fall 1998, 2000.

- Legend**
- Monitoring Well Locations
 - Production Well Locations
 - Piezometer Well Locations
 - Approximate Cottage Grove Site Boundary
 - Fault - Letters represent vertical displacement: U - Up, or D - Down
 - Fault - Line indicates aerial trace of anticline or syncline
 - SL Lawrence Formation
 - P. de la Cruz Group
 - Franciscan Formation
 - Jordan Sandstone

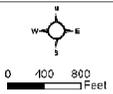


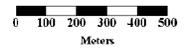
Figure 3-1
Well Locations
and Geologic Map
Cottage Grove Site

File Y:\Cottage Grove\mnd\shl loc Geology from Part.mxd, 5/6/2014 2:07:58 PM, John



Legend

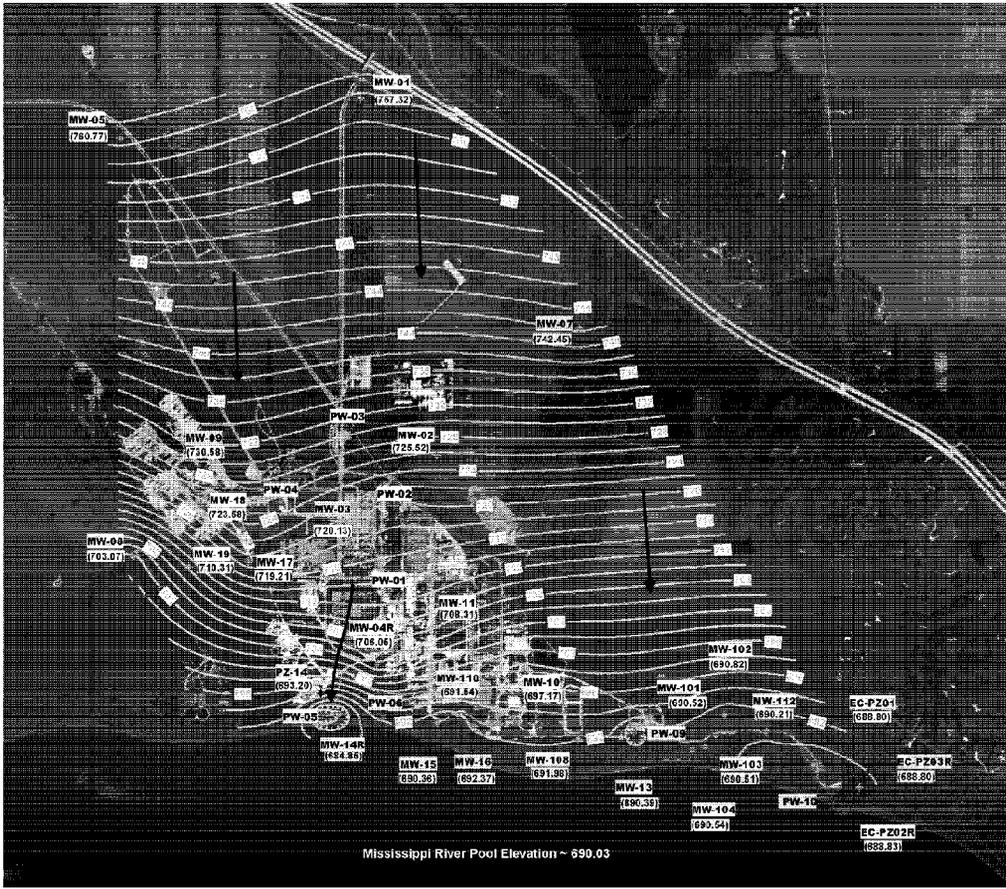
- ◆ Production Well Location (PW)
- ⊕ Monitoring Well Location (MW/ECPZ)
- ◆ Piezometer Location (PZ)
- Groundwater Elevation Contour (contour interval 2 feet)
- 741.92 Groundwater Elevation (ft MSL)
- Groundwater Flow Direction

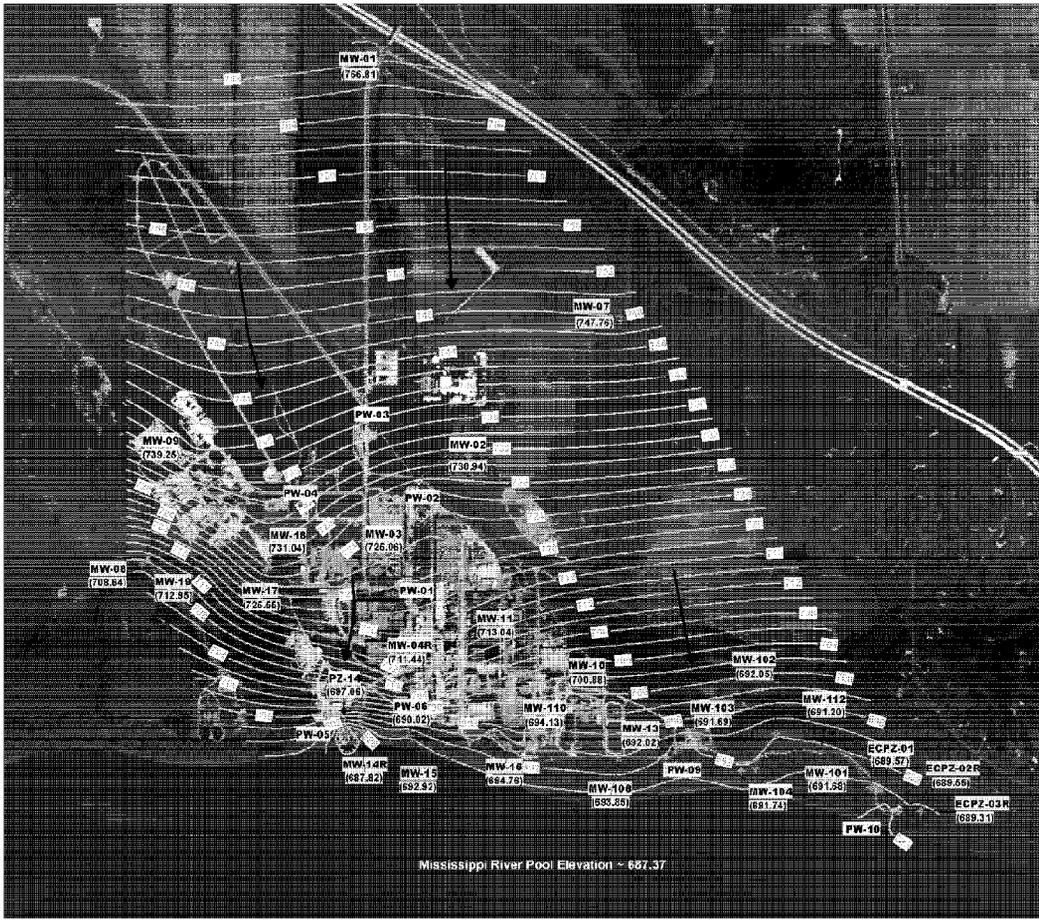


Note: Groundwater Elevations collected from MW-12 and MW-105 were not used to construct groundwater elevation contours. MW-12 has well integrity issues, and MW-105 is screened in a separate water-bearing zone.



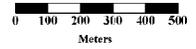
Figure 3-2
Groundwater Elevation Contour Map
22 January 2014
PW-02, PW-05 and PW-09 operating
Cottage Grove Site
Cottage Grove, Minnesota





Legend

- + Pumping Well Location (PW)
- ◆ Monitoring Well Location (MW/ECPZ)
- ◆ Piezometer Location (PZ)
- Groundwater Elevation Contour (contour interval: 2 ft)
- (747.76) Groundwater Elevation (± MSL)
- ↗ Groundwater Flow Direction

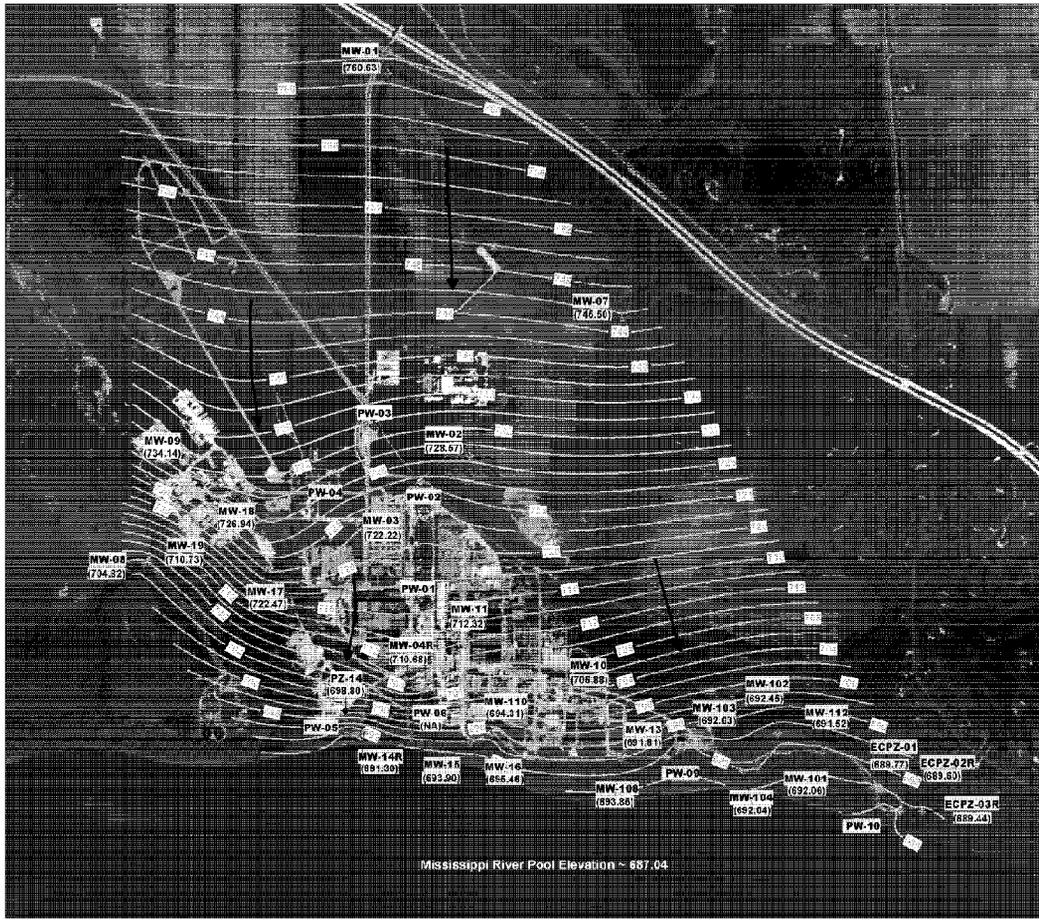


Note: Groundwater Elevations collected from MW-12 and MW-105 not used to construct groundwater elevation contours. MW-12 has well integrity issues and MW-105 is screened in separate flow-bearing zone than other wells in area.

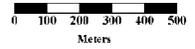
Figure 3-4

Groundwater Elevation Contour Map
6 August 2014
PW-02, PW-05, PW-09 and PW-10 Operating
Cottage Grove Facility
Cottage Grove, Minnesota

3M_MN01595985



- Legend**
- ◆ Pumping Well Location (PW)
 - ◇ Monitoring Well Location (MW/ECPZ)
 - ◆ Piezometer Location (PZ)
 - Groundwater Elevation Contour (contour interval: 2 ft)
 - [746.50] Groundwater Elevation (ft MSL)
 - ↗ Groundwater Flow Direction



Note: Groundwater Elevations collected from MW-12 and MW-165 not used to construct groundwater elevation contours. MW-12 has well integrity issues and MW-165 is screened in separate water-bearing zone than other wells in area.

Figure 3-5
Groundwater Elevation Contour Map
29 December 2014
PW-02, PW-05, PW-09 and PW-10 Operating
Cottage Grove Facility
Cottage Grove, Minnesota



Table 3-1
Depth-to-Groundwater and Groundwater Elevation Data
October 2012 - December 2014
Cottage Grove Site, Cottage Grove, Minnesota

Well ID	TOC Elevation (ft MSL)	Oct-12		Feb-13		Apr-13		Jul-13		Oct-13	
		Depth to Groundwater (ft btoe)	Groundwater Elevation (ft MSL)	Depth to Groundwater (ft btoe)	Groundwater Elevation (ft MSL)	Depth to Groundwater (ft btoe)	Groundwater Elevation (ft MSL)	Depth to Groundwater (ft btoe)	Groundwater Elevation (ft MSL)	Depth to Groundwater (ft btoe)	Groundwater Elevation (ft MSL)
MW-01	822.02	65.12	756.90	67.91	754.11	66.01	755.01	59.82	762.20	62.49	759.53
MW-02	805.32	86.37	720.55	86.08	719.84	83.43	722.49	79.67	726.25	81.41	724.51
MW-03	810.07	100.41	709.66	99.71	710.36	93.09	716.98	89.96	720.11	91.72	718.35
MW-04	806.80	105.59	699.91	105.45	700.45	104.62	702.28	101.42	705.48	101.92	704.98
MW-04R	805.18	NA	NA	NA	NA	NA	NA	99.46	705.72	100.00	705.18
MW-05	808.18	60.10	758.08	52.62	756.20	48.35	760.46	45.17	763.64	46.68	760.15
MW-07	794.08	53.91	740.17	55.03	739.05	53.66	740.42	50.55	743.53	52.16	741.92
MW-06	765.97	63.41	702.56	65.32	700.65	63.96	701.99	60.49	705.48	62.16	703.79
MW-06	760.17	63.00	716.17	48.31	720.86	42.65	726.52	36.51	733.68	38.68	730.59
MW-1C	788.28	91.70	696.58	92.03	696.25	91.92	696.36	90.93	697.35	91.56	696.72
MW-11	804.34	102.35	701.99	101.22	703.12	96.50	705.84	96.65	707.69	96.96	707.35
MW-12	781.03	90.75	691.18	99.81	682.12	112.78	689.15	93.50	698.43	98.84	683.00
MW-13	783.12	90.76	692.36	91.22	691.90	93.61	689.51	92.67	690.45	94.03	689.09
MW-14R	703.89	NA	NA	NA	NA	NA	NA	19.97	683.92	19.77	684.12
MW-15	783.64	93.45	690.19	93.60	690.04	93.35	690.29	93.21	690.43	93.46	690.15
MW-16	784.2	91.55	692.65	91.95	692.26	92.03	692.18	91.77	692.44	92.16	692.05
MW-17	785.16	77.37	707.79	76.44	708.72	70.13	715.03	65.18	719.98	67.03	718.13
MW-16	762.93	73.15	709.70	71.00	711.93	63.63	719.30	59.00	724.93	58.41	724.52
MW-15	757.35	51.20	706.16	52.55	704.81	50.62	706.74	47.24	710.12	47.37	709.99
PZ-14	755.95	61.57	694.39	64.31	691.65	63.41	692.55	62.36	693.60	63.03	692.93
MW-10I	765.04	80.92	684.82	84.35	681.49	85.56	680.26	85.27	680.57	85.02	680.02
MW-102	783.25	90.93	692.32	91.42	691.83	92.65	690.90	92.36	690.89	92.90	690.35
MW-103	770.73	78.85	691.88	79.26	691.47	80.52	690.21	80.17	690.55	80.72	690.01
MW-104	771.94	80.82	691.12	80.45	691.49	81.74	690.20	81.36	690.59	82.00	689.94
MW-105	782.06	88.22	693.84	89.00	693.06	89.94	692.12	89.67	692.39	90.10	691.95
MW-103	787.61	94.62	692.99	95.06	692.55	96.01	691.60	95.63	691.98	96.19	691.42
MW-109	782.95	43.22	739.73	43.90	739.05	42.82	740.13	42.11	740.84	42.84	740.11
MW-110	783.03	91.49	691.54	91.87	691.15	91.49	691.54	91.32	691.71	91.70	691.33
MW-112	783.02	NA	NA	NA	NA	NA	NA	92.77	690.25	93.32	689.70
ECPZ-01	762.92	72.87	690.05	73.05	689.87	74.51	688.41	74.15	688.77	74.76	688.16
ECPZ-02R	693.3	3.00	690.31	3.20	690.11	4.87	688.44	4.37	688.94	5.16	688.13
ECPZ-03R	690.70	1.17	689.53	1.22	689.48	2.10	688.60	1.68	688.81	2.31	688.39
INF-PZC1	NA	36.03	NC	37.65	NC	37.82	NC	34.77	NC	36.73	NC

Note: Monitoring wells MW-04R, MW-14R, and MW-112 were installed in May 2013.
ft MSL - feet above mean sea level.
ft btoe - feet below top of casing.
NA - Not available.

3M_MN01595987



Table 3-1 (cont'd)
 Depth-to-Groundwater and Groundwater Elevation Data
 October 2012 - December 2014
 Cottage Grove Site, Cottage Grove, Minnesota

Well ID	TCC Elevation (ft MSL)	Jan-14		Apr-14		Aug-14		Dec-14		Groundwater Elevation Data			
		Depth to Groundwater (ft btoc)	Groundwater Elevation (ft MSL)	Depth to Groundwater (ft btoc)	Groundwater Elevation (ft MSL)	Depth to Groundwater (ft btoc)	Groundwater Elevation (ft MSL)	Depth to Groundwater (ft btoc)	Groundwater Elevation (ft MSL)	Minimum	Maximum	Range	Standard Deviation
NW-01	822.02	64.40	757.52	64.70	757.32	55.21	763.81	61.39	760.63	754.11	766.81	12.70	3.81
NW-02	805.92	82.70	725.22	80.40	725.52	74.58	733.54	77.95	728.57	719.84	730.34	11.10	3.58
NW-03	810.07	50.85	749.22	49.94	750.13	85.01	723.08	87.85	722.22	709.68	726.06	15.40	5.09
NW-04	803.90	101.61	705.29	101.10	705.80	95.57	711.23	96.47	710.43	699.91	711.23	11.32	3.92
MW-04R	805.18	59.66	705.52	59.13	706.05	59.74	711.44	54.52	710.66	705.19	711.44	6.26	2.83
NW-05	803.18	53.23	759.58	48.04	759.77			47.70	751.11	756.23	763.01	6.72	2.08
NW-07	784.08	52.09	741.99	51.63	742.45	46.32	747.75	46.98	745.90	739.05	747.76	8.71	2.74
NW-08	765.97	63.09	702.89	62.90	703.07	57.33	703.64	61.15	704.82	700.65	708.64	7.99	2.33
NW-09	789.17	39.77	730.40	38.80	730.68	20.02	733.25	35.03	734.14	716.17	730.25	23.08	7.04
NW-10	788.28	91.80	696.48	91.11	697.17	87.40	703.88	82.40	705.88	696.25	705.88	9.63	3.22
NW-11	804.34	95.90	707.44	96.03	706.31	91.30	713.04	92.02	712.32	701.99	713.04	11.05	3.66
NW-12	781.93	NA	885.15	891.8	22.03	8.50							
NW-13	783.12	95.25	687.87	92.73	690.39	91.10	692.02	91.31	691.81	687.87	692.36	4.49	1.55
MW-14R	703.89	19.02	684.87	19.04	684.85	16.07	687.83	12.60	691.30	683.82	691.30	7.38	2.89
NW-15	783.64	53.80	689.84	53.28	690.36	50.72	692.92	49.74	693.90	689.84	693.90	4.06	1.45
NW-16	784.21	52.52	691.69	51.84	692.37	49.45	694.76	48.75	695.46	691.69	695.46	3.77	1.31
NW-17	785.16	65.21	718.95	65.95	719.21	59.61	723.55	62.69	722.47	707.79	725.55	17.76	5.90
NW-18	702.93	59.54	723.39	59.35	723.50	51.89	731.04	55.99	726.94	706.79	731.04	21.26	6.92
NW-19	757.36	45.98	710.39	47.05	710.31	44.41	712.95	46.83	710.73	704.81	712.95	8.14	2.62
PZ-14	755.96	62.85	693.11	62.76	693.20	58.50	697.09	57.16	698.80	691.65	698.80	7.15	2.31
MW-101	789.04	55.89	668.05	55.32	669.32	54.16	691.09	53.70	692.06	608.05	692.06	2.21	0.66
MW-102	783.25	93.16	690.09	92.43	690.82	91.20	692.05	90.80	692.45	690.09	692.45	2.36	0.90
MW-103	770.73	80.97	689.75	80.22	690.51	79.04	691.99	78.70	692.03	689.75	692.03	2.27	0.87
MW-104	771.94	82.29	689.65	81.40	690.54	80.20	691.74	79.90	692.04	689.65	692.04	2.38	0.83
MW-105	782.06	80.65	691.41	80.80	692.26	80.26	693.80	87.56	694.50	691.41	694.50	3.09	1.04
MW-106	787.61	65.79	690.82	65.63	691.98	63.78	693.85	63.30	694.31	690.82	694.31	3.49	1.15
MW-109	782.95	43.22	739.73	42.47	740.48	42.21	743.74	43.25	739.70	739.05	740.84	1.79	0.57
MW-110	783.03	82.00	691.03	91.49	691.54	88.50	694.13	88.71	694.52	691.03	694.52	3.29	1.26
MW-112	783.02	93.09	689.94	92.81	690.21	91.82	691.20	91.50	691.52	689.73	691.52	1.82	0.73
ECFZ-01	782.92	74.01	688.91	74.12	688.80	73.35	693.57	73.15	699.77	688.19	690.05	1.86	0.68
ECFZ-02R	693.31	-4.07	689.24	4.48	686.83	3.76	683.55	3.71	689.60	688.13	690.31	2.18	0.73
ECFZ-03R	690.70	1.70	688.00	1.50	686.80	1.39	683.31	1.26	689.44	688.38	689.53	1.14	0.42
INF-P201	NA	33.23	NA	NC	NC	3.26	--						

Note: Monitoring wells MW-04R, MW-101, MW-102, MW-103, MW-104, MW-105, MW-106, MW-109, MW-110, MW-112, ECFZ-01, ECFZ-02R, ECFZ-03R are installed in the upper aquifer.
 ft MSL - feet above mean sea level.
 ft btoc - feet below top of casing.
 NA - Not available.

3M Confidential - Not for Distribution

3M_MN01595988



Table 3-2
Summary of Groundwater PFC Analytical Data
October 2012 - October 2014
Cottage Grove Site, Cottage Grove, MN

Well ID	Location	Date	PFC Groundwater Results (ppb, µg/L)											
			PFBA	PFBS	PFDA	PFDoA	PFHA	PFHpA	PFHS	PFNA	PFOA	PFOS	PFPeA	PFUnA
MW-07	Background	10/15/2012	2.55	< 0.0250	< 0.0250	< 0.0250	0.076	0.025	< 0.0500	< 0.0500	0.298	0.026	0.165	< 0.0250
		1/29/2013	2.09	< 0.0250	-	-	-	-	< 0.0250	-	0.259	0.044	-	-
		4/22/2013	2.61	0.027	< 0.0250	< 0.0250	0.064	0.265	< 0.0250	< 0.0250	0.240	0.046	0.153	< 0.0250
		7/23/2013	1.71	0.040	-	-	-	-	0.026	-	0.303	0.241	-	-
		10/28/2013	3.07	0.029	-	-	-	-	< 0.0250	-	0.428	0.104	-	-
		1/13/2014	2.88	< 0.100	-	-	-	-	0.045	-	0.390	0.104	-	-
		4/23/2014	2.48	0.036	< 0.0250	< 0.0250	0.078	< 0.0250	< 0.0250	< 0.0250	0.344	0.049	0.153	< 0.0250
		7/16/2014	3.05	0.0762	-	-	-	-	0.0416	-	0.602	0.237	-	-
10/28/2014	3.01	0.070	-	-	-	-	0.050	-	0.581	0.191	-	-		
MW-101	D1/D2	10/18/2012	1860	23.9	0.424	< 0.0250	121	101	427	11.6	147	154	98.9	< 0.0250
		1/30/2013	1630	25.2	-	-	-	-	455	-	124	206	-	-
		4/23/2013	1830	19.5	0.365	< 0.0250	85.4	85.6	459	91.1	121	188	96.4	0.046
		7/25/2013	1680	24.0	-	-	-	-	464	-	112	189	-	-
		10/28/2013	1020	25.5	-	-	-	-	314	-	86.7	158	-	-
		1/15/2014	1510	25.4	-	-	-	-	398	-	79.4	159	-	-
		4/25/2014	1330	37.8	0.241	< 0.0250	61.1	62.3	364	6.96	76.0	135	72.2	< 0.0250
		7/17/2014	1460	29.3	-	-	-	-	480	-	83.9	193	-	-
10/29/2014	1410	29.6	-	-	-	-	452	-	74.8	170	-	-		
MW-102	D1/D2	10/18/2012	648	5.52	0.262	< 0.0250	14.8	9.52	129	3.2	42.2	107	18.2	< 0.0250
		4/25/2013	621	4.57	0.345	< 0.0250	18.7	13.3	401	12.0	62.0	250	23.2	0.075
		4/25/2014	919	7.12	0.136	< 0.0250	28.8	16.0	147	1.87	58.1	67.4	30.7	< 0.0250
MW-103	D1/D2	10/18/2012	226	7.16	0.036	< 0.0250	54.9	29.7	26.2	0.045	115	4.02	25.5	< 0.0250
		4/25/2013	241	7.01	< 0.0250	< 0.0250	49.5	25.6	24.7	8.01	126	1.55	26.7	< 0.0250
		4/25/2014	241	6.51	< 0.0250	< 0.0250	57.4	30.9	21.8	0.053	121	7.82	28.2	< 0.0250



Table 3-2 (cont'd)
Summary of Groundwater PFC Analytical Data
October 2012 - October 2014
Cottage Grove Site, Cottage Grove, MN

Well ID	Location	Date	PFC Groundwater Results (ppb, µg/L)											
			PFBA	PFBS	PFDA	PFDoA	PFHA	PFHpA	PFHS	PFNA	PFOA	PFOS	PFPeA	PFUnA
MW-104	D1/D2	10/18/2012	36.0	12.6	2.78	<0.0250	3.89	2.51	8.69	2.26	42.0	127	3.10	0.052
		1/30/2013	36.3	11.4	-	-	-	-	9.07	-	44.8	201	-	-
		4/25/2013	74.7	9.75	2.90	<0.0250	12.1	7.11	13.4	10.8	60.7	185	7.64	0.196
		7/25/2013	54.3	8.16	-	-	-	-	11.8	-	74.7	176	-	-
		10/28/2013	53.6	7.95	-	-	-	-	10.1	-	72.5	165	-	-
		1/15/2014	202	7.50	-	-	-	-	14.2	-	35.1	4.25	-	-
		4/25/2014	107	8.10	3.26	<0.0250	19.7	9.11	12.6	2.59	82.1	234	12.6	0.056
		7/17/2014	47.9	8.68	-	-	-	-	8.95	-	76.5	233	-	-
10/29/2014	30.9	7.72	-	-	-	-	6.24	-	50.0	177	-	-		
MW-112	D1/D2	7/24/2013	4.42	0.166	-	-	-	-	0.367	-	1.18	0.96	-	-
MW-105	D9	10/19/2012	52.7	9.46	0.991	<0.0250	16.2	14.7	18.9	0.836	98.3	120	6.11	<0.0250
		1/30/2013	54.4	8.03	-	-	-	-	21.1	-	119	129	-	-
		4/25/2013	57.0	5.72	0.683	<0.0250	11.4	9.64	16.0	9.22	104	95.0	5.13	0.116
		7/25/2013	71.6	6.49	-	-	-	-	6.35	-	70.6	92.3	-	-
		10/28/2013	38.9	4.98	-	-	-	-	6.01	-	87.2	467	-	-
		1/14/2014	55.6	6.15	-	-	-	-	12.6	-	68.3	152	-	-
		4/25/2014	71.0	7.68	0.334	<0.0250	11.8	8.64	8.59	0.583	44.3	58.7	6.05	<0.0250
		7/17/2014	39.2	4.89	-	-	-	-	6.65	-	27.7	36.2	-	-
10/29/2014	46.3	4.01	-	-	-	-	18.9	-	92.8	73.5	-	-		



Table 3-2 (cont'd)
Summary of Groundwater PFC Analytical Data
October 2012 - October 2014
Cottage Grove Site, Cottage Grove, MN

Well ID	Location	Date	PFC Groundwater Results (ppb, µg/L)											
			PFBA	PFBS	PFDA	PFDoA	PFHA	PFHpA	PFHS	PFNA	PFOA	PFOS	PFPeA	PFUnA
MW-13	D9	10/17/2012	7.10	0.619	0.040	<0.0250	0.330	0.101	0.487	0.060	3.33	3.85	0.544	<0.0250
		1/29/2013	5.60	0.560	-	-	-	-	0.396	-	2.56	3.92	-	-
		4/24/2013	9.34	0.972	0.033	<0.0250	0.227	0.116	0.931	0.050	5.46	4.05	0.569	<0.0250
		7/23/2013	8.30	1.07	-	-	-	-	1.05	-	6.17	4.02	-	-
		10/28/2013	6.48	0.571	-	-	-	-	0.366	-	4.01	1.40	-	-
		1/14/2014	9.80	1.20	-	-	-	-	0.969	-	9.78	3.08	-	-
		4/24/2014	7.14	0.752	<0.0250	<0.0250	0.257	0.078	0.556	0.045	8.63	2.33	0.465	<0.0250
		7/16/2014	8.08	0.973	-	-	-	-	0.713	-	14.4	4.48	-	-
10/28/2014	9.64	1.09	-	-	-	-	0.720	-	10.0	4.89	-	-		
PW-09	D9	4/25/2013	3.72	<0.0250	<0.0250	<0.0250	0.0627	<0.0250	<0.0250	<0.0250	0.223	0.324	0.191	<0.0250
		6/19/2013 ⁽¹⁾	-	-	-	-	-	-	-	-	0.462	0.376	-	-
		7/24/2013	2.95	0.058	-	-	-	-	0.040	-	0.624	0.622	-	-
		8/29/2013 ⁽¹⁾	-	-	-	-	-	-	-	-	0.605	0.818	-	-
		9/30/2013 ⁽¹⁾	-	-	-	-	-	-	-	-	0.873	1.38	-	-
		10/28/2013	4.53	0.137	-	-	-	-	0.076	-	1.02	1.52	-	-
		1/15/2014	4.76	0.173	-	-	-	-	0.109	-	1.33	2.13	-	-
		4/25/2014	4.14	0.148	<0.0250	<0.0250	0.135	0.0471	0.084	0.037	1.05	1.60	0.272	<0.0250
		7/17/2014	4.32	0.155	-	-	-	-	0.081	-	1.16	1.79	-	-
10/30/2014	3.87	0.106	-	-	-	-	0.054	-	0.701	0.838	-	-		



Table 3-2 (cont'd)
Summary of Groundwater PFC Analytical Data
October 2012 - October 2014
Cottage Grove Site, Cottage Grove, MN

Well ID	Location	Date	PFC Groundwater Results (ppb, µg/L)											
			PFBA	PFBS	PFDA	PFDoA	PFHA	PFHpA	PFHS	PFNA	PFOA	PFOS	PFPeA	PFUnA
MW-108	WWTP	10/18/2012	155	30.5	0.615	<0.0250	31.1	17.6	20.7	2.76	550	55.2	25.4	<0.0250
		1/30/2013	101	26.5	-	-	-	-	11.1	-	348	45.5	-	-
		4/25/2013	42.5	16.1	0.470	<0.0250	6.90	4.50	4.95	83.2	201	37.3	7.43	0.049
		7/24/2013	38.0	14.6	-	-	-	-	4.94	-	202	26.8	-	-
		10/28/2013	51.7	21.6	-	-	-	-	4.47	-	218	33.2	-	-
		1/14/2014	51.6	17.0	-	-	-	-	4.13	-	169	33.5	-	-
		4/25/2014	65.4	17.4	0.562	<0.0250	10.2	4.15	3.58	1.26	164	43.3	11.5	<0.0250
		7/17/2014	222	60.7	-	-	-	-	11.3	-	522	58	-	-
10/29/2014	87.0	22.7	-	-	-	-	8.50	-	268	42.0	-	-		
MW-110	D5	10/18/2012	144	39.6	0.062	<0.0250	22.1	12.2	17.6	0.115	193	21.6	15.7	<0.0250
		1/30/2013	171	46.6	-	-	-	-	10.3	-	141	28.6	-	-
		4/25/2013	183	74.7	0.085	<0.0250	26.4	10.5	11.8	8.43	160	26.4	20.7	<0.0250
		7/24/2013	304	43.8	-	-	-	-	9.41	-	144	17.8	-	-
		10/28/2013	174	94.8	-	-	-	-	15.3	-	190	21.1	-	-
		1/14/2014	179	85.1	-	-	-	-	19.4	-	248	12.1	-	-
		4/24/2014	189	86.1	<0.0250	<0.0250	32.9	12.7	20.9	0.194	310	12.5	24.0	<0.0250
		7/16/2014	185	82.3	-	-	-	-	24.1	-	390	15.4	-	-
10/28/2014	294	57.6	-	-	-	-	23.5	-	417	12.9	-	-		



Table 3-2 (cont'd)
Summary of Groundwater PFC Analytical Data
October 2012 - October 2014
Cottage Grove Site, Cottage Grove, MN

Well ID	Location	Date	PFC Groundwater Results (ppb, µg/L)											
			PFBA	PFBS	PFDA	PFDoA	PFHA	PFHpA	PFHS	PFNA	PFOA	PFOS	PFPeA	PFUnA
MW-12	D5	10/19/2012	975	94.3	3.38	<0.0250	93.3	33.3	20.9	3.95	1390	122	84.9	0.105
		1/30/2013	484	91.4	-	-	-	-	13.7	-	946	143	-	-
		4/25/2013	622	105	2.65	<0.0250	61.1	20.9	16.4	84.0	1020	145	72.7	0.625
		7/24/2013	1110	175	-	-	-	-	23.9	-	2000	204	-	-
		10/28/2013	408	87.7	-	-	-	-	13.0	-	954	131	-	-
		1/16/2014	312	82.1	-	-	-	-	13.1	-	668	121	-	-
		4/25/2014	220	68.9	1.85	0.162	26.8	11.7	9.82	1.76	548	117	24.8	0.424
		7/17/2014	154	50.4	-	-	-	-	8.11	-	402	96.9	-	-
10/30/2014	127	46.1	-	-	-	-	7.78	-	348	92.5	-	-		
MW-16	D5	10/18/2012	33.6	25.7	0.198	<0.0250	5.02	1.88	2.71	0.283	45.9	23.1	6.43	<0.0250
		1/29/2013	31.5	28.3	-	-	-	-	2.54	-	42.6	20.2	-	-
		4/24/2013	33.2	27.5	0.173	<0.0250	4.43	1.83	2.74	8.47	43.8	21.8	6.11	<0.0250
		7/24/2013	26.3	29.6	-	-	-	-	2.83	-	39.9	20.1	-	-
		10/28/2013	45.0	42.1	-	-	-	-	3.95	-	73.9	38.2	-	-
		1/14/2014	42.9	40.0	-	-	-	-	5.52	-	91.0	53.1	-	-
		4/24/2014	40.7	37.9	0.334	<0.0250	6.50	3.30	4.49	0.535	85.7	51.9	6.72	<0.0250
		7/16/2014	45.7	37.9	-	-	-	-	5.46	-	98.3	57	-	-
10/28/2014	38.9	33.9	-	-	-	-	4.58	-	75.8	42.6	-	-		
PW-06	D8	10/23/2012	82.2	26.4	0.497	<0.0250	13.4	4.52	4.90	0.598	172	37.9	12.9	<0.0250
		2/1/2013	85.0	37.5	-	-	-	-	4.74	-	188	35.5	-	-
		7/26/2013	82.5	42.9	-	-	-	-	4.16	-	217	42.3	-	-



Table 3-2 (cont'd)
Summary of Groundwater PFC Analytical Data
October 2012 - October 2014
Cottage Grove Site, Cottage Grove, MN

Well ID	Location	Date	PFC Groundwater Results (ppb, µg/L)											
			PFBA	PFBS	PFDA	PFDoA	PFHA	PFHpA	PFHS	PFNA	PFOA	PFOS	PFPeA	PFUnA
MW-14R	D8	7/24/2013	219	10.9	-	-	-	-	6.23	-	91.7	13.2	-	-
		1/13/2014	295	17.5	-	-	-	-	12.3	-	139	24.9	-	-
		4/23/2014	650	18.2	2.10	<0.0250	197	26.1	20.0	1.72	543	353	278	<0.0250
		7/16/2014	671	29.6	-	-	-	-	20.1	-	426	158	-	-
		10/29/2014	570	38.1	-	-	-	-	16.6	-	353	82.6	-	-
PW-10	EAST COVE	4/25/2013	4.25	0.594	<0.0250	<0.0250	0.153	0.049	0.197	<0.0250	0.428	0.396	0.290	<0.0250
		6/19/2013 ⁽¹⁾	-	-	-	-	-	-	-	-	0.415	0.401	-	-
		7/24/2013	3.37	0.540	-	-	-	-	0.185	-	0.420	0.380	-	-
		8/29/2013 ⁽¹⁾	-	-	-	-	-	-	-	-	0.378	0.315	-	-
		9/30/2013 ⁽¹⁾	-	-	-	-	-	-	-	-	0.528	0.465	-	-
		10/28/2013	6.09	0.703	-	-	-	-	0.193	-	0.524	0.458	-	-
		1/22/2014	4.40	0.261	-	-	-	-	0.049	-	0.212	0.124	-	-
		4/25/2014	5.07	0.471	<0.0250	<0.0250	0.151	0.047	0.110	<0.0250	0.421	0.437	0.252	<0.0250
		7/17/2014	4.9	0.54	-	-	-	-	0.104	-	0.484	0.503	-	-
10/30/2014	4.43	0.400	-	-	-	-	0.0777	-	0.376	0.337	-	-		
ECPZ-02R	E. COVE	7/24/2013	7.80	1.61	-	-	-	-	0.939	-	1.17	1.46	-	-
ECPZ-03R	E. COVE	7/24/2013	3.58	0.379	-	-	-	-	0.205	-	0.41	0.892	-	-
MW-04R	PLANT	7/25/2013	3.72	0.223	-	-	-	-	0.886	-	0.301	0.200	-	-

Notes:
 ppb - parts per billion
 µg/l. - micrograms per liter
 "-" - not analyzed
⁽¹⁾ Sampled as part of extended pumping test program.



**Table 3-3
Mann-Kendall Trend Test Summary
PFOS, PFOA, PFBA, PFBS and PFHS
Groundwater Analytical Data
Cottage Grove Site**

Well ID	Location	Trend Analysis Data Range	PFOS		PFOA		PFBA		PFBS		PFHS	
			Number of Data Points	Trend	Number of Data Points	Trend	Number of Data Points	Trend	Number of Data Points	Trend	Number of Data Points	Trend
MW-07	Background	3/2005 - 10/2014	13	Increasing	13	Increasing	9	NS	11	Increasing	13	Increasing
MW-101	D1/D2	3/2005 - 10/2014	13	Decreasing	13	Decreasing	9	Decreasing	12	Increasing	13	Decreasing
MW-104	D1/D2	9/2006 - 10/2014	9	NS	10	NS	9	NS ³	10	Decreasing	10	NS
MW-13	D9	3/2005 - 10/2014	10	NS	10	NS	9	NS	10	NS	10	NS
MW-105	D9	9/2006 - 10/2014	9	NS	9	Decreasing	10	NS	9	Decreasing	9	NS
PW-09	D9	4/2013 - 10/2014	9	Increasing	9	Increasing	7	NS	7	NS	7	NS
MW-108	WWTP	9/2006 - 10/2014	9	NS	9	NS ²	10	NS	9	NS	10	Decreasing
MW-110	D5	9/2006 - 10/2014	10	Decreasing	10	Increasing	9	Increasing ¹	10	Increasing	10	Increasing
MW-12	D5	3/2005 - 10/2014	10	Decreasing ¹	10	Decreasing	9	Decreasing	10	Decreasing	10	Decreasing
MW-16	D5	3/2005 - 10/2014	10	NS	10	Increasing	9	NS	10	Increasing	10	Increasing
MW-14R	D8	7/2013 - 10/2014	5	NS ⁴	5	NS ⁴	5	NS ⁴	5	Increasing	5	NS
PW-10	East Cove	4/2013 - 10/2014	9	NS	9	NS	7	NS	7	NS	9	Decreasing

Notes:

Trend analysis performed using PFC analytical data for each well from March 2005 through October 2014. Results are for the Mann-Kendall test for trend at a significance level of 0.05

NS = No statistically significant trend identified

¹ Previous trend was not statistically significant

² Previous trend was decreasing

³ Previous trend was increasing

⁴ Previous not applicable

3M_MN01595995



**Table 3-4
Summary of Pore Water and Surface Water Sample Results
September/October 2006 through October 2014
3M Cottage Grove Site**

LOCATION	Approximate distance from shoreline	ANALYTE	POREWATER (µg/L; ppb)					SURFACE WATER (µg/L; ppb)					
			Sept/Oct 06	Jun-11	Oct-12	Aug-13	Oct-14	Sept/Oct 06		Jun-11	Oct-12	Aug-13	Oct-14
			IW	IW	IW	IW	IW	Shallow	Deep	Composite	Composite	Composite	Composite
IW-09b	50 feet	PFBA	1.58	67.6	1.67	30.3	46.0	<0.100	<0.050	<0.100	0.553	<0.025	<0.0500
		PFOA	0.585	43.2	2.03	109	8.10	<0.050	0.530	<0.025	0.187	<0.024	0.050
		PFOS	0.114	45.6	1.27	2.84	2.95	<0.050	<0.050	<0.050	0.124	<0.0232	<0.0232
IW-09	100 feet	PFBA	5.01	2.32	4.65	8.10	6.71	1.24	NR	<0.100	0.300	<0.025	<0.0500
		PFOA	2.69	1.25	1.41	5.74	1.39	0.192	0.187	<0.025	0.139	<0.024	<0.0240
		PFOS	1.09	0.415	0.958	0.822	0.383	0.162	0.183	<0.050	0.088	<0.0232	<0.0232
IW-09d	300 feet	PFBA	0.112	83.3	11.8	0.057	2.94	NR	<0.200	<0.100	0.158	<0.025	<0.0500
		PFOA	<0.050	48.5	0.355	<0.0240	0.789	0.065	NR	<0.025	0.064	<0.024	<0.0240
		PFOS	<0.050	0.533	0.141	<0.0232	0.027	<0.050	<0.050	<0.050	0.043	<0.0232	<0.0232
IW-09f	500 feet	PFBA	NR	2.98	<0.0500	30.7	15.4	0.405	0.477	<0.100	0.204	<0.025	<0.0500
		PFOA	0.054	0.158	<0.0240	2.83	0.524	<0.050	0.054	<0.025	0.085	<0.024	<0.0240
		PFOS	<0.050	0.090	<0.0232	0.061	0.024	<0.050	<0.050	<0.050	0.051	<0.0232	<0.0232
IW-14b	50 feet	PFBA	695	79.9	24.9	32.1	24.4	NR	0.540	<0.100	0.345	0.0415	<0.0500
		PFOA	436	467	123	221	102	0.195	0.158	<0.025	1.36	0.061	0.0750
		PFOS	12.2	64.9	40.9	25.5	37.1	0.063	0.058	<0.050	0.780	<0.0232	<0.0232
IW-14	100 feet	PFBA	281	38.8	142	192	2.16	0.414	NR	<0.100	0.0618	<0.025	<0.0500
		PFOA	300	205	529	517	4.24	0.053	0.057	<0.025	<0.024	<0.024	<0.0240
		PFOS	12.4	28.3	18.3	4.22	0.251	NR	<0.050	<0.050	<0.0232	<0.0232	<0.0232
IW-14d	300 feet	PFBA	0.282	0.318	0.747	1.14	0.851	0.329	0.318	<0.100	<0.050	<0.025	<0.0500
		PFOA	<0.050	0.054	0.257	0.115	0.216	<0.100	<0.100	<0.025	<0.024	<0.024	<0.0240
		PFOS	<0.050	0.089	0.947	0.032	0.095	<0.050	<0.050	<0.050	<0.0232	<0.0232	<0.0232
IW-14f	500 feet	PFBA	0.178	33.3	78.4	39.1	76.1	<0.050	<0.050	<0.100	<0.050	<0.025	<0.0500
		PFOA	<0.050	0.813	59.3	0.926	4.19	<0.050	<0.050	<0.025	<0.024	<0.024	<0.0240
		PFOS	<0.025	0.086	1.23	0.05	0.048	<0.050	<0.050	<0.050	<0.0232	<0.0232	<0.0232

ppb - parts per billion

µg/L - micrograms per liter

IW - Interstitial Water (Pore water)

NR - Not reported due to quality control issues

NS - Not sampled

Note: In September/October 2006 two surface water samples were collected at discrete depth intervals at each location (i.e., shallow and deep, 0.2 and 0.8 of the total depth of the water column). In June 2011, October 2012, August 2013, and October 2014 one composite sample was collected at each location, composited from the same depth intervals.



Table 3-4 (cont'd)
Summary of Pore Water and Surface Water Sample Results
September/October 2006 through October 2014
3M Cottage Grove Site

LOCATION	Approximate distance from shoreline	ANALYTE	POREWATER (µg/L; ppb)					SURFACE WATER (µg/L; ppb)					
			Sept/Oct 06	Jun-11	Oct-12	Aug-13	Oct-14	Sept/Oct 06		Jun-11	Oct-12	Aug-13	Oct-14
			IW	IW	IW	IW	IW	Shallow	Deep	Composite	Composite	Composite	Composite
IW-19b	50 feet	PFBA	NR	198	154	157	69.2	2.81	1.96	<0.100	0.223	0.115	0.068
		PFOA	118.5	34.4	10.5	6.96	147	0.154	0.129	<0.025	0.557	0.434	0.203
		PFOS	53.1	43.2	95.6	40.4	31.2	0.127	0.089	<0.050	0.160	0.417	0.137
IW-19	100 feet	PFBA	86.6	107	112	18.3	62.6	NR	NR	<0.100	0.089	0.037	<0.0500
		PFOA	28.9	257	8.43	1.63	9.15	0.119	0.126	<0.025	0.041	0.108	0.091
		PFOS	NR	30.3	81.1	0.691	92.3	0.105	0.097	<0.050	<0.0232	<0.0232	<0.0232
IW-19d	300 feet	PFBA	1.40	0.143	4.07	8.00	13.4	0.095	0.117	<0.100	0.069	<0.025	<0.0500
		PFOA	0.134	0.097	0.206	0.206	0.114	<0.050	<0.050	<0.025	<0.024	<0.024	<0.0240
		PFOS	0.081	0.118	0.246	0.207	<0.0232	<0.050	<0.050	<0.050	<0.0232	<0.0232	<0.0232
IW-19f	500 feet	PFBA	118	0.57	141	47.1	51.5	0.245	0.516	<0.100	0.051	<0.025	<0.0500
		PFOA	6.84	<0.0250	15.3	185	231	<0.050	NR	<0.025	0.028	<0.024	<0.0240
		PFOS	1.71	<0.0500	1.26	10.9	0.913	<0.050	<0.050	<0.050	<0.0232	<0.0232	<0.0232
IW-25b	50 feet	PFBA	NS	56	1.47	37.2	53.9	NS	NS	12.5	0.686	0.897	0.553
		PFOA	NS	97.3	1.2	169	374	NS	NS	1.01	0.202	0.328	0.173
		PFOS	NS	595	2.41	86	1300	NS	NS	1.10	0.151	0.122	0.148
IW-25	100 feet	PFBA	23.1	13.6	34.7	35.5	58.9	NR	NR	6.25	0.819	3.64	0.673
		PFOA	129	110	74.7	9.98	23.6	0.163	0.146	0.583	0.575	1.26	0.181
		PFOS	206	82.4	0.916	3.28	16.2	0.095	0.102	0.586	0.422	0.311	0.127
IW-25d	300 feet	PFBA	NS	25	15.9	38.6	10.8	NS	NS	<0.100	0.389	0.043	<0.0500
		PFOA	NS	16.6	2.04	19.5	36.7	NS	NS	<0.025	0.335	<0.024	0.045
		PFOS	NS	0.138	19.1	0.141	0.094	NS	NS	<0.050	0.236	<0.0232	<0.0232
IW-25f	500 feet	PFBA	NS	2.02	1.23	6.05	16.2	NS	NS	<0.100	0.760	<0.025	<0.0500
		PFOA	NS	1.25	0.482	5.18	10.0	NS	NS	<0.025	0.163	<0.024	<0.0240
		PFOS	NS	2.64	0.201	2.18	6.65	NS	NS	<0.050	0.087	<0.0232	<0.0232

ppb - parts per billion
µg/L - micrograms per liter
IW - Interstitial Water (Pore water)
NR - Not reported due to quality control issues
NS - Not sampled

Note: In September/October 2006 two surface water samples were collected at discrete depth intervals at each location (i.e., shallow and deep, 0.2 and 0.8 of the total depth of the water column). In June 2011, October 2012, August 2013, and October 2014 one composite sample was collected at each location, composited from the same depth intervals.

Table 3-2 PW SW Results.xlsx

3M_MN01595997



4. FINDINGS

The Groundwater, Pore Water and Surface Water PFC Sampling Plan (WESTON, 2012) was developed to collect analytical data that could be used to assess long-term groundwater and surface water conditions in the area within, and surrounding, the Cottage Grove Site. Groundwater elevation data are also collected to determine the long-term effect of the continuous pumping of the groundwater remediation system on water levels at the Site. In addition, pore water and surface water samples have been collected in the Mississippi River to determine whether any trends are apparent in PFC concentrations. The baseline groundwater, pore water, and surface water sampling event was performed in October 2012. The results and findings of this sampling were provided to the MPCA by 3M in June 2013 (WESTON, 2013).

The following findings are summarized for groundwater analytical data, surface water and pore water analytical data, and hydrogeologic data collected in 2014 at the Site.

4.1 GROUNDWATER ANALYTICAL DATA

- PFBA, PFOA, PFBS and PFHS are detected at low concentrations in background (upgradient) monitoring well MW-07. 3M is not aware of the source for PFCs in groundwater at this upgradient location.
- PFC concentrations are highest in groundwater samples collected from monitoring wells in the former D1/D2 (MW-101, MW-102, MW-103 and MW-104) and D5 (MW-12) Areas.
- The Mann-Kendall statistical trend results indicated a statistically significant increasing or decreasing trend at the following locations:
 - Decreasing:
 - PFOS in monitoring wells MW-101, MW-110 and MW-12;
 - PFOA in monitoring wells MW-101, MW-105 and MW-12;
 - PFBA in monitoring wells MW-101 and MW-12;
 - PFBS in monitoring wells MW-104, MW-105 and MW-12;



- PFHS in monitoring wells MW-101, MW-108, MW-12 and production wells PW-10.
- Increasing:
 - PFOS in monitoring wells MW-07 and production well PW-09.
 - PFOA production well PW-09 and monitoring wells MW-07, MW-110 and MW-16.
 - PFBA in monitoring well MW-110.
 - PFBS in monitoring wells MW-07, MW-101, MW-110, MW-16 and MW-14R, and production wells PW-09 and PW-10;
 - PFHS in production well PW-10 and monitoring wells MW-07, MW-110 and MW-16.

4.2 SURFACE WATER / PORE WATER ANALYTICAL DATA

- PFC concentrations in pore water are higher than surface water PFC concentrations.
- Pore water PFC concentrations fluctuate with no discernible trend at this time.
- Surface water PFC concentrations were primarily below laboratory quantitation limits except for a few locations close to the shore at transects IW-19 and IW-25.
- At the majority of pore water sampling locations, PFBA was detected at the highest concentration of the PFC compounds.

4.3 SITE HYDROGEOLOGIC CONDITIONS

- The direction of groundwater flow is from north to south towards the Mississippi River.
- The extended pilot pump test was completed in February 2014. Production wells PW-09 and PW-10 continued to operate at approximately 300 gpm. A report summarizing the results of the extended pilot pump test was provided to MPCA in March 2015 under separate cover.
- Groundwater levels in monitoring wells closest to the Mississippi River showed less fluctuation in response to recharge from the spring melt than those monitoring wells more distant from the river.



5. FUTURE COURSE OF ACTION

The analytical and hydraulic data resulting from the monitoring of Site groundwater conditions will continue to be used to establish normal concentration variations and historic ranges, assess possible trends in the concentrations of certain PFCs in groundwater, and to assess the impact of long-term pumping of the Site production wells on groundwater levels. After the groundwater interception system is expanded described in Section 3.2, confirmation that groundwater capture is being maintained over the required areas will be provided in the annual reports. In accordance with the Sampling Plan, the following activities will be performed in the approximate time-frames noted:

Groundwater Monitoring

- Quarterly groundwater sampling was completed in January 2015. The annual sampling event is scheduled for April 2015 and additional quarterly sampling events will be performed in August and November 2015.
- Groundwater elevation data will be collected on a quarterly basis at a minimum.

Future Submittals to MPCA

- The 2015 annual groundwater monitoring report will be submitted to MPCA presenting a summary of the groundwater analytical data collected at the Site in the first quarter of 2016.



6. REFERENCES

Gilbert, R.O., 1987. *Statistical Methods for Environmental Pollution Monitoring*. New York, van Nostrand Reinhold.

USEPA, 2009. *Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities Unified Guidance*. U.S. Environmental Protection Agency, EPA/530/R/09-007. Washington, D.C., March 2009.

WESTON (Weston Solutions, Inc.). 2008. *Feasibility Study*. Prepared by Weston Solutions, Inc., for the 3M Company. March 2008.

WESTON (Weston Solutions, Inc.). 2009. *Remedial Design/Response Action (RD/RA) Plan*. Prepared by Weston Solutions, Inc., for the 3M Company. March 2008.

WESTON (Weston Solutions, Inc.). 2012. *Groundwater, Pore Water, and Surface Water Sampling Plan for Perfluorochemicals (PFCs), 3M Cottage Grove Site*. Prepared by Weston Solutions, Inc., for the 3M Company. April 2012.

WESTON (Weston Solutions, Inc.). 2013. *Groundwater Extraction Well Extended Pump Test Conceptual Work Plan, 3M Cottage Grove Site*. Prepared by Weston Solutions, Inc., for the 3M Company. January 2013.

WESTON (Weston Solutions, Inc.). 2013. *Baseline Monitoring Report for Perfluorochemicals (PFCs) in Groundwater, Pore Water, and Surface Water, 3M Cottage Grove Site*. Prepared by Weston Solutions, Inc., for the 3M Company. June 2013.

WESTON (Weston Solutions, Inc.). 2015. *Extended Groundwater Pilot Pump Test and Design of Groundwater Recovery System, 3M Cottage Grove Site*. Prepared by Weston Solutions, Inc., for the 3M Company. March 2015.



ATTACHMENT A
SUMMARY OF WELL CONSTRUCTION INFORMATION

3M_MN01596002

3630.0053



**Summary of Well Information
Cottage Grove Site,
Cottage Grove, MN**

Well ID	Well Type	MDH Unique Well Number	TOC Elevation (ft MSL)	Depth to Top of Screen/ Open Hole* (ft BGS)	Depth to Bottom of Screen/ Bottom of Hole* (ft BGS)	Total Boring Depth* (ft BGS)	Well Diameter*	Unit Monitored	Northing (UTM meters)	Easting (UTM meters)
Production/Extraction Wells										
PW-01	Production	231867	804.35	NA	205	205	14	NA	4959573	506968
PW-02	Production	231868	805.50	NA	202	202	20	Cj / Csl	4959856	506985
PW-03	Production	231869	NA	120	224	224	16	Cj / Csl	4960110	506830
PW-04	Production	231870	808.22	142	275	275	16	Cj / Csl / Cf	4959810	506610
PW-05	Production	231871	700.71	85	110	110	24	Glaciofluvial sediment:s	4959167	506747
PW-06	Production	229117	705.77	92	117	143	24	Glaciofluvial sediment:s	4959164	506947.8
PW-07 (ABD)	Production	233576	823.22	NA	200	200	NA	NA	4960664	507187
PW-08	Production	NA	806.93	NA	NA	NA	NA	NA	4960087	506742
PW-09 (EW01)	Production	767283	781.23	164	224	225	12	Glaciofluvial sediment:s	4959117	507776
PW-10 (EW02)	Extraction	767284	711.11	90	150	150	12	Glaciofluvial sediment:s	4958932	508411
Monitor Wells										
MW-01	Monitoring	233567	822.32	NA	200	200	6	Cj / Csl	4961188	506875
MW-02	Monitoring	233568	805.92	NA	192	192	6	Cj / Csl	4960019	507126
MW-03	Monitoring	233569	810.07	NA	210	210	6	Cj / Csl	4959807	506782
MW-04	Monitoring	233570	806.90	NA	200	200	6	Cj / Csl	4959350	506931
MW-04R	Monitoring	797615	805.18	187	197	200	2	Cj	4959338	506928
MW-05	Monitoring	233571	808.18	NA	210	210	6	Cj / Csl	4961057	506085
MW-06 (ABD)	Monitoring	233572	814.93	84	219	219	6	Opo / Cj / Csl	4960161	506433
MW-07	Monitoring	233573	794.08	NA	146	146	6	Cj	4960436	507595
MW-08	Monitoring	233574	765.97	NA	173	173	6	Cj / Csl	4959687	506125
MW-09	Monitoring	233575	769.17	NA	104	104	4	Cj	4960016	506278
MW-10	Monitoring	233554	788.28	198	237	237	8	Cj / Csl	4959320	507415
MW-11	Monitoring	233850	804.34	NA	200	200	4	Cj	4959460	507120
MW-12	Monitoring	233951	781.93	122	141	141	5	Glaciofluvial sediment:s	4959193	507137
MW-13	Monitoring	NA	783.12	114	134	134	5	Glaciofluvial sediment:s	4959136	507763.2
MW-14 (ABD)	Monitoring	421705	710.34	43	64	64	4	Glaciofluvial sediment:s	4959179	506945
MW-14R	Monitoring	797616	703.90	59.1	69.1	70	2	Glaciofluvial sediment:s	4959174	506901
MW-15	Monitoring	431237	783.34	166	186	186	4	Glaciofluvial sediment:s	4959145	507128
MW-16	Monitoring	431238	784.21	120	140	140	4	Glaciofluvial sediment:s	4959133	507247
MW-17	Monitoring	570322	785.16	92	112	112	4	Cj	4959671	506505
MW-18	Monitoring	570323	782.93	72	92	92	4	Opo / Cj	4959779	506489
MW-19	Monitoring	NA	757.36	NA	120	120	4	NA	4959655	506309
PZ-14	Piezometer	NA	755.96	NA	NA	187	2	NA	4959241	506744
MW-101	Monitoring	680685	785.94	90	100	100	2	Glaciofluvial sediment:s	4959104	508044
MW-102	Monitoring	680686	783.25	86	96	96	2	Glaciofluvial sediment:s	4959171	508072
MW-103	Monitoring	727759	770.73	78	88	88	2	Glaciofluvial sediment:s	4959065	508009
MW-104	Monitoring	727770	771.94	78	88	90	2	Glaciofluvial sediment:s	4959056	507966
MW-105	Monitoring	727763	782.38	86.5	96.5	98.5	2	Glaciofluvial sediment:s	4959122	507827
MW-106 (ABD)	Monitoring	NA	781.53	85	95	95	2	Glaciofluvial sediment:s	4959164	507823
MW-107 (ABD)	Monitoring	NA	778.74	81.5	91.5	92	2	Glaciofluvial sediment:s	4959194	507843
MW-108	Monitoring	727764	787.51	93.5	103.5	103.5	2	Glaciofluvial sediment:s	4959091	507590
MW-109	Monitoring	727765	782.95	36.5	46.5	46.5	2	Glaciofluvial sediment:s	4959171	507192
MW-110	Monitoring	727766	783.03	100	110	113.5	2	Glaciofluvial sediment:s	4959170	507195
MW-111	Monitoring	NA	783.26	NA	NA	NA	NA	NA	4959170	507274
MW-112	Monitoring	797614	783.06	130	140	140	2	Glaciofluvial sediment:s	4959114	508210
ECPZ-01	Monitoring	747072	762.92	102	112	112	2	Glaciofluvial sediment:s	4959007	508365
ECPZ-02 (ABD)	Monitoring	747073	691.75	33	43	46.5	2	Glaciofluvial sediment:s	4958935	508452
ECPZ-02R	Monitoring	780910	693.31	35	45	47	2	Glaciofluvial sediment:s	4958934	508452
ECPZ-03 (ABD)	Monitoring	747074	689.22	29.4	39.4	41	2	Glaciofluvial sediment:s	4958936	508518
ECPZ-03R	Monitoring	780911	690.70	30.2	40.2	42	2	Glaciofluvial sediment:s	4958932	508517
INF-PZ01	Monitoring	763546	NA	44.7	54.75	55	2	Glaciofluvial sediment:s	4958966	507541

Notes:

- * - Information obtained from well completion reports.
- (ABD) - Well Abandoned.
- ft BGS - Feet below ground surface.
- ft MSL - Feet above mean sea level.
- NA - Not Available

- Opo - Oneota Dolomite
- Cj - Jordan Sandstone
- Csl - St. Lawrence Formation
- Cf - Franconia Formation



**ATTACHMENT B
PORE WATER AND SURFACE WATER SAMPLING SHEETS
SEPTEMBER/OCTOBER 2014 SAMPLING EVENT**

3M_MN01596004

3630.0055

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-09	DATE:	10/1/2014
Drainage System:	Mississippi River	County:	Washington
		State:	MN
GPS Latitude:	44.784585	GPS Longitude:	-92.909316
Location Description: IW-09 transect line			
Water Conditions:	() Low Turbidity (X) Moderate Turbidity () High Turbidity		
	Water Conditions: () Calm (X) Choppy () Visible Current		
Weather Conditions:	1-Oct Temp. 50 °F () Partly Sunny (X) Cloudy (X) Rain (X) Windy		

SURFACE WATER

Depth to Sediment: 13.7' ftbws Sediment Bottom: () Firm (X) Soft
 Water Sample Method: (X) Peristaltic Pump

Water Sample Data				
Sample Depth (ftbws)	Composite (2.74' and 9.96')			
Flow Rate (gallons per minute)	0.1 gpm			
Sample Date	10/01/2014			
Sample Time	14:10			
pH / SC / T (°C) / DO (mg/L) / ORP	8.33	349	16.03	6.78 28
Sample ID	CGMN-SW-MRIW09-0-141001			
Duplicate ID	CGMN-SW-MRIW09-DB-141001			
Field matrix spike (1.0 ng/mL)	-			

COMMENTS: Rinse Blank CGMN-SW-MRIW09-RB-01-141001

PORE WATER

Well Diameter (in) 1
 Depth to Sediment (ftbws) 13.7
 Depth to Top of Screen (ftbws) 14.2
 Depth to Bottom of Screen (ftbws) 14.7
 Water column 14.0'
 Well Volume (gal) 1.26'
 Purge Start (time) 14:29
 Purge Stop (time) 14:37
 Volume Purged (gal) 1.2 gallons
 Well Purged Dry? (X) Yes () No
 Sample Method: (X) Peristaltic Pump

Water Sample Data				
Sample Depth Interval (ftbws)	14.20' to 14.70'			
Sample Date	10/02/2014			
Sample Time	09:05			
pH / SC / T (°C) / DO (mg/L) / ORP	8.32	299	15.92	6.73 51.5
Sample ID	CGMN-IW-MRIW09-0-141002			
Duplicate ID	CGMN-IW-MRIW09-DB-141002			
Field matrix spike (10 or 100 ng/mL)	CGMN-IW-MRIW09-FMS-141002			

COMMENTS: Sampling- DTR: 1.10', DTW: 13:20; Not much water in point; Gray clay

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-09d	DATE:	10/1/2014
Drainage System:	Mississippi River	County:	Washington
		State:	MN
GPS Latitude:	44.784018	GPS Longitude:	-92.909633
Location Description: IW-09 transect line			
Water Conditions:	<input type="checkbox"/> Low Turbidity <input checked="" type="checkbox"/> Moderate Turbidity <input type="checkbox"/> High Turbidity <input type="checkbox"/> Calm <input checked="" type="checkbox"/> Choppy <input type="checkbox"/> Visible Current		
Weather Conditions:	1-Oct Temp. 50 °F <input type="checkbox"/> Partly Sunny <input checked="" type="checkbox"/> Cloudy <input checked="" type="checkbox"/> Rain <input checked="" type="checkbox"/> Windy		

SURFACE WATER

Depth to Sediment: 11.7 ftbws **Sediment Bottom:** Firm Soft
Water Sample Method: Peristaltic Pump

Water Sample Data					
Sample Depth (ftbws)	Composite (2.34 and 9.36)				
Flow Rate (gallons per minute)	0.1 gpm				
Sample Date	10/01/2014				
Sample Time	13:25				
pH / SC / T (°C) / DO (mg/L) / ORP	8.35	328	15.6	7.16	57
Sample ID	CGMN-SW-MRIW09d-0-141001				
Duplicate ID	CGMN-SW-MRIW09d-DB-141001				
Field matrix spike (1.0 ng/mL)	CGMN-SW-MRIW09d-FMS-141001				
Rinsate Sample ID	-				

COMMENTS: DTR: 2.40'; DTW: 2.42'; Gray silty sand

PORE WATER

Well Diameter (in) 1
Depth to Sediment (ftbws) 11.7'
Depth to Top of Screen (ftbws) 13.5'
Depth to Bottom of Screen (ftbws) 14.0'
Well Volume (gal) 1.22 gallons
Purge Start (time) 13:40
Purge Stop (time) 13:52
Volume Purged (gal) 1.80 gallons
Well Purged Dry? Yes No
Sample Method: Peristaltic Pump

Water Sample Data					
Sample Depth Interval (ftbws)	13.5'-14.0'				
Sample Date	10/01/2014				
Sample Time	13:55				
pH / SC / T (°C) / DO (mg/L) / ORP	7.5	377	15.63	2.07	-112
Sample ID	CGMN-IW-MRIW09d-0-141001				
Duplicate ID	CGMN-IW-MRIW09d-DB-141001				
Field matrix spike (10 or 100 ng/mL)	Not Applicable				
Rinsate Sample ID	Not Applicable				

COMMENTS: Gray silty sand

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-09b	DATE:	10/1/2014
Drainage System:	Mississippi River	County:	Washington
		State:	MN
GPS Latitude:	44.784765	GPS Longitude:	-92.909144
Location Description: IW-09 transect line			
Water Conditions:	() Low Turbidity (X) Moderate Turbidity () High Turbidity		
	Water Conditions: () Calm (X) Choppy () Visible Current		
Weather Conditions:	1-Oct Temp. 50 °F () Partly Sunny (X) Cloudy (X) Rain (X) Windy		

SURFACE WATER

Depth to Sediment: 8.5' ftbws Sediment Bottom: () Firm (X) Soft

Water Sample Method: (X) Peristaltic Pump

Water Sample Data				
Sample Depth (ftbws)	Composite (1.70 and 6.80)			
Flow Rate (gallons per minute)	0.1 gpm			
Sample Date	10/01/2014			
Sample Time	14:50			
pH / SC / T (°C) / DO (mg/L) / ORP	8.35	346	16	6.9 6.1
Sample ID	CGMN-SW-MRIW09b-0-141001			
Duplicate ID	CGMN-SW-MRIW09b-DB-141001			
Field matrix spike (1.0 ng/mL)	-			

COMMENTS: _____

PORE WATER

Well Diameter (in) 1

Depth to Sediment (ftbws) 8.50'

Depth to Top of Screen (ftbws) 11.0'

Depth to Bottom of Screen (ftbws) 11.5'

Well Volume (gal) 1.03 gallons

Purge Start (time) 15:02

Purge Stop (time) 15:09

Volume Purged (gal) 1.05 gallons

Well Purged Dry? (X) Yes () No

Sample Method: (X) Peristaltic Pump

Water Sample Data				
Sample Depth Interval (ftbws)	11.0'-11.5'			
Sample Date	10/01/2014			
Sample Time	09:25			
pH / SC / T (°C) / DO (mg/L) / ORP	7.9	329	15.88	6.36 -30
Sample ID	CGMN-IW-MRIW09b-0-141002			
Duplicate ID	CGMN-IW-MRIW09b-DB-141002			
Field matrix spike (10 or 100 ng/mL)	-			
Rinsate Sample ID	-			

COMMENTS: Gray clay, soft; DTR: 4.55'; DTW: 4.20'

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-14	DATE:	9/30/2014
Drainage System:	Mississippi River	County:	Washington
		State:	MN
GPS Latitude:	44.783699	GPS Longitude:	-92.904236
Location Description: IW-14 transect line			
Water Conditions:	<input type="checkbox"/> Low Turbidity <input checked="" type="checkbox"/> Moderate Turbidity <input type="checkbox"/> High Turbidity <input type="checkbox"/> Calm <input checked="" type="checkbox"/> Choppy <input type="checkbox"/> Visible Current		
Weather Conditions:	Temp. 50 °F <input type="checkbox"/> Sunny <input checked="" type="checkbox"/> Cloudy <input type="checkbox"/> Rain <input checked="" type="checkbox"/> Windy		

SURFACE WATER

Depth to Sediment: 17.5 ftbws **Sediment Bottom:** Firm Soft
Water Sample Method: Peristaltic Pump

Water Sample Data				
Sample Depth (ftbws)	Composite (3.5' and 14')			
Flow Rate (gallons per minute)	0.1 gpm			
Sample Date	09/30/2014			
Sample Time	16:10			
pH / SC / T (°C) / DO (mg/L) / ORP	8.44	353	17.65	7.59
Sample ID	CGMN-SW-MRIW014-0-140930			
Duplicate ID	CGMN-SW-MRIW14-DB-140930			
Field matrix spike (1.0 ng/mL)	CGMN-SW-MRIW14-FMS-140930			

COMMENTS:

PORE WATER

Well Diameter (in) 1
Depth to Sediment (ftbws) 17.5
Depth to Top of Screen (ftbws) 19.3'
Depth to Bottom of Screen (ftbws) 19.8'
Water column 15.46
Well Volume (gal) 1.39
Purge Start (time) 12:06
Purge Stop (time) 12:19
Volume Purged (gal) 1.95
Well Purged Dry? Yes No
Sample Method: Peristaltic Pump

Water Sample Data				
Sample Depth Interval (ftbws)	19.3'-19.8'			
Sample Date	10/01/2014			
Sample Time	12:20			
pH / SC / T (°C) / DO (mg/L) / ORP	7.9	633	14.86	7.2
Sample ID	CGMN-IW-MRIW14-0-141001			
Duplicate ID	CGMN-IW-MRIW14-DB-141001			
Field matrix spike (10 or 100 ng/mL)	-			

COMMENTS:

Sandy sediment; DTR: 1.16; DTW: 2.04

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-14b			DATE:	9/30/2014
Drainage System:	Mississippi River	County:	Washington	State:	MN
GPS Latitude:	44.783879	GPS Longitude:	-92.904248		
Location Description:	IW-14 transect line				
Water Conditions:	() Low Turbidity (X) Moderate Turbidity () High Turbidity				
	Water Conditions: () Calm (X) Choppy () Visible Current				
Weather Conditions:	Temp. 50 °F () Sunny (X) Cloudy () Rain (X) Windy				

SURFACE WATER

Depth to Sediment: 6.6 ftbws Sediment Bottom: (X) Firm () Soft
 Water Sample Method: (X) Peristaltic Pump

Water Sample Data					
Sample Depth (ftbws)	Composite (1.32' and 5.28')				
Flow Rate (gallons per minute)	0.1 gpm				
Sample Date	09/30/2014				
Sample Time	16:35				
pH / SC / T (°C) / DO (mg/L) / ORP	8.46	344	17.8	7.7	103
Sample ID	CGMN-SW-MRIW014b-0-140930				
Duplicate ID	CGMN-SW-MRIW14b-DB-140930				
Field matrix spike (1.0 ng/mL)	-				

COMMENTS:

PORE WATER

Well Diameter (in) 1
 Depth to Sediment (ftbws) 6.6'
 Depth to Top of Screen (ftbws) 7.55'
 Depth to Bottom of Screen (ftbws) 8.05'
 Water column 7.3'
 Well Volume (gal) 0.67
 Purge Start (time) 12:31
 Purge Stop (time) 12:36
 Volume Purged (gal) 0.75
 Well Purged Dry? (X) Yes () No
 Sample Method: (X) Peristaltic Pump

Water Sample Data					
Sample Depth Interval (ftbws)	7.55' to 8.05'				
Sample Date	10/02/2014				
Sample Time	08:40				
pH / SC / T (°C) / DO (mg/L) / ORP	7.74	405	15.36	7.74	-65
Sample ID	CGMN-IW-MRIW14b-0-141002				
Duplicate ID	CGMN-IW-MRIW14b-DB-141002				
Field matrix spike (10 or 100 ng/mL)	-				

COMMENTS:

Gray/ brown sand; DTR: 4.45'; DTW: 4.70'

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-14d			DATE:	9/30/2014
Drainage System:	Mississippi River	County:	Washington	State:	MN
GPS Latitude:	44.783114	GPS Longitude:	-92.904237		
Location Description:	IW-14 transect line				
Water Conditions:	() Low Turbidity (X) Moderate Turbidity () High Turbidity				
	Water Conditions: (X) Calm () Choppy () Visible Current				
Weather Conditions:	Temp. 50 °F () Sunny (X) Cloudy () Rain (X) Windy				

SURFACE WATER

Depth to Sediment: 7.90' ftbws Sediment Bottom: () Firm () Soft
 Water Sample Method: (X) Peristaltic Pump

Water Sample Data					
Sample Depth (ftbws)	Composite (1.58' and 6.32')				
Flow Rate (gallons per minute)	0.1 gpm				
Sample Date	09/30/2014				
Sample Time	15:35				
pH / SC / T (°C) / DO (mg/L) / ORP	8.43	373	17.8	7.47	84
Sample ID	CGMN-SW-MRIW014d-0-140930				
Duplicate ID	CGMN-SW-MRIW14d-DB-140930				
Field matrix spike (1.0 ng/mL)	-				

COMMENTS:**PORE WATER**

Well Diameter (in) 1
 Depth to Sediment (ftbws) 7.90'
 Depth to Top of Screen (ftbws) 8.80'
 Depth to Bottom of Screen (ftbws) 9.3
 Water column 7.95
 Well Volume (gal) 0.7
 Purge Start (time) 15:45
 Purge Stop (time) 15:50
 Volume Purged (gal) 0.8
 Well Purged Dry? (X) Yes () No
 Sample Method: (X) Peristaltic Pump

Water Sample Data					
Sample Depth Interval (ftbws)	7.9'-8.3'				
Sample Date	10/01/2014				
Sample Time	10:00				
pH / SC / T (°C) / DO (mg/L) / ORP	8.42	380	17.52	7.46	90
Sample ID	CGMN-IW-MRIW14d-0-141001				
Duplicate ID	CGMN-IW-MRIW14d-DB-141001				
Field matrix spike (10 or 100 ng/mL)	CGMN-IW-MRIW14d-FMS-141001				
	-				

COMMENTS:

Gray Clay; Some sediment in sample/ sl. turbid; DTR: 1.70'; DTW: 3.05'

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-14f		DATE:	9/30/2014	
Drainage System:	Mississippi River	County:	Washington	State:	MN
GPS Latitude:	44.782583	GPS Longitude:	-92.904238		
Location Description:	IW-14 transect line				
Water Conditions:	() Low Turbidity (X) Moderate Turbidity () High Turbidity				
	Water Conditions: () Calm (X) Choppy () Visible Current				
Weather Conditions:	Temp. 50 °F () Sunny (X) Cloudy () Rain (X) Windy				

SURFACE WATER

Depth to Sediment: 7.35 ftbws Sediment Bottom: () Firm () Soft
 Water Sample Method: (X) Peristaltic Pump

Water Sample Data					
Sample Depth (ftbws)	Composite (1.47' and 5.88')				
Flow Rate (gallons per minute)	0.1 gpm				
Sample Date	09/30/2014				
Sample Time	14:45				
pH / SC / T (°C) / DO (mg/L) / ORP	8.44	329	17.6	7.36	77
Sample ID	CGMN-SW-MRIW014f-0-140930				
Duplicate ID	CGMN-SW-MRIW14f-DB-140930				
Field matrix spike (1.0 ng/mL)	-				
Rinsate Sample ID	-				

COMMENTS:

PORE WATER

Well Diameter (in) 1
 Depth to Sediment (ftbws) 7.35'
 Depth to Top of Screen (ftbws) 8.20'
 Depth to Bottom of Screen (ftbws) 8.70'
 Water column 7.40'
 Well Volume (gal) 0.66
 Purge Start (time) 15:05
 Purge Stop (time) 15:09
 Volume Purged (gal) 0.6
 Well Purged Dry? (X) Yes () No
 Sample Method: (X) Peristaltic Pump

Water Sample Data					
Sample Depth Interval (ftbws)	8.20' to 8.70'				
Sample Date	10/02/2014				
Sample Time	08:25				
pH / SC / T (°C) / DO (mg/L) / ORP	8.45	349	16.91	7.1	88
Sample ID	CGMN-IW-MRIW14f-0-141002				
Duplicate ID	CGMN-IW-MRIW14f-DB-141002				
Field matrix spike (10 or 100 ng/mL)	-				
Rinsate Sample ID	-				

COMMENTS:

Gray Clay; DTR: 2.30'; DTW: 3.60'

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-19b	DATE:	9/30/2014
Drainage System:	Mississippi River	County:	Washington
		State:	MN
GPS Latitude:	44.784180	GPS Longitude:	-92.899002
Location Description: IW-19 transect line			
Water Conditions:	() Low Turbidity (X) Moderate Turbidity () High Turbidity		
	Water Conditions: () Calm (X) Choppy () Visible Current		
Weather Conditions:	Temp. 50 °F () Sunny (X) Cloudy () Rain (X) Windy		

SURFACE WATER

Depth to Sediment: 5.6 ftbws Sediment Bottom: () Firm (X) Soft

Water Sample Method: (X) Peristaltic Pump

Water Sample Data				
Sample Depth (ftbws)	Composite (1.12' and 4.50")			
Flow Rate (gallons per minute)	0.1 gpm			
Sample Date	09/30/2014			
Sample Time	13:40			
pH / SC / T (°C) / DO (mg/L) / ORP	8.42	349	17.81	7.32 52
Sample ID	CGMN-SW-MRIW019b-0-140930			
Duplicate ID	CGMN-SW-MRIW19b-DB-140930			
Field matrix spike (1.0 ng/mL)	-			

COMMENTS: _____

PORE WATER

Probe Diameter (in) 1

Depth to Sediment (ftbws) 5.6

Depth to Top of Screen (ftbws) 7.6

Depth to Bottom of Screen (ftbws) 8.1

Water column 6.75

Well Volume (gal) 0.6

Purge Start (time) 13:59

Purge Stop (time) 14:02

Volume Purged (gal) 0.6

Well Purged Dry? (X) Yes () No

Sample Method: (X) Peristaltic Pump

Water Sample Data				
Sample Depth Interval (ftbws)	7.6' to 8.1'			
Sample Date	10/01/2014			
Sample Time	09:30			
pH / SC / T (°C) / DO (mg/L) / ORP	8.31	386	7.67	6.01 61
Sample ID	CGMN-IW-MRIW19b-0-141001			
Duplicate ID	CGMN-IW-MRIW19b-DB-141001			
Field matrix spike (10 or 100 ng/mL)	-			

COMMENTS: Gray clay; DTR: 2.5'; DTW: 4.25'

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-19f		DATE:	9/30/2014	
Drainage System:	Mississippi River	County:	Washington	State:	MN
GPS Latitude:	44.782929	GPS Longitude:	-92.899194		
Location Description:	IW-19 transect line				
Water Conditions:	<input type="checkbox"/> Low Turbidity <input checked="" type="checkbox"/> Moderate Turbidity <input type="checkbox"/> High Turbidity <input type="checkbox"/> Calm <input checked="" type="checkbox"/> Choppy <input type="checkbox"/> Visible Current				
Weather Conditions:	Temp. 45 °F	<input type="checkbox"/> Sunny <input checked="" type="checkbox"/> Cloudy <input type="checkbox"/> Rain <input checked="" type="checkbox"/> Windy			

SURFACE WATER

Depth to Sediment: 12.1 ftbws Sediment Bottom: Firm Soft
 Water Sample Method: Peristaltic Pump

Water Sample Data					
Sample Depth (ftbws)	Composite (2.42' and 9.68')				
Flow Rate (gallons per minute)	0.1 gpm				
Sample Date	09/30/2014				
Sample Time	11:15				
pH / SC / T (°C) / DO (mg/L) / ORP	8.34	338	16.02	6.77	13
Sample ID	CGMN-SW-MRIW019f-0-140930				
Duplicate ID	CGMN-SW-MRIW19f-DB-140930				
Field matrix spike (1.0 ng/mL)	-				
Rinsate Sample ID	Note Applicable				

COMMENTS:

PORE WATER

Well Diameter (in) 1
 Depth to Sediment (ftbws) 12.1
 Depth to Top of Screen (ftbws) 13.6
 Depth to Bottom of Screen (ftbws) 14.1
 Total Probe Depth (ftbws) 14.1
 Water column 13.05
 Well Volume (gal) 1.2
 Purge Start (time) 8:45
 Purge Stop (time) 8:54
 Volume Purged (gal) 1.35

Well Purged Dry? Yes NoSample Method: Peristaltic Pump

Water Sample Data					
Sample Depth Interval (ftbws)	13.6' to 14.1'				
Sample Date	10/02/2014				
Sample Time	08:05				
pH / SC / T (°C) / DO (mg/L) / ORP	7.67	498	14.47	4.1	-48
Sample ID	CGMN-IW-MRIW19f-0-141002				
Duplicate ID	CGMN-IW-MRIW19f-DB-141002				
Field matrix spike (10 or 100 ng/mL)	-				
Rinsate Sample ID	Not Applicable				

COMMENTS:

Gray Clay; DTR: 4.11'; DTW: 6.05'

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-25b	DATE:	9/30/2014
Drainage System:	Mississippi River	County:	Washington
State:	MN		
GPS Latitude:	44.78281	GPS Longitude:	-92.89304
Location Description: IW-25 transect line			
Water Conditions:	<input type="checkbox"/> Low Turbidity <input checked="" type="checkbox"/> Moderate Turbidity <input type="checkbox"/> High Turbidity <input type="checkbox"/> Calm <input checked="" type="checkbox"/> Choppy <input type="checkbox"/> Visible Current		
Weather Conditions:	Temp. 45 °F <input type="checkbox"/> Sunny <input checked="" type="checkbox"/> Cloudy <input type="checkbox"/> Rain <input checked="" type="checkbox"/> Windy		

SURFACE WATER

Depth to Sediment: 1.05 ftbws Sediment Bottom: Firm Soft
 Water Sample Method: Peristaltic Pump

Water Sample Data				
Sample Depth (ftbws)	0.75			
Flow Rate (gallons per minute)	0.1 gpm			
Sample Date	09/30/2014			
Sample Time	10:25			
pH / SC / T (°C) / DO (mg/L) / ORP	8.24	335	16.14	6.56 20
Sample ID	CGMN-SW-MRIW25b-0-140930			
Duplicate ID	CGMN-SW-MRIW25b-DB-140930			
Field matrix spike (1.0 ng/mL)	-			

COMMENTS:

PORE WATER

Well Diameter (in) 1"
 Depth to Sediment (ftbws) 1.05
 Depth to Top of Screen (ftbws) 1.55
 Depth to Bottom of Screen (ftbws) 2.05
 Well Volume (gal) 0.2
 Purge Start (time) 10:28
 Purge Stop (time) 10:40
 Volume Purged (gal) 11
 Well Purged Dry? Yes No
 Sample Method: Peristaltic Pump

Water Sample Data				
Sample Depth Interval (ftbws)	1.05' to 1.55'			
Sample Date	09/30/2014			
Sample Time	10:45			
pH / SC / T (°C) / DO (mg/L) / ORP	6.85	1063	16.33	0.43 -110
Sample ID	CGMN-IW-MRIW25b-0-140930			
Duplicate ID	CGMN-IW-MRIW25b-DB-140930			
Field matrix spike (10 or 100 ng/mL)	-			

COMMENTS:

Poorly sorted light gray sand (f-cs), trace fines; DTR:3.27'; DTW: 3.22'

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-25d	DATE:	10/24/2012
Drainage System:	Mississippi River	County:	Washington
		State:	MN
GPS Latitude:	44.78204	GPS Longitude:	-92.89319
Location Description: IW-25 transect line			
Water Conditions:	() Low Turbidity (X) Moderate Turbidity () High Turbidity		
	Water Conditions: () Calm (X) Choppy () Visible Current		
Weather Conditions:	Temp. 45 °F () Sunny (X) Cloudy () Rain (X) Windy		

SURFACE WATER

Depth to Sediment: 5.5 ftbws Sediment Bottom: () Firm () Soft

Water Sample Method: (X) Peristaltic Pump

Water Sample Data				
Sample Depth (ftbws)	Composite (1.1' and 4.4')			
Flow Rate (gallons per minute)	0.1 gpm			
Sample Date	09/30/2014			
Sample Time	8:25			
pH / SC / T (°C) / DO (mg/L) / ORP	8.37	350	16.72	6.5 200.3
Sample ID	CGMN-SW-MRIW25d-0-140929			
Duplicate ID	CGMN-SW-MRIW25d-DB-140929			
Field matrix spike (1.0 ng/mL)	-			
Rinsate Sample ID	Not Applicable			

COMMENTS: _____

PORE WATER

Well Diameter (in) 1

Depth to Sediment (ftbws) 5.5

Depth to Top of Screen (ftbws) 6

Depth to Bottom of Screen (ftbws) 6.5

Water column 6.1

Well Volume (gal) 0.55

Purge Start (time) 8:53

Purge Stop (time) 8:57

Volume Purged (gal) 0.6

Well Purged Dry? (X) Yes () No

Sample Method: (X) Peristaltic Pump

Water Sample Data				
Sample Depth Interval (ftbws)	6.0' to 6.5'			
Sample Date	10/01/2014			
Sample Time	08:10			
pH / SC / T (°C) / DO (mg/L) / ORP	8.3	328	15.2	6.67 nm
Sample ID	CGMN-IW-MRIW25d-0-141001			
Duplicate ID	CGMN-IW-MRIW25d-DB-141001			
Field matrix spike (10 or 100 ng/mL)	-			
Rinsate Sample ID	Not Applicable			

COMMENTS: Gray sandy (v. fine- fine) silt/ clay; DTW: 5.9'; DTR: 4.35'

PORE SAMPLE DATA SHEET

SAMPLE LOCATION	IW-25f	DATE:	9/29/2014
Drainage System:	Mississippi River	County:	Washington
State:	MN		
GPS Latitude:	44.78153	GPS Longitude:	-92.89331
Location Description: IW-25 transect line			
Water Conditions:	<input type="checkbox"/> Low Turbidity <input checked="" type="checkbox"/> Moderate Turbidity <input type="checkbox"/> High Turbidity		
	Water Conditions: <input type="checkbox"/> Calm <input checked="" type="checkbox"/> Choppy <input type="checkbox"/> Visible Current		
Weather Conditions:	Temp. 59 °F <input type="checkbox"/> Sunny <input checked="" type="checkbox"/> Cloudy <input checked="" type="checkbox"/> Rain <input type="checkbox"/> Windy		

SURFACE WATER

Depth to Sediment: 7.7 ftbws Sediment Bottom: Firm Soft
 Water Sample Method: Peristaltic Pump

Water Sample Data				
Sample Depths (ftbws)	Composite (1.54' and 6.16')			
Flow Rate (gallons per minute)	0.1 gpm			
Sample Date	09/29/2014			
Sample Time	16:40			
pH / SC / T (°C) / DO (mg/L) / ORP	8.23	378	19.36	7.13 195
Sample ID	CGMN-SW-MRIW25f-0-140929			
Duplicate ID	CGMN-SW-MRIW25f-DB-140929			
Field matrix spike (1.0 ng/mL)	CGMN-SW-MRIW25f-FMS-140929			

COMMENTS:

PORE WATER

Well Diameter (in) 1
 Depth to Sediment (ftbws) 7.7
 Depth to Top of Screen (ftbws) nm
 Depth to Bottom of Screen (ftbws) nm
 1 Probe Volume (gal) 0.68
 Purge Start (time) nm
 Purge Stop (time) nm
 Volume Purged (gal) nm
 Well Purged Dry? Yes No
 Sample Method: Peristaltic Pump

Water Sample Data				
Sample Depth Interval (ftbws)	7.9'			
Sample Date	10/01/2014			
Sample Time	08:25			
pH / SC / T (°C) / DO (mg/L) / ORP	8.25	345	18.96	7.26 nm
Sample ID	CGMN-IW-MRIW25f-0-141001			
Duplicate ID	CGMN-IW-MRIW25f-DB-141001			
Field matrix spike (10 or 100 ng/mL)	CGMN-IW-MRIW25f-FMS-141001			

COMMENTS:

Gray Clay; DTR: 1.05'; DTW: 2.41'

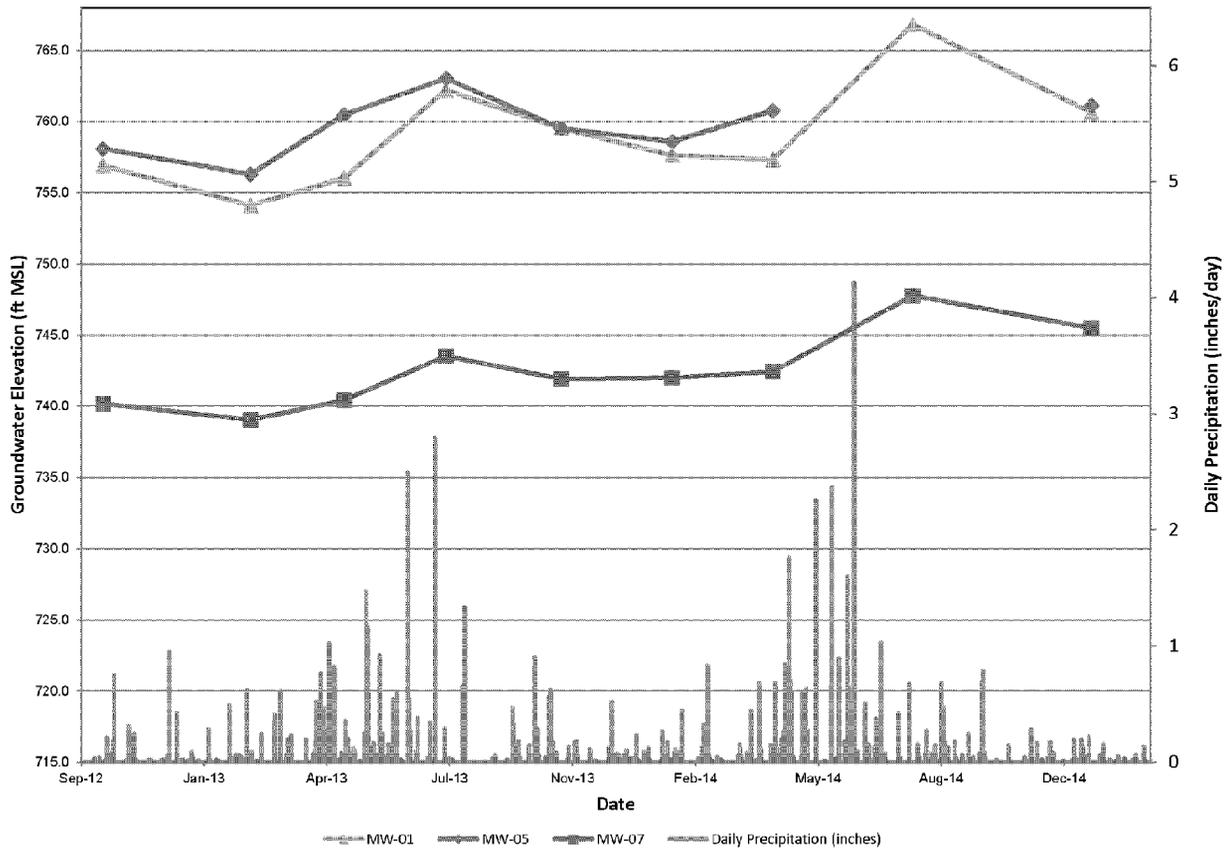


ATTACHMENT C
HYDROGRAPHS FOR SITE MONITORING WELLS

3M_MN01596021

3630.0072

Groundwater Hydrograph
 Monitoring Wells MW-01, MW-05, and MW-07
 October 2012 - December 2014

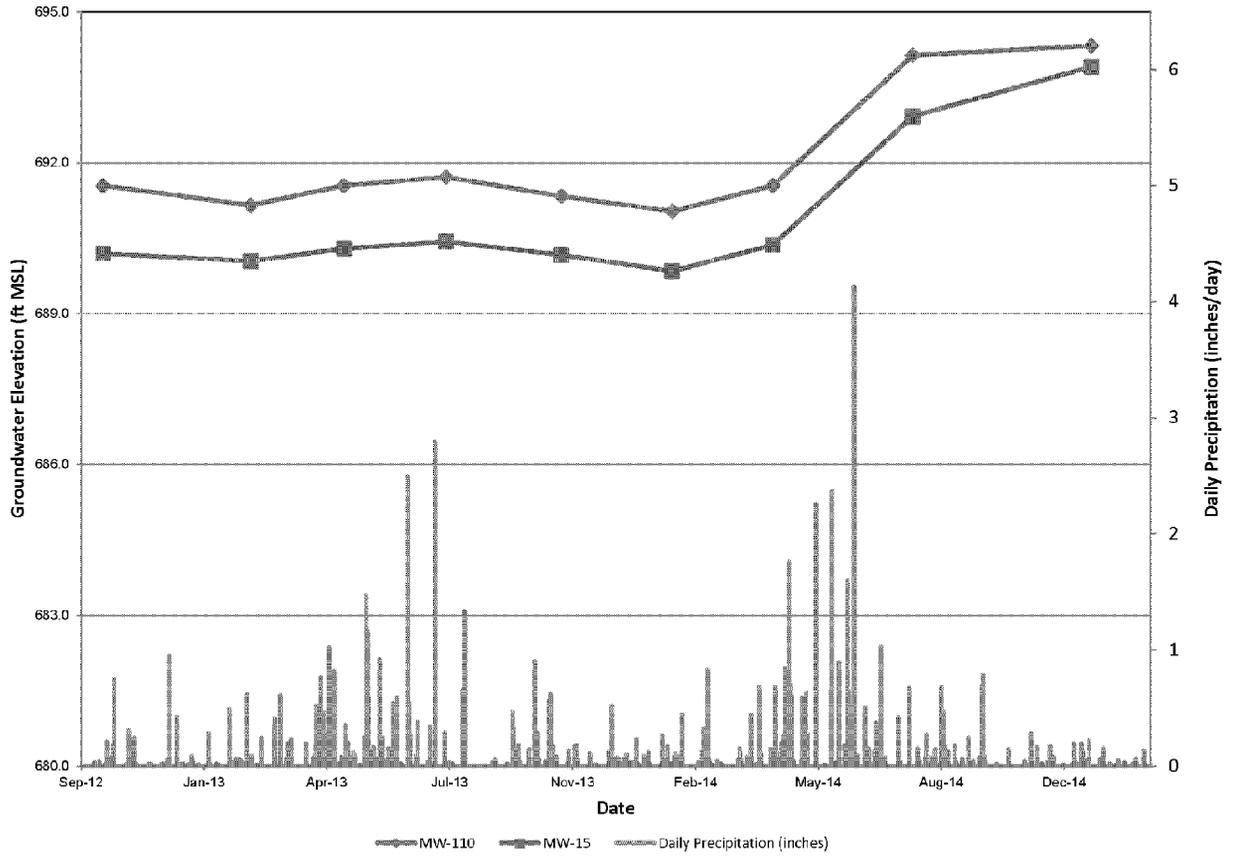


CGMN-CWELEVs-Hydrographs.xlsx\MW-1,-5,-7 CHT

3M_MN01596022

Groundwater Hydrograph

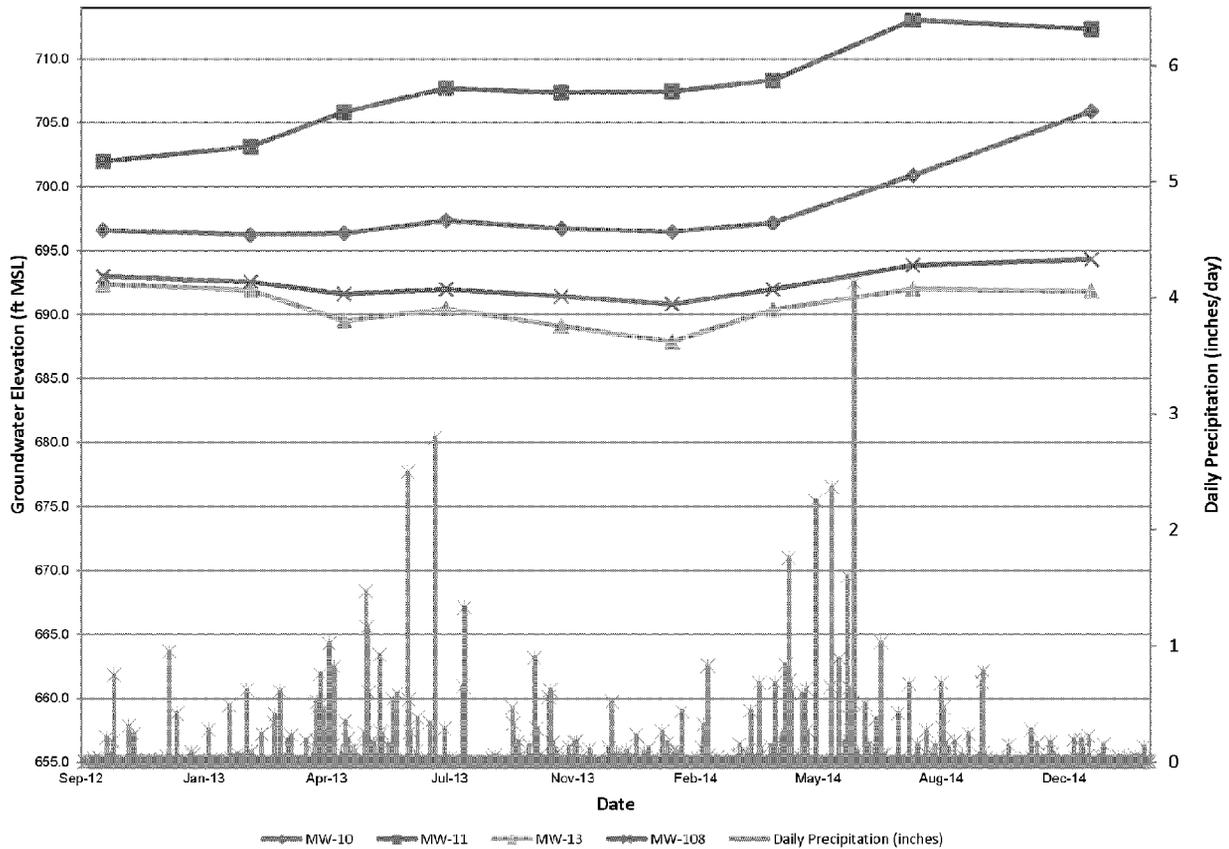
Monitoring Wells MW-110 and MW-15
October 2012 - December 2014



CGMN-GWELEVs-Hydrographs.xlsx\MW-110,-15,-12 CHT

3M_MN01596023

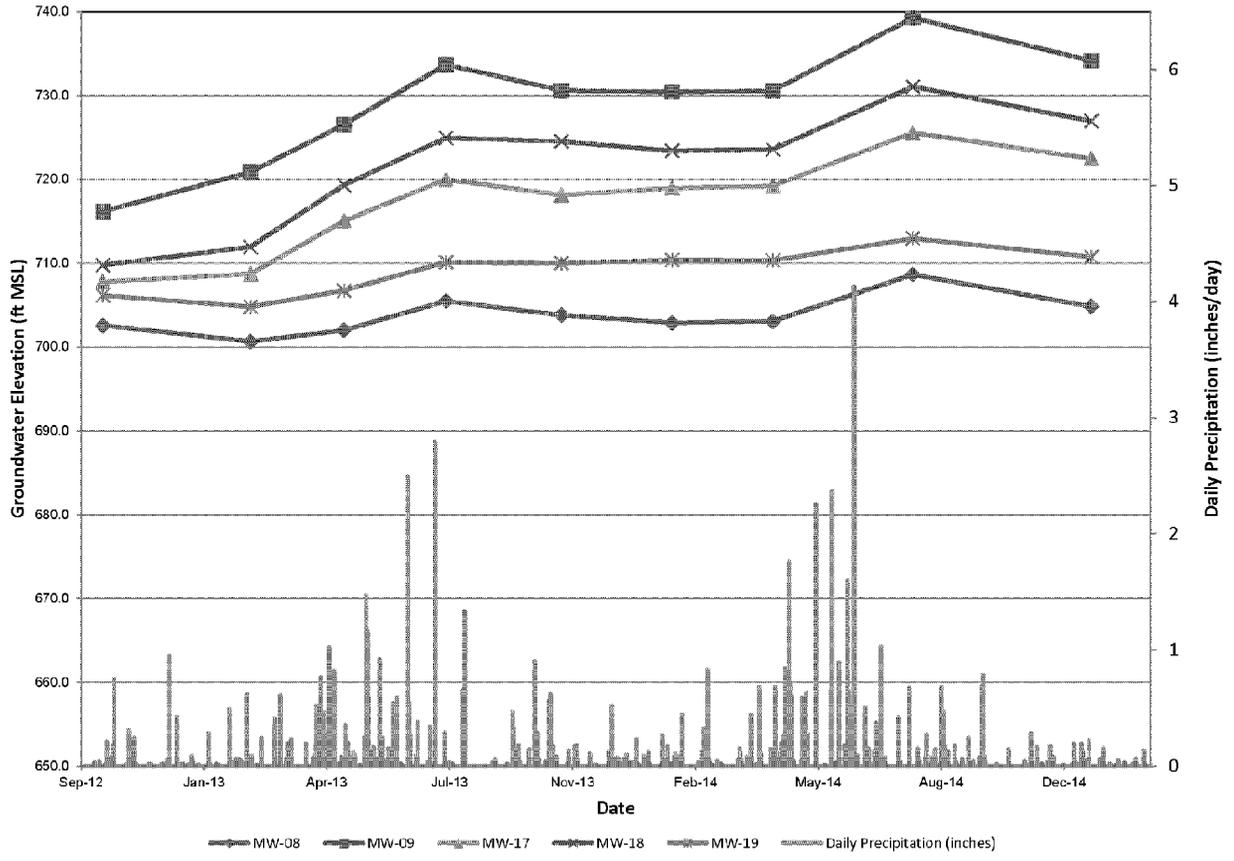
Groundwater Hydrograph
 Monitoring Wells MW-10, MW-11, MW-13 and MW-108
 October 2012 - December 2014



CGMN-GWELEVs-Hydrographs.xlsx\MW-10, -11, -13, -108 CHT

3M_MN01596024

Groundwater Hydrograph
 Monitoring Wells MW-08, MW-09, MW-17, MW-18 and MW-19
 October 2012 -December 2014

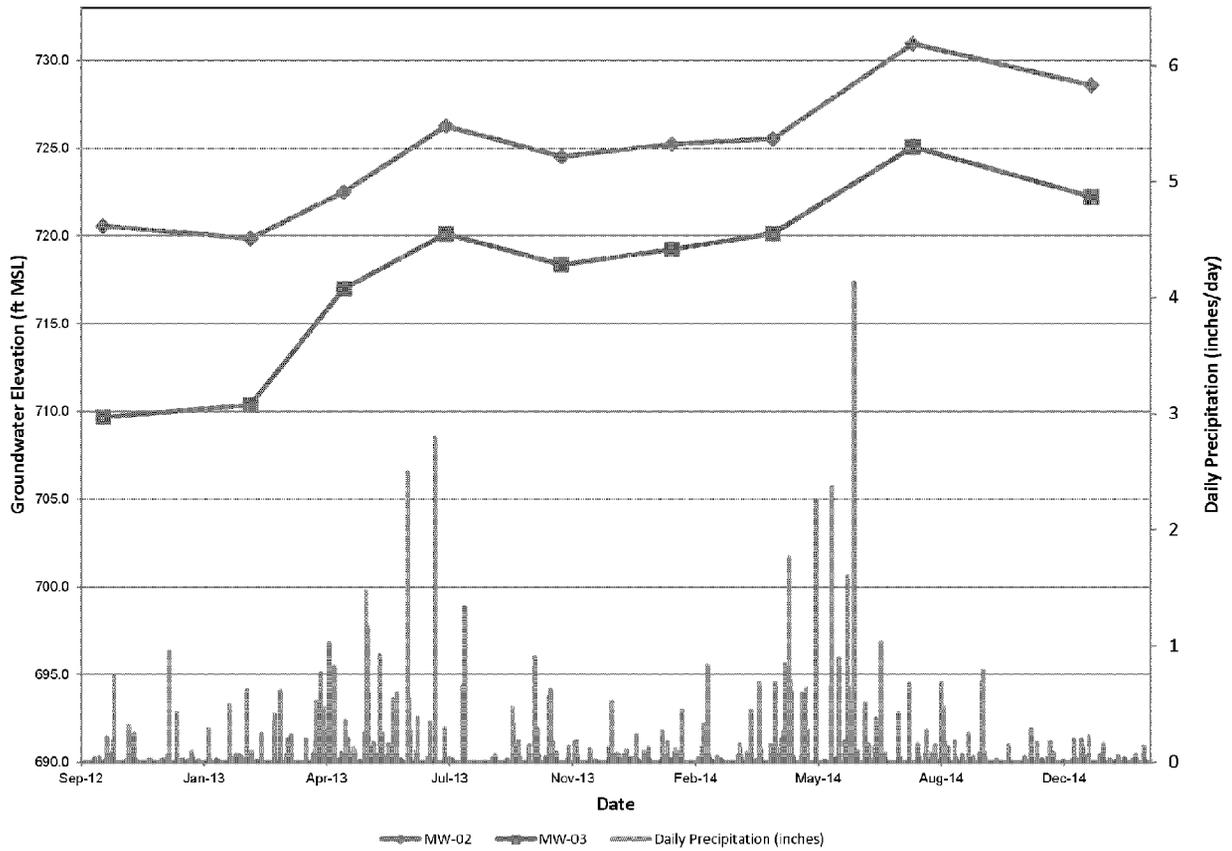


CGMN-GWELVs-Hydrographs.xlsx\MW-8,-9,-17,-18,-19 CHT

3M_MN01596025

Groundwater Hydrograph

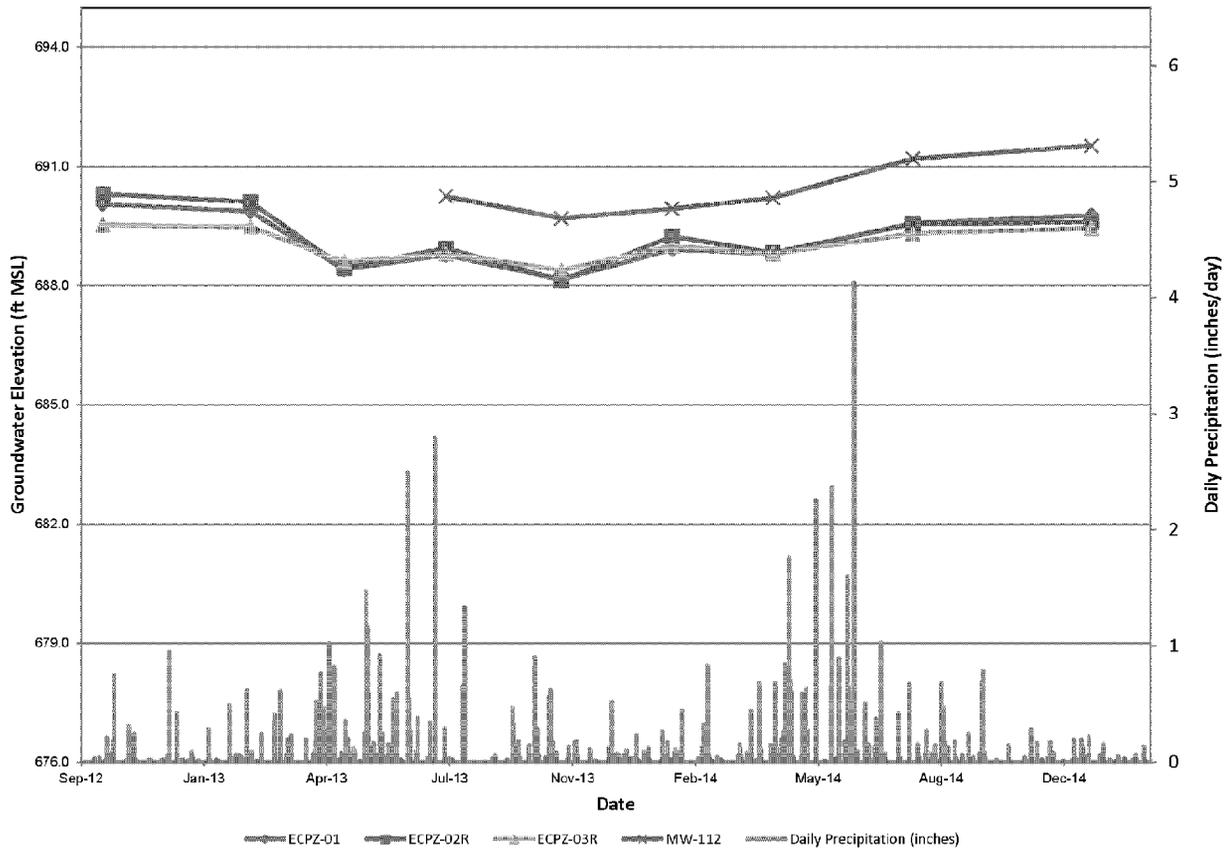
Monitoring Wells MW-02 and MW-03
October 2012 - December 2014



CGMN-GWELEVs-hydrographs.xlsx\MW-2_-3 CHT

3M_MN01596026

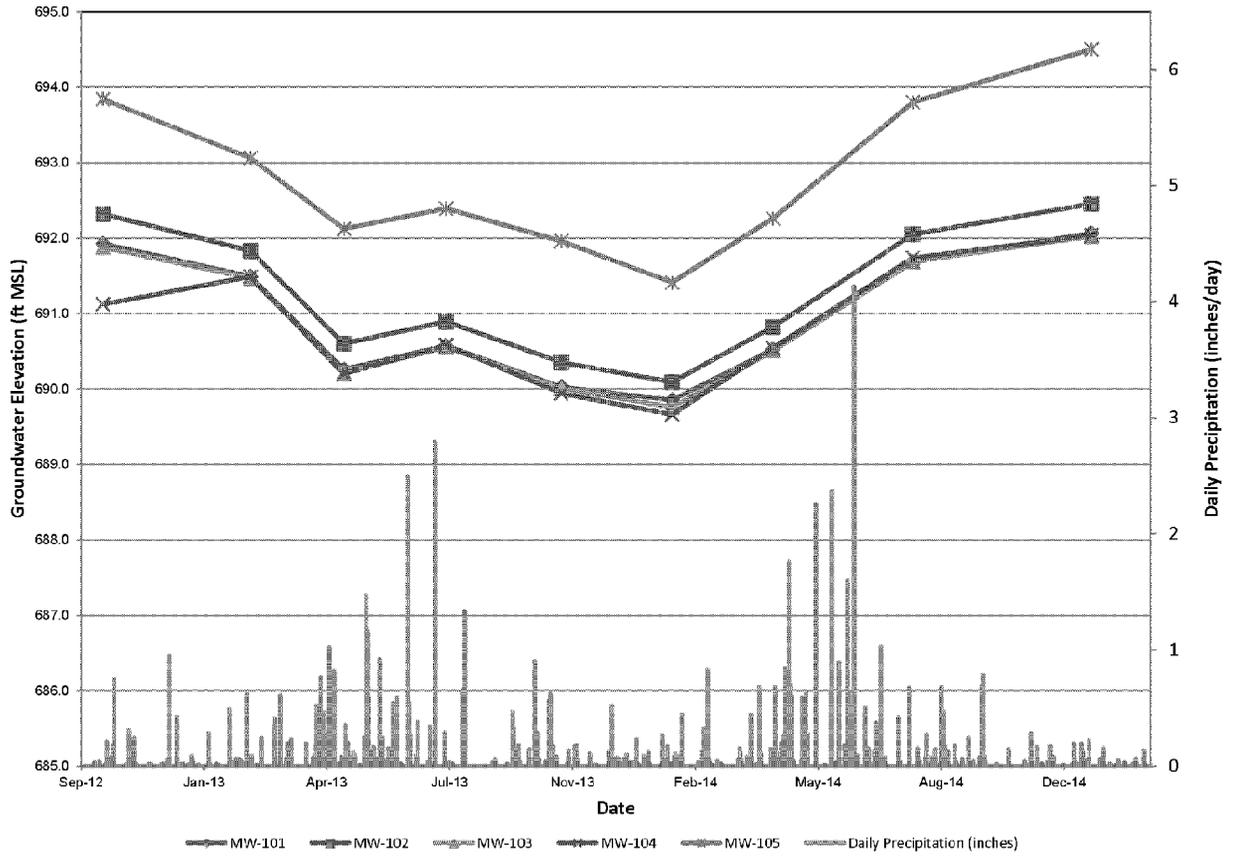
Groundwater Hydrograph
 Monitoring Wells ECPZ-01, ECPZ-02R, ECPZ-03R and MW-112
 October 2012 - December 2014



CGMN-GWE.EVs-Hydrographs.xlsxECPZ-01, -02R, -03R, MW-112 CHT

3M_MN01596027

Groundwater Hydrograph
 Monitoring Wells MW-101, MW-102, MW-103, MW-104 and MW-105
 October 2012 - December 2014

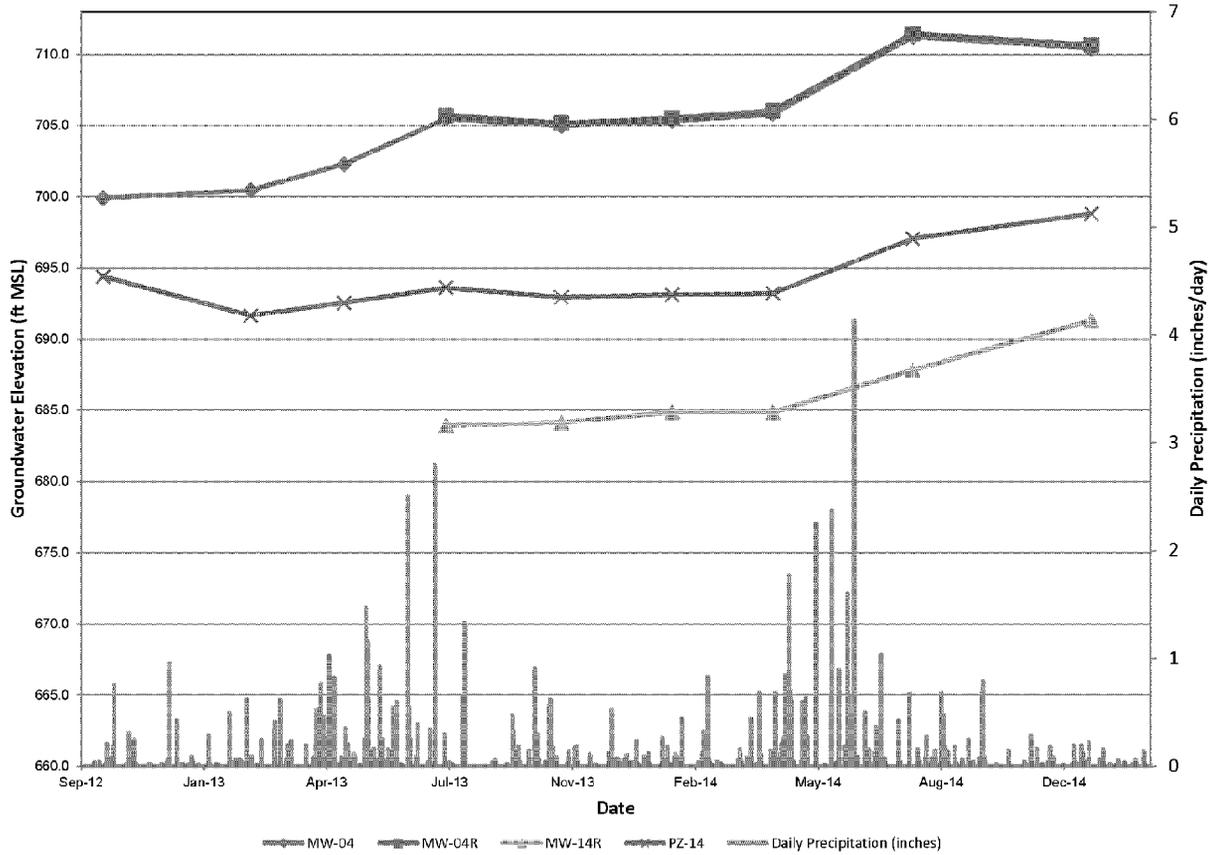


CGM\N-GWELVs-Hydrographs.xlsx\MW-101, -102, -103, -104, -105

3M_MN01596028

Groundwater Hydrograph

Monitoring Wells MW-04, MW-04R, MW-14R and PZ-14
October 2012 - December 2014



CGMN-GWELVs-Hydrographs.xlsx\MW-4, -4R, 14R, PZ-14 CHT

3M_MN01596029



**ATTACHMENT D
LABORATORY ANALYTICAL PACKAGES AND
CHAIN-OF-CUSTODY DOCUMENTATION FOR
GROUNDWATER SAMPLING EVENTS**

3M_MN01596030

3630.0081



JANUARY 2014

Final Report

Analysis of PFBA, PFOA, PFBS, PFHS, and PFOS in Aqueous Samples, Cottage Grove Groundwater Sampling 1st Quarter 2014

Laboratory Request Number: ISO11-01-03-13

Report Date – Date of Last Signature

Testing Laboratory

3M EHS&S Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Gary Hohenstein
3M EHS&S Operations
3M Building 224-5W-03
Saint Paul, MN 55144-1000
Phone: (651) 737-3570



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.

3M Principal Analytical Investigator: Susan Wolf

Report Author: Kevin Eich

Analytical Report ISO11-01-03-13

Analysis of PFBA, PFOA, PFBS, PFHS, and PFOS in Aqueous Samples,
Cottage Grove Groundwater Sampling – 1st Quarter 2014

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater samples collected by Weston Solutions personnel at the 3M Cottage Grove facility. Samples were collected on January 13, 14, 15, 16, and 22, 2014. Samples were returned to the 3M Environmental Laboratory on January 17, 2014 on ice and January 23, 2014 at room temperature for the analysis of Perfluorobutanoic acid (PFBA), Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS) and Perfluorooctane sulfonate (PFOS) under laboratory project number ISO11-01-03-13.

The 3M Environmental Laboratory prepared sample containers for twelve sampling locations. Each sample set consisted of a field sample, field sample duplicate, and a target analyte field matrix spike. Each empty container was marked with a “fill to here” line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with internal standards and surrogate recovery standards prior to being sent to the field for sample collection.

Samples were prepared and analyzed for PFBA, PFOA, PFBS, PFHS, and PFOS using method ETS-8-044.1 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”. Internal standards were used to aid in the data quality objectives for the analysis of select samples, were applicable.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 “General Requirements for the Competence of Testing and Calibration Laboratories”, in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory’s quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
ISO11-01-03-13-001	CGMN-MW07-0-140113	3.01	0.407	<0.100	0.0508	0.104
ISO11-01-03-13-002	CGMN-MW07-DB-140113	2.75	0.373	<0.100	0.0386	0.104
Average		2.88⁽²⁾	0.390⁽²⁾	<0.100⁽²⁾	0.0447⁽²⁾	0.104⁽²⁾
%RPD Sample/Sample Dup		9.0	8.7	NA	27⁽³⁾	0.0
ISO11-01-03-13-004	CGMN-MW12-0-140116	327	682	85.4	14.1	129
ISO11-01-03-13-005	CGMN-MW12-DB-140116	297	653	78.8	12.1	113
Average		312	668	82.1	13.1	121
%RPD Sample/Sample Dup		9.6	4.3	8.0	15	13
ISO11-01-03-13-007	CGMN-MW13-0-140114	9.69	9.65	1.12	0.857	3.02
ISO11-01-03-13-008	CGMN-MW13-DB-140114	9.90	9.90	1.27	1.08	3.14
Average		9.80⁽²⁾	9.78⁽²⁾	1.20⁽²⁾	0.969⁽²⁾	3.08⁽²⁾
%RPD Sample/Sample Dup		2.1	2.6	13	23⁽³⁾	3.9
ISO11-01-03-13-010	CGMN-MW16-0-140114	43.4	93.1	41.1	5.51	51.0
ISO11-01-03-13-011	CGMN-MW16-DB-140114	42.3	88.9	38.8	5.52	55.2
Average		42.9⁽²⁾	91.0⁽²⁾	40.0⁽²⁾	5.52⁽²⁾	53.1⁽²⁾
%RPD Sample/Sample Dup		2.6	4.6	5.8	0.18	7.9
ISO11-01-03-13-013	CGMN-MW101-0-140115	1590	80.0	25.7	383	160
ISO11-01-03-13-014	CGMN-MW101-DB-140115	1430	78.7	25.1	413	158
Average		1510	79.4	25.4	398	159
%RPD Sample/Sample Dup		11	1.6	2.4	7.5	1.3
ISO11-01-03-13-016	CGMN-MW104-0-140115	194	35.1	7.34	14.4	4.22
ISO11-01-03-13-017	CGMN-MW104-DB-140115	210	35.0	7.65	13.9	4.27
Average		202	35.1	7.50	14.2	4.25
%RPD Sample/Sample Dup		7.9	0.29	4.1	3.5	1.2
ISO11-01-03-13-019	CGMN-MW105-0-140114	56.0	68.5	6.48	12.4	152
ISO11-01-03-13-020	CGMN-MW105-DB-140114	55.1	68.0	5.81	12.8	152
Average		55.6	68.3	6.15	12.6	152
%RPD Sample/Sample Dup		1.6	0.73	11	3.2	0.0
ISO11-01-03-13-022	CGMN-MW108-0-140114	48.9	171	18.1	4.19	33.2
ISO11-01-03-13-023	CGMN-MW108-DB-140114	54.2	166	15.9	4.06	33.7
Average		51.6	169	17.0	4.13	33.5
%RPD Sample/Sample Dup		10	3.0	13	3.2	1.5
ISO11-01-03-13-025	CGMN-MW110-0-140114	177	247	88.4	20.2	12.0
ISO11-01-03-13-026	CGMN-MW110-DB-140114	181	248	81.7	18.6	12.2
Average		179	248	85.1	19.4	12.1
%RPD Sample/Sample Dup		2.2	0.40	7.9	8.2	1.7

NA = Not Applicable

- (1) Samples were reported by external standard calibration, except where noted. The analytical method uncertainties associated with the reported results by external standard calibration are as follows: PFBA ± 19%, PFOA ± 16%, PFBS ± 14%, PFHS ± 16%, and PFOS ± 20%.
- (2) Samples were reported by internal standard calibration. The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFBA ± 14%, PFOA ± 16%, PFBS ± 18%, PFHS ± 28%, and PFOS ± 16%.
- (3) The sample/sample dup %RPD did not meet the method criteria of <20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
ISO11-01-03-13-031	CGMN-PW09-0-140115	5.00	1.40	0.167	0.117	2.29
ISO11-01-03-13-032	CGMN-PW09-DB-140115	4.52	1.26	0.178	0.101	1.97
Average		4.76⁽²⁾	1.33⁽²⁾	0.173⁽²⁾	0.109⁽²⁾	2.13⁽²⁾
%RPD Sample/Sample Dup		10	11	6.4	15	15
ISO11-01-03-13-034	CGMN-PW10-0-140122	4.03	0.223	0.254	0.0426	0.121
ISO11-01-03-13-035	CGMN-PW10-DB-140122	4.77	0.200	0.268	0.0543	0.126
Average		4.40⁽²⁾	0.212⁽²⁾	0.261⁽²⁾	0.0485⁽²⁾	0.124
%RPD Sample/Sample Dup		17	11	5.4	24⁽³⁾	4.0
ISO11-01-03-13-037	CGMN-MW14R-0-140113	300	137	17.3	12.1	25.0
ISO11-01-03-13-038	CGMN-MW14R-DB-140113	289	141	17.7	12.4	24.8
Average		295	139	17.5	12.3	24.9
%RPD Sample/Sample Dup		3.7	2.9	2.3	2.4	0.80

NA = Not Applicable

- (1) Samples were reported by external standard calibration, except where noted. The analytical method uncertainties associated with the reported results by external standard calibration are as follows: PFBA ± 19%, PFOA ± 16%, PFBS ± 14%, PFHS ± 16%, and PFOS ± 20%.
 (2) Samples were reported by internal standard calibration. The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFBA ± 14%, PFOA ± 16%, PFBS ± 18%, PFHS ± 28%, and PFOS ± 16%.
 (3) The sample/sample dup %RSD did not meet the method criteria of <20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorobutanoic Acid (C4 Acid)	PFBA	Linear
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched

2.2 Sample Collection

Samples were collected on January 13, 14, 15, 16, and 22, 2014 in Nalgene™ (high-density polyethylene) bottles prepared at the 3M Environmental Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. Collected sample bottles were returned to the laboratory on ice on January 17, 2014 and at room temperature on January 23, 2014.

2.3 Sample Preparation

Sample concentrations were expected to range from <0.025 ng/mL to >1000 ng/mL. Sampling locations that were expected to have concentration <100 ng/mL were analyzed by internal standard calibration analysis. Sampling locations that were expected to have concentration >100 ng/mL were analyzed by external standard calibration analysis. The following sample preparation procedures were followed for each type of analysis.

Internal standard calibration analysis: Samples analyzed by internal standard calibration were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2).

During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples.

External standard calibration analysis: Samples analyzed by external standard calibration required dilution prior to analysis. Samples requiring a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples requiring a 1:50 dilution were prepared by diluting 0.2 mL of a well-mixed sample with 9.8 mL of methanol.

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to generate the reported results:

1/24/14 - Internal standard calibration; **MW07, MW13, MW16, PW09, Rinseate** and **Trip Blanks** (sample and low spike) for PFBA, PFOA, PFBS, PFHS, PFOS and surrogates.

1/28/14 - External standard calibration; **MW12, MW101, MW104, MW105, MW108, MW110, and Trip Blank high spike** for PFBA, PFOA, PFBS, PFHS, and PFOS; **MW14R** for PFOA, PFBS, PFHS, and PFOS.

1/30/14 - Internal standard calibration; **PW10** for PFBA, PFOA, PFBS, PFHS, PFOS and surrogates.

1/31/14 - External standard calibration; **MW14R** for PFBA.

Table 3. Instrument Parameters.

Instrument Name	ETS Buster
Liquid Chromatograph	Agilent 1100
Analysis Method	ETS-8-044.1
Analysis Date	1/24/14, 1/28/14, 1/30/14, 1/31/14
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 μ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 μ
Injection Volume	10, 30, 40 μL
Mass Spectrometer	Applied Biosystems API 4000
Ion Source	Turbo Spray
Polarity	Negative
Software	Analyst 1.6.1

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.1 Analysis				
Step Number	Total Time (min)	Flow Rate (μL/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	750	97.0	3.0
1	0.50	750	97.0	3.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	97.0	3.0
9	19.0	750	97.0	3.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard ⁽¹⁾	Mass Transition Q1/Q3
PFBA	213/169	[¹³ C ₄]-PFBA	217/172
PFOA	413/369	[¹³ C ₈]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/80	[¹⁸ O ₂]-PFBS	303/84
	299/99		
PFHS	399/99	[¹³ C ₃]-PFHS	402/99
	399/80		
PFOS	499/99	[¹³ C ₈]-PFOS	507/80
	499/80		
	499/130		
[¹³ C ₃]-PFBA	216/172	[¹³ C ₄]-PFBA	217/172
[¹³ C ₄]-PFOA	417/372	[¹³ C ₈]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₈]-PFOS	507/80

Dwell time was 50 or 100 msec for each transition. The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standard was not used for the samples analyzed by solvent dilution external standard calibration.

3 Data Analysis

3.1 Calibration

Solvent dilution analysis using internal standard calibration: Samples were analyzed for all analytes against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 methanol:laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. Calibration standards ranging from 0.0125 ng/mL to 50 ng/mL (nominal) were analyzed (0.0125 ng/mL to 10 ng/mL (nominal) for the SRSs). Low or high points may have been disabled to meet method criteria. A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

Solvent dilution analysis using external standard calibration: Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution into 50 mL of 90:10 methanol: laboratory Milli-Q™ water. Calibration standards ranging from 0.1 ng/mL to 100 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. Low or high points were disabled to meet method criteria. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

For both method of analysis, each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area counts or peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes with the following exceptions:

1/24/14 Analysis: The system suitability area counts exceeded 5% for PFBA (7.7%), PFBS (6.5%), and PFHS (6.3%).

1/30/14 Analysis: The system suitability area counts exceeded 5% PFBA (7.3%), PFBS (5.6%), and ¹³C₄-PFOA (7.1%).

A method deviation is included with the raw data.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.1 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the Table 6 below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 1/24/14 Analysis	LOQ, ng/mL ⁽²⁾ 1/28/14 Analysis	LOQ, ng/mL ⁽¹⁾ 1/30/14 Analysis	LOQ, ng/mL ⁽²⁾ 1/31/14 Analysis
PFBA	0.200	0.250	0.0500	0.250
PFOA	0.0480	0.0958	0.0240	NA
PFBS	0.100	0.100	0.0500	NA
PFHS	0.0250	0.100	0.0250	NA
PFOS	0.0232	0.0927	0.0232	NA

NA = Not Applicable

(1) A dilution factor of 2 was applied to the LOQ.

(2) A dilution factor was not applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±25%.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: method/solvent blanks, field/trip blanks, and sampling equipment blanks. Each blank result was reviewed and used to evaluate method performance. The method/solvent blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs prepared for internal standard calibration analysis were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. LCSs prepared for external standard calibration analysis were prepared by spiking known amounts of the analytes into 1.0 mL of laboratory reagent water and 9.0 mL of methanol to produce the desired concentration. Method ETS-8-044.1 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria with the following exceptions:

- 1/24/14 internal standard analysis: The average recovery of the mid set of LCS for PFHS was 128%.
- 1/30/14 internal standard analysis: The average recovery of the low set of LCS for ¹³C₃-PFBA was 128%.

A method deviation is included with the raw data for those LCSs that did not meet method acceptance criteria. All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report.

The following calculations were used to generate data in Table 7.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%$$

$$\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%$$

Table 7. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 1/24/14	PFBA			PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140122-1	0.198	<0.200	NA	0.190	0.211	111
LCS-140122-2	0.198	<0.200	NA	0.190	0.181	95.1
LCS-140122-3	0.198	<0.200	NA	0.190	0.178	93.4
Average ± %RSD	NA			99.8% ± 9.7%		
LCS-140122-4	9.92	11.7	118	9.50	11.4	120
LCS-140122-5	9.92	10.9	110	9.50	10.7	112
LCS-140122-6	9.92	12.1	122	9.50	11.8	124
Average ± %RSD	117% ± 5.2%			119% ± 5.1%		
LCS-140122-7	39.6	43.4	110	38.0	40.0	105
LCS-140122-8	39.6	40.5	102	38.0	37.3	98.3
LCS-140122-9	39.6	44.2	112	38.0	40.6	107
Average ± %RSD	108% ± 4.9%			103% ± 4.4%		

NA = Not Applicable

(1) LCSs average recovery did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 1/24/14	PFBS			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140122-1	0.198	0.174	87.7	0.198	0.254	128
LCS-140122-2	0.198	0.200	101	0.198	0.237	120
LCS-140122-3	0.198	0.144	72.9	0.198	0.205	103
Average ± %RSD	87.2% ± 16%			117% ± 11%		
LCS-140122-4	9.92	11.8	118	9.92	13.7	138
LCS-140122-5	9.92	9.76	98.4	9.92	12.2	123
LCS-140122-6	9.92	10.5	106	9.92	12.1	122
Average ± %RSD	107% ± 9.2%			128% ± 7.0%⁽¹⁾		
LCS-140122-7	39.6	44.6	113	39.6	42.3	107
LCS-140122-8	39.6	39.3	99.2	39.6	46.3	117
LCS-140122-9	39.6	41.4	105	39.6	48.2	122
Average ± %RSD	106% ± 6.6%			115% ± 6.6%		

ETS-8-044.1 Internal standard calibration Analyzed 1/24/14	PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140122-1	0.184	0.188	102
LCS-140122-2	0.184	0.212	115
LCS-140122-3	0.184	0.208	113
Average ± %RSD	110% ± 6.4%		
LCS-140122-4	9.20	10.7	117
LCS-140122-5	9.20	9.45	103
LCS-140122-6	9.20	10.2	111
Average ± %RSD	110% ± 6.4%		
LCS-140122-7	36.7	40.9	111
LCS-140122-8	36.7	39.0	106
LCS-140122-9	36.7	39.3	107
Average ± %RSD	108% ± 2.4%		

NA = Not Applicable

(1) LCSs average recovery did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 1/24/14						
¹³ C ₃ -PFBA				¹³ C ₄ -PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140122-1	0.197	0.231	117	0.198	0.252	127
LCS-140122-2	0.197	0.221	112	0.198	0.214	108
LCS-140122-3	0.197	0.231	117	0.198	0.214	108
Average ± %RSD	115% ± 2.5%			114% ± 9.6%		
LCS-140122-4	1.97	2.34	119	1.98	2.29	116
LCS-140122-5	1.97	2.11	107	1.98	2.43	123
LCS-140122-6	1.97	2.14	108	1.98	2.39	121
Average ± %RSD	111% ± 6.0%			120% ± 3.0%		

ETS-8-044.1 Internal standard calibration Analyzed 1/24/14			
¹³ C ₄ -PFOS			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140122-1	0.189	0.222	117
LCS-140122-2	0.189	0.225	119
LCS-140122-3	0.189	0.235	125
Average ± %RSD	120% ± 3.5%		
LCS-140122-4	1.90	2.27	120
LCS-140122-5	1.90	2.13	112
LCS-140122-6	1.90	2.25	118
Average ± %RSD	117% ± 3.6%		

NA = Not Applicable

(1) LCSs average recovery did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External standard calibration Analyzed 1/28/14						
PFBA				PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140128-1	1.00	0.980	98.0	0.958	0.923	96.3
LCS-140128-2	1.00	0.954	95.4	0.958	0.890	92.9
LCS-140128-3	1.00	0.837	83.7	0.958	0.832	86.9
Average ± %RSD	92.4% ± 8.2%			92.0% ± 5.2%		
LCS-140128-4	10.0	10.6	106	9.58	9.52	99.3
LCS-140128-5	10.0	10.3	103	9.58	9.32	97.3
LCS-140128-6	10.0	9.87	98.7	9.58	9.42	98.3
Average ± %RSD	103% ± 3.6%			98.3% ± 1.0%		
LCS-140128-7	39.8	35.4	89.1	38.2	33.4	87.4
LCS-140128-8	39.8	37.6	94.5	38.2	33.9	88.8
LCS-140128-9	39.8	38.6	97.0	38.2	36.6	95.7
Average ± %RSD	93.5% ± 4.3%			90.6% ± 4.9%		

ETS-8-044.1 External standard calibration Analyzed 1/28/14						
PFBS				PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140128-1	1.00	0.986	98.6	1.00	1.05	105
LCS-140128-2	1.00	1.01	101	1.00	0.905	90.5
LCS-140128-3	1.00	0.929	92.9	1.00	0.925	92.5
Average ± %RSD	97.5% ± 4.3%			96.0% ± 8.2%		
LCS-140128-4	10.0	10.1	101	10.0	10.7	107
LCS-140128-5	10.0	10.1	101	10.0	10.1	101
LCS-140128-6	10.0	10.2	102	10.0	10.0	100
Average ± %RSD	101% ± 0.57%			103% ± 3.7%		
LCS-140128-7	39.8	34.9	87.6	39.8	36.2	91.0
LCS-140128-8	39.8	36.9	92.7	39.8	37.9	95.3
LCS-140128-9	39.8	39.2	98.5	39.8	38.7	97.1
Average ± %RSD	92.9% ± 5.9%			94.5% ± 3.3%		

NA = Not Applicable

(1) LCSs average recovery did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External standard calibration Analyzed 1/28/14			
PFOS (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140128-1	0.927	0.880	94.9
LCS-140128-2	0.927	0.842	90.9
LCS-140128-3	0.927	0.808	87.1
Average ± %RSD	91.0% ± 4.3%		
LCS-140128-4	9.27	8.85	95.4
LCS-140128-5	9.27	8.84	95.4
LCS-140128-6	9.27	8.98	96.8
Average ± %RSD	95.9% ± 0.84%		
LCS-140128-7	36.9	32.8	88.8
LCS-140128-8	36.9	34.0	92.1
LCS-140128-9	36.9	36.1	97.9
Average ± %RSD	92.9% ± 5.0%		

ETS-8-044.1 Internal standard calibration Analyzed 1/30/14				PFOA (Linear + Branched)		
PFBA				PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140130-1	0.198	0.242	122	0.190	0.188	98.9
LCS-140130-2	0.198	0.213	108	0.190	0.200	105
LCS-140130-3	0.198	0.233	118	0.190	0.218	115
Average ± %RSD	116% ± 6.2%			106% ± 7.6%		
LCS-140130-4	9.92	11.8	119	9.50	11.0	115
LCS-140130-5	9.92	11.1	112	9.50	9.87	104
LCS-140130-6	9.92	11.0	111	9.50	10.9	115
Average ± %RSD	114% ± 3.8%			111% ± 5.7%		
LCS-140130-7	39.6	42.9	108	38.0	41.0	108
LCS-140130-8	39.6	41.6	105	38.0	39.0	103
LCS-140130-9	39.6	45.7	116	38.0	38.9	102
Average ± %RSD	110% ± 5.2%			104% ± 3.1%		

NA = Not Applicable

(1) LCSs average recovery did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 1/30/14						
Lab ID	PFBS			PFHS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140130-1	0.198	0.228	115	0.198	0.182	92.2
LCS-140130-2	0.198	0.182	91.7	0.198	0.261	132
LCS-140130-3	0.198	0.176	88.7	0.198	0.238	120
Average ± %RSD	98.5% ± 15%			115% ± 18%		
LCS-140130-4	9.92	11.0	111	9.92	11.3	114
LCS-140130-5	9.92	11.0	111	9.92	10.4	105
LCS-140130-6	9.92	11.1	112	9.92	10.5	106
Average ± %RSD	111% ± 0.52%			108% ± 4.6%		
LCS-140130-7	39.6	38.4	97.1	39.6	39.9	101
LCS-140130-8	39.6	37.9	95.6	39.6	44.0	111
LCS-140130-9	39.6	40.0	101	39.6	40.7	103
Average ± %RSD	97.9% ± 2.8%			105% ± 5.0%		

ETS-8-044.1 Internal standard calibration Analyzed 1/30/14			
Lab ID	PFOS (Linear + Branched)		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140130-1	0.184	0.192	105
LCS-140130-2	0.184	0.207	113
LCS-140130-3	0.184	0.211	115
Average ± %RSD	111% ± 4.8%		
LCS-140130-4	9.20	11.0	120
LCS-140130-5	9.20	10.9	118
LCS-140130-6	9.20	10.8	117
Average ± %RSD	118% ± 1.3%		
LCS-140130-7	36.7	41.3	112
LCS-140130-8	36.7	40.7	111
LCS-140130-9	36.7	39.5	108
Average ± %RSD	110% ± 1.9%		

NA = Not Applicable

(1) LCSs average recovery did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 1/30/14						
¹³ C ₃ -PFBA				¹³ C ₄ -PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140130-1	0.197	0.236	120	0.198	0.217	109
LCS-140130-2	0.197	0.246	125	0.198	0.230	116
LCS-140130-3	0.197	0.274	139	0.198	0.262	132
Average ± %RSD	128% ± 7.7%⁽¹⁾			119% ± 9.9%		
LCS-140130-4	1.97	2.36	120	1.98	2.22	112
LCS-140130-5	1.97	2.24	114	1.98	1.96	98.9
LCS-140130-6	1.97	2.20	112	1.98	2.33	118
Average ± %RSD	115% ± 3.6%			110% ± 8.9%		

ETS-8-044.1 Internal standard calibration Analyzed 1/30/14			
¹³ C ₄ -PFOS			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140130-1	0.189	0.221	117
LCS-140130-2	0.189	0.207	109
LCS-140130-3	0.189	0.238	126
Average ± %RSD	117% ± 7.2%		
LCS-140130-4	1.90	2.02	106
LCS-140130-5	1.90	2.19	115
LCS-140130-6	1.90	2.08	110
Average ± %RSD	110% ± 4.1%		

NA = Not Applicable

(1) LCSs average recovery did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External standard calibration Analyzed 1/31/14	PFBA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140131-1	1.00	1.05	105
LCS-140131-2	1.00	0.985	98.5
LCS-140131-3	1.00	0.803	80.3
Average ± %RSD	94.6% ± 14%		
LCS-140131-4	10.0	9.94	99.4
LCS-140131-5	10.0	10.3	103
LCS-140131-6	10.0	10.4	104
Average ± %RSD	102% ± 2.4%		
LCS-140131-7	39.8	41.4	104
LCS-140131-8	39.8	39.2	98.6
LCS-140131-9	39.8	39.2	98.5
Average ± %RSD	100% ± 3.1%		

NA = Not Applicable

(1) LCSs average recovery did not meet acceptance criteria of 100 ± 20%.

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.1, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

The analytical method uncertainty as calculated by ETS-12-012.2 for PFHS by internal standard calibration was ±24%. However, the recovery of the mid set of LCS analyzed on 1/24/14 was 128% for PFHS by internal standard calibration for the samples analyzed on this date. Therefore, the analytical method uncertainty was expanded for samples analyzed by internal standard calibration for PFHS to ±28%.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFBA	Internal	7.15	±14%
PFOA	Internal	7.96	±16%
PFBS	Internal	8.90	±18%
PFHS	Internal	NA	±28%
PFOS	Internal	7.97	±16%
PFBA	External	9.59	±19%
PFOA	External	7.88	±16%
PFBS	External	7.15	±14%
PFHS	External	8.10	±16%
PFOS	External	9.99	±20%

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample was collected at each sampling point to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

$$\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%$$

Table 9. Field and Lab Matrix Spike Concentrations

Location	Spike Level	Final Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
MW07, PW09, and PW10	FMS	2.00	1.92	2.00	2.00	1.85
MW13	FMS	5.00	4.79	5.00	5.00	4.64
MW16	FMS	24.9	24.8	25.0	25.0	25.0
MW101, MW104, MW105, MW108, MW110, and MW14R	FMS	99.4	99.2	100	100	99.8
MW12	FMS	497	496	500	500	499
Trip Blank	Low	2.00	1.92	2.00	2.00	1.85
	High	99.4	99.2	100	100	99.8

3.9 Lab Matrix Spikes (LMS)

The field matrix spike level prepared for location MW14R was not sufficient for PFBA. Since this was the first sampling of this location, a laboratory matrix spike (LMS) sample was prepared for PFBA to verify that the analytical method is applicable to the collected matrix. The LMS sample was generated by adding a measured volume of standard solution to an aliquot of the primary sample. Since the MW14R samples required dilution prior to sample analysis, the spike amount added was based on the on-column instrument concentration. The actual spike concentration for PFBA is presented in Table 10.

LMS recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the extraction and analysis of the analytes of interest. LMS concentrations must be 50% of the sample concentration to be considered an appropriate field spike. LMSs are presented in section 4 of this report.

The following calculation was used to calculate the lab matrix spike recovery in Section 4 of the report:

$$\text{LMS Recovery} = \frac{(\text{Sample Concentration of LMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%$$

Table 10. Lab Matrix Spike Levels.

Sampling Location	PFBA Final Concentration (ng/mL)
MW14R	297

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

All field matrix spike and surrogate recovery spikes (where applicable) met method acceptance criteria with the following exceptions:

CGMN GW MW13: The FMS level was not appropriate for PFOA. The FMS spike met method acceptance criteria for the other target analytes and surrogate recoveries met method acceptance criteria. No additional QC samples were prepared for PFOA.

CGMN GW MW16: The FMS level was not appropriate for PFOA and PFOS. The FMS spike met method acceptance criteria for the other target analytes and surrogate recoveries met method acceptance criteria. No additional QC samples were prepared for PFOA and PFOS.

CGMN GW MW101: The FMS level was not appropriate for PFBA and PFHS. The FMS spike met method acceptance criteria for the other target analytes. No additional QC samples were prepared for PFBA and PFHS.

CGMN GW MW110: The FMS level was not appropriate for PFOA. The FMS spike met method acceptance criteria for the other target analytes. No additional QC samples were prepared for PFOA.

CGMN GW PW09: The FMS level was not appropriate for PFBA. The FMS spike met method acceptance criteria for the other target analytes and surrogate recoveries met method acceptance criteria. No additional QC samples were prepared for PFBA.

CGMN GW PW10: The FMS level was not appropriate for PFBA. The FMS spike met method acceptance criteria for the other target analytes and surrogate recoveries met method acceptance criteria. No additional QC samples were prepared for PFBA.

CGMN GW MW14R: The FMS level was not appropriate for PFBA. The FMS spike met method acceptance criteria for the other target analytes and an additional laboratory matrix spike was prepared and met method acceptance criteria for PFBA.

Table 11. CGMN GW MW07 140113

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-001	CGMN-MW07-0-140113	3.01	NA	0.407	NA
ISO11-01-03-13-002	CGMN-MW07-DB-140113	2.75	NA	0.373	NA
ISO11-01-03-13-003	CGMN-MW07-FMS-140113	5.04	108	2.35	102
Average Concentration (ng/mL) ± %RPD		2.88 ng/mL ± 9.0%		0.390 ng/mL ± 8.7%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-001	CGMN-MW07-0-140113	<0.100	NA	0.0508	NA	0.104	NA
ISO11-01-03-13-002	CGMN-MW07-DB-140113	<0.100	NA	0.0386	NA	0.104	NA
ISO11-01-03-13-003	CGMN-MW07-FMS-140113	2.08	104	2.09	102	1.82	92.6
Average Concentration (ng/mL) ± %RPD		<0.100 ng/mL		0.0447 ng/mL ± 27%⁽¹⁾		0.104 ng/mL ± 0.0%	

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₂ -PFOA	¹³ C ₂ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-13-001	CGMN-MW07-0-140113	105	99.4	112
ISO11-01-03-13-002	CGMN-MW07-DB-140113	99.1	98.6	85.4
ISO11-01-03-13-003	CGMN-MW07-FMS-140113	92.8	112	97.0
Average Recovery (%) ± %RSD		98.8% ± 6.0%	103% ± 7.3%	98.2% ± 14%

NA = Not Applicable
 Samples were diluted 1:1 with methanol and analyzed by internal standard calibration.
 (1) Sample/sample duplicate %RPD did not meet method acceptance criteria of <20%.

Table 12. CGMN GW MW12 140116

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-004	CGMN-MW12-0-140116	327	NA	682	NA
ISO11-01-03-13-005	CGMN-MW12-DB-140116	297	NA	653	NA
ISO11-01-03-13-006	CGMN-MW12-FMS-140116	866	111	1190	105
Average Concentration (ng/mL) ± %RPD		312 ng/mL ± 9.6%		668 ng/mL ± 4.3%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-004	CGMN-MW12-0-140116	85.4	NA	14.1	NA	129	NA
ISO11-01-03-13-005	CGMN-MW12-DB-140116	78.8	NA	12.1	NA	113	NA
ISO11-01-03-13-006	CGMN-MW12-FMS-140116	588	101	505	98.4	614	98.8
Average Concentration (ng/mL) ± %RPD		82.1 ng/mL ± 8.0%		13.1 ng/mL ± 15%		121 ng/mL ± 13%	

NA = Not Applicable
 Samples were diluted 1:50 and analyzed by external standard calibration.

Table 13. CGMN GW MW13 140114

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-007	CGMN-MW13-0-140114	9.69	NA	9.65	NA
ISO11-01-03-13-008	CGMN-MW13-DB-140114	9.90	NA	9.90	NA
ISO11-01-03-13-009	CGMN-MW13-FMS-140114	13.8	80.1	13.4	NC
Average Concentration (ng/mL) ± %RPD		9.80 ng/mL ± 2.1%		9.78 ng/mL ± 2.6%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-007	CGMN-MW13-0-140114	1.12	NA	0.857	NA	3.02	NA
ISO11-01-03-13-008	CGMN-MW13-DB-140114	1.27	NA	1.08	NA	3.14	NA
ISO11-01-03-13-009	CGMN-MW13-FMS-140114	6.54	107	6.03	101	7.53	96.0
Average Concentration (ng/mL) ± %RPD		1.20 ng/mL ± 13%		0.969 ng/mL ± 23%⁽¹⁾		3.08 ng/mL ± 3.9%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-13-007	CGMN-MW13-0-140114	103	108	89.9
ISO11-01-03-13-008	CGMN-MW13-DB-140114	96.8	118	97.7
ISO11-01-03-13-009	CGMN-MW13-FMS-140114	88.3	98.3	94.3
Average Recovery (%) ± %RSD		95.9% ± 7.4%	108% ± 9.1%	94.0% ± 4.1%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 with methanol and analyzed by internal standard calibration.
 (1) Sample/sample duplicate %RPD did not meet method acceptance criteria of <20%.

Table 14. CGMN GW MW16 140114

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-0'0	CGMN-MW16-0-140114	43.4	NA	93.1	NA
ISO11-01-03-13-0'1	CGMN-MW16-DB-140114	42.3	NA	88.9	NA
ISO11-01-03-13-012	CGMN-MW16-FMS-140114	63.2	81.9	>ULOQ	NC
Average Concentration (ng/mL) ± %RPD		42.9 ng/mL ± 2.6%		91.0 ng/mL ± 4.6%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-0'0	CGMN-MW16-0-140114	41.1	NA	5.51	NA	51.0	NA
ISO11-01-03-13-0'1	CGMN-MW16-DB-140114	38.8	NA	5.52	NA	55.2	NA
ISO11-01-03-13-012	CGMN-MW16-FMS-140114	65.3	101	32.8	109	78.0	NC
Average Concentration (ng/mL) ± %RPD		40.0 ng/mL ± 5.8%		5.52 ng/mL ± 0.18%		53.1 ng/mL ± 7.9%	

3M LIMS ID	Description	¹² C ₂ -PFBA	¹² C ₄ -PFOA	¹² C ₂ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-13-0'0	CGMN-MW16-0-140114	92.7	91.0	75.3
ISO11-01-03-13-0'1	CGMN-MW16-DB-140114	88.9	95.3	88.8
ISO11-01-03-13-012	CGMN-MW16-FMS-140114	80.0	101	95.8
Average Recovery (%) ± %RSD		87.2% ± 7.5%	95.8% ± 5.2%	86.6% ± 12%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 with methanol and analyzed by internal standard calibration.

Table 15. CGMN GW MW101 140115

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-0' 3	CGMN-MW101-0-140115	1590	NA	80.0	NA
ISO11-01-03-13-0' 4	CGMN-MW101-DB-140115	1430	NA	78.7	NA
ISO11-01-03-13-0' 5	CGMN-MW101-FMS-140115	1530	NC	164	85.3
Average Concentration (ng/mL) ± %RPD		1510 ng/mL ± 11%		79.4 ng/mL ± 1.6%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-0' 3	CGMN-MW101-0-140115	25.7	NA	383	NA	160	NA
ISO11-01-03-13-0' 4	CGMN-MW101-DB-140115	25.1	NA	413	NA	158	NA
ISO11-01-03-13-0' 5	CGMN-MW101-FMS-140115	117	91.6	477	NC	240	81.2
Average Concentration (ng/mL) ± %RPD		25.4 ng/mL ± 2.4%		398 ng/mL ± 7.5%		159 ng/mL ± 1.3%	

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:50 and analyzed by external standard calibration.

Table 16. CGMN GW MW104 140115

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-0'6	CGMN-MW104-0-140115	194	NA	35.1	NA
ISO11-01-03-13-0'7	CGMN-MW104-DB-140115	210	NA	35.0	NA
ISO11-01-03-13-0'8	CGMN-MW104-FMS-140115	285	83.5	129	94.7
Average Concentration (ng/mL) ± %RPD		202 ng/mL ± 7.9%		35.1 ng/mL ± 0.29%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-0'6	CGMN-MW104-0-140115	7.34	NA	14.4	NA	4.22	NA
ISO11-01-03-13-0'7	CGMN-MW104-DB-140115	7.65	NA	13.9	NA	4.27	NA
ISO11-01-03-13-0'8	CGMN-MW104-FMS-140115	111	104	114	99.9	96.2	92.1
Average Concentration (ng/mL) ± %RPD		7.50 ng/mL ± 4.1%		14.2 ng/mL ± 3.5%		4.25 ng/mL ± 1.2%	

NA = Not Applicable
 Samples were diluted 1:10 and analyzed by external standard calibration.

Table 17. CGMN GW MW105 140114

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-019	CGMN-MW105-0-140114	56.0	NA	68.5	NA
ISO11-01-03-13-020	CGMN-MW105-DB-140114	55.1	NA	68.0	NA
ISO11-01-03-13-021	CGMN-MW105-FMS-140114	152	97.0	156	88.5
Average Concentration (ng/mL) ± %RPD		55.6 ng/mL ± 1.6%		68.3 ng/mL ± 0.73%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-019	CGMN-MW105-0-140114	6.48	NA	12.4	NA	152	NA
ISO11-01-03-13-020	CGMN-MW105-DB-140114	5.81	NA	12.8	NA	152	NA
ISO11-01-03-13-021	CGMN-MW105-FMS-140114	101	94.9	106	93.4	244	92.2
Average Concentration (ng/mL) ± %RPD		6.15 ng/mL ± 11%		12.6 ng/mL ± 3.2%		152 ng/mL ± 0.0%	

NA = Not Applicable
 Samples were diluted 1:10 and analyzed by external standard calibration.

Table 18. CGMN GW MW108 140114

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-022	CGMN-MW108-0-140114	48.9	NA	171	NA
ISO11-01-03-13-023	CGMN-MW108-DB- 40114	54.2	NA	166	NA
ISO11-01-03-13-024	CGMN-MW108-FMS-140114	141	90.0	264	96.3
Average Concentration (ng/mL) ± %RPD		51.6 ng/mL ± 10%		169 ng/mL ± 3.0%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-022	CGMN-MW108-0-140114	18.1	NA	4.19	NA	33.2	NA
ISO11-01-03-13-023	CGMN-MW108-DB- 40114	15.9	NA	4.06	NA	33.7	NA
ISO11-01-03-13-024	CGMN-MW108-FMS-140114	124	107	106	102	134	101
Average Concentration (ng/mL) ± %RPD		17.0 ng/mL ± 13%		4.13 ng/mL ± 3.2%		33.5 ng/mL ± 1.5%	

NA = Not Applicable
 Samples were diluted 1:10 and analyzed by external standard calibration.

Table 19. CGMN GW MW110 140114

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-025	CGMN-MW110-0-140114	177	NA	247	NA
ISO11-01-03-13-026	CGMN-MW110-DB-140114	181	NA	248	NA
ISO11-01-03-13-027	CGMN-MW110-FMS-140114	305	127	340	NC
Average Concentration (ng/mL) ± %RPD		179 ng/mL ± 2.2%		248 ng/mL ± 0.40%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-025	CGMN-MW110-0-140114	88.4	NA	20.2	NA	12.0	NA
ISO11-01-03-13-026	CGMN-MW110-DB-140114	81.7	NA	18.6	NA	12.2	NA
ISO11-01-03-13-027	CGMN-MW110-FMS-140114	192	107	125	106	106	94.1
Average Concentration (ng/mL) ± %RPD		85.1 ng/mL ± 7.9%		19.4 ng/mL ± 8.2%		12.1 ng/mL ± 1.7%	

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:10 and analyzed by external standard calibration.

Table 20. CGMN GW PW09 140115

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-031	CGMN-PW09-0-140115	5.00	NA	1.40	NA
ISO11-01-03-13-032	CGMN-PW09-DB-140115	4.52	NA	1.26	NA
ISO11-01-03-13-033	CGMN-PW09-FMS-140115	7.30	NC	2.90	81.9
Average Concentration (ng/mL) ± %RPD		4.76 ng/mL ± 10%		1.33 ng/mL ± 11%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-031	CGMN-PW09-0-140115	0.167	NA	0.117	NA	2.29	NA
ISO11-01-03-13-032	CGMN-PW09-DB-140115	0.178	NA	0.101	NA	1.97	NA
ISO11-01-03-13-033	CGMN-PW09-FMS-140115	2.23	103	2.27	108	4.08	105
Average Concentration (ng/mL) ± %RPD		0.173 ng/mL ± 6.4%		0.109 ng/mL ± 15%		2.13 ng/mL ± 15%	

3M LIMS ID	Description	¹² C ₂ -PFBA	¹² C ₄ -PFOA	¹² C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-13-031	CGMN-PW09-0-140115	84.0	103	98.5
ISO11-01-03-13-032	CGMN-PW09-DB-140115	90.3	99.5	90.4
ISO11-01-03-13-033	CGMN-PW09-FMS-140115	101	86.6	110
Average Recovery (%) ± %RSD		91.6% ± 9.1%	99.7% ± 3.2%	99.6% ± 9.9%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 with methanol and analyzed by internal standard calibration.

Table 21. CGMN GW PW10 140115

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-034	CGMN-PW10-0-140122	4.03	NA	0.223	NA
ISO11-01-03-13-035	CGMN-PW10-DB-140122	4.77	NA	0.200	NA
ISO11-01-03-13-036	CGMN-PW10-FMS-140122	6.79	NC	2.23	105
Average Concentration (ng/mL) ± %RPD		4.40 ng/mL ± 17%		0.212 ng/mL ± 11%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-034	CGMN-PW10-0-140122	0.254	NA	0.0426	NA	0.127	NA
ISO11-01-03-13-035	CGMN-PW10-DB-140122	0.268	NA	0.0543	NA	0.126	NA
ISO11-01-03-13-036	CGMN-PW10-FMS-140122	2.53	113	1.89	92.1	1.99	101
Average Concentration (ng/mL) ± %RPD		0.261 ng/mL ± 5.4%		0.0485 ng/mL ± 24%⁽¹⁾		0.124 ng/mL ± 4.0%	

3M LIMS ID	Description	¹³ C ₁ -PFBA	¹³ C ₁ -PFOA	¹³ C ₁ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-13-034	CGMN-PW10-0-140122	99.6	116	83.4
ISO11-01-03-13-035	CGMN-PW10-DB-140122	102	89.1	73.7
ISO11-01-03-13-036	CGMN-PW10-FMS-140122	99.6	95.0	103
Average Recovery (%) ± %RSD		100% ± 1.1%	100% ± 14%	86.6% ± 17%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 with methanol and analyzed by internal standard calibration.
 (1) Sample/sample duplicate %RPD did not meet method acceptance criteria of <20%.

Table 22. CGMN GW MW14R 140113

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-037	CGMN-MW14R-0-140113	300	NA	137	NA
ISO11-01-03-13-038	CGMN-MW14R-DB-140113	289	NA	141	NA
ISO11-01-03-13-039	CGMN-MW14R-FMS-140113	361	NC	227	88.7
ISO11-01-03-13-037	CGMN-MW14R-0-140113 LMS	551	86.3	NA	NA
Average Concentration (ng/mL) ± %RPD		295 ng/mL ± 3.7%		139 ng/mL ± 2.9%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-037	CGMN-MW14R-0-140113	17.3	NA	12.1	NA	25.0	NA
ISO11-01-03-13-038	CGMN-MW14R-DB-140113	17.7	NA	12.4	NA	24.8	NA
ISO11-01-03-13-039	CGMN-MW14R-FMS-140113	117	99.5	114	102	120	95.3
Average Concentration (ng/mL) ± %RPD		17.5 ng/mL ± 2.3%		12.3 ng/mL ± 2.4%		24.9 ng/mL ± 0.80%	

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:10 and analyzed by external standard calibration.

Table 23. CGMN GW MW104-RB 140115 and TRIP BLANKS

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-043	CGMN-GW-MW104-RB- 40115	<0.200	NA	0.227	NA
ISO11-01-03-13-040	CGMN-TRIP-0-140113	<0.200	NA	<0.0480	NA
ISO11-01-03-13-041	CGMN-TRIP-LS-140113	2.23	112	1.90	99.2
ISO11-01-03-13-042	CGMN-TRIP-HS-140113	99.7	100	95.0	95.8

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-13-043	CGMN-GW-MW104-RB- 40115	<0.100	NA	<0.0250	NA	0.235	NA
ISO11-01-03-13-040	CGMN-TRIP-0-140113	<0.100	NA	<0.0250	NA	<0.0232	NA
ISO11-01-03-13-041	CGMN-TRIP-LS-140113	1.72	86.0	2.50	125	1.75	94.4
ISO11-01-03-13-042	CGMN-TRIP-HS-140113	106	106	102	102	102	102

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₄ -PFOA	¹³ C ₂ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-13-043	CGMN-GW-MW104-RB- 40115	99.0	93.3	102
ISO11-01-03-13-040	CGMN-TRIP-0-140113	86.2	98.0	97.6
ISO11-01-03-13-041	CGMN-TRIP-LS-140113	97.5	96.4	107

NA = Not Applicable
 Samples were diluted 1:1 and analyzed by internal standard calibration with the exception of the TRIP HS, which was diluted 1:10 and analyzed by external standard calibration.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 11-23 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Attachments

Appendix A: Target Analyte Historical Trend Data for Cottage Grove Monitoring Wells MW07, MW12, MW13, MW16, MW101, MW104, MW105, MW108, MW110, PW09, and PW10.

8 Signatures



Digitally signed by Susan T. Wolf
DN: c=US, st=MN, l=St. Paul, ou=3M
Environmental Laboratory - authenticated
by LRA, email=stwolf@mmm.com,
o=3M, cn=Susan T. Wolf
Reason: I am the author of this document
Date: 2014.02.18 11:50:28 -06'00'

Susan T. Wolf, 3M Principal Analytical Investigator



Digitally signed by William K. Reagen
DN: c=US, st=MN, l=St. Paul,
ou=Laboratory Director, ou=3M
Environmental Laboratory -
authenticated by LRA,
email=wkreagen@mmm.com, o=3M,
cn=William K. Reagen
Reason: I am approving this document
Date: 2014.02.18 13:16:54 -06'00'

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.



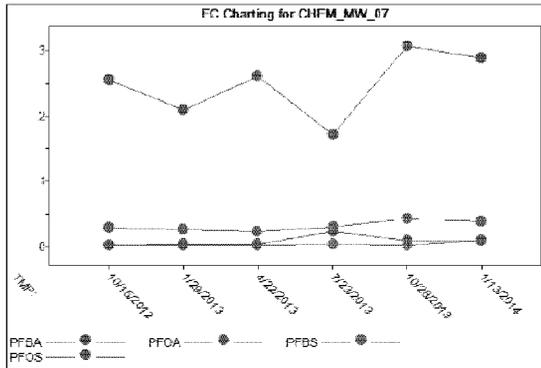
Digitally signed by Casey Howell
DN: c=US, st=MN, l=St. Paul, ou=Quality
Assurance Unit, ou=3M Environmental
Laboratory - authenticated by LRA,
email=chowall@mmm.com, o=3M,
cn=Casey Howell
Reason: I agree to the terms defined by the
placement of my signature on this document
Date: 2014.02.19 09:46:41 -05'00'

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M Environmental Laboratory.

Appendix A: Target Analyte Historical Trend Data

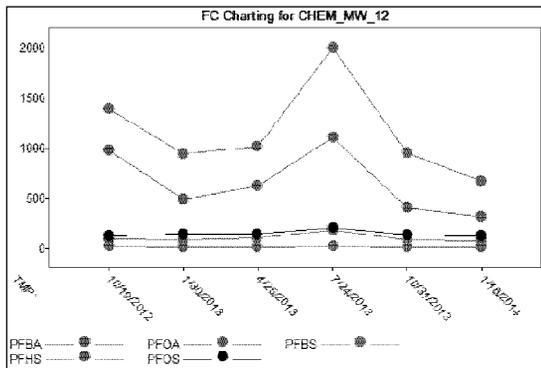
Cottage Grove MW07 – units are ng/mL



MW07	10/15/2012	1/29/2013	4/22/2013	7/23/2013	10/28/2013	1/13/2014
PFBA	2.55	2.09	2.61	1.71	3.07	2.88
PFOA	0.298	0.259	0.240	0.303	0.428	0.390
PFBS	<0.0250	<0.0250	0.0268	0.0396	0.0291	<0.100
PFOS	0.0257	0.0439	0.0459	0.241	0.104	0.104

Samples were below the limit of quantitation for PFHS.

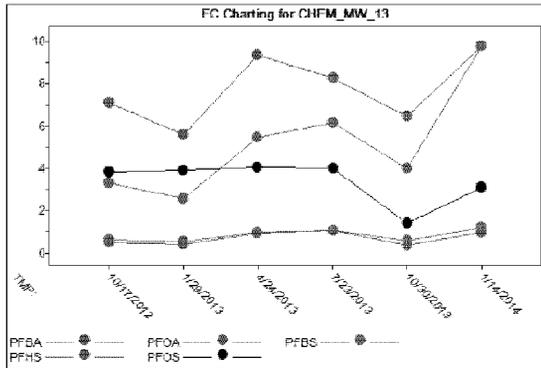
Cottage Grove MW12 – units are ng/mL



MW12	10/19/2012	1/30/2013	4/25/2013	7/24/2013	10/31/2013	1/16/2014
PFBA	975	484	622	1110	408	312
PFOA	1390	946	1020	2000	954	668
PFBS	94.3	91.4	105	175	87.7	82.1
PFHS	20.9	13.7	16.4	23.9	13.0	13.1
PFOS	122	143	145	204	131	121

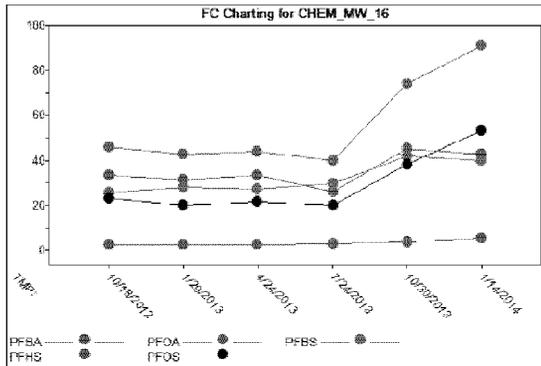
Appendix A: Target Analyte Historical Trend Data

Cottage Grove MW13 – units are ng/mL



MW13	10/17/2012	1/29/2013	4/24/2013	7/23/2013	10/30/2013	1/14/2014
PFBA	7.10	5.60	9.34	8.30	6.48	9.80
PFOA	3.33	2.56	5.46	6.17	4.01	9.78
PFBS	0.619	0.560	0.972	1.07	0.571	1.20
PFHS	0.487	0.396	0.931	1.05	0.366	0.969
PFOS	3.85	3.92	4.05	4.02	1.40	3.08

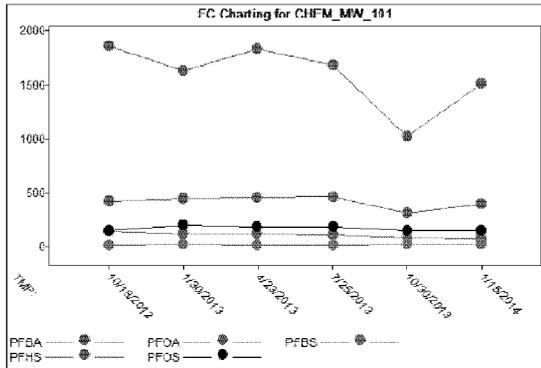
Cottage Grove MW16 – units are ng/mL



MW16	10/18/2012	1/29/2013	4/24/2013	7/24/2013	10/30/2013	1/14/2014
PFBA	33.6	31.5	33.2	26.3	45.0	42.9
PFOA	45.9	42.6	43.8	39.9	73.9	91.0
PFBS	25.7	28.3	27.5	29.6	42.1	40.0
PFHS	2.71	2.54	2.74	2.83	3.95	5.52
PFOS	23.1	20.2	21.8	20.1	38.2	53.1

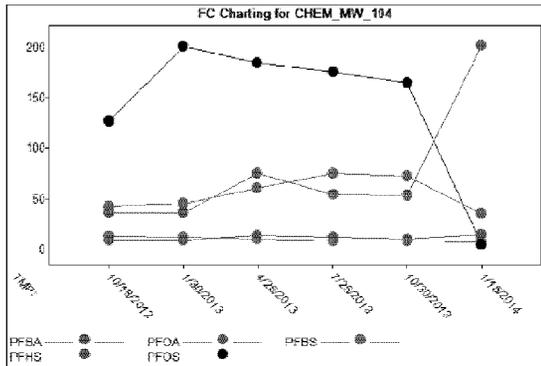
Appendix A: Target Analyte Historical Trend Data

Cottage Grove MW101 – units are ng/mL



MW101	10/18/2012	1/30/2013	4/23/2013	7/25/2013	10/30/2013	1/15/2014
PFBA	1860	1630	1830	1680	1020	1510
PFOA	147	124	121	112	86.7	79.4
PFBS	23.9	25.2	19.5	24.0	25.5	25.4
PFHS	427	455	459	464	314	398
PFOS	154	206	188	189	158	159

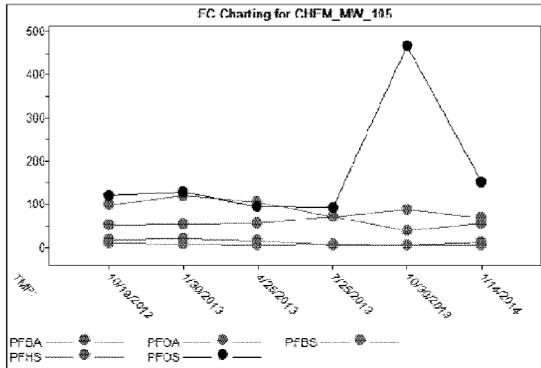
Cottage Grove MW104 – units are ng/mL



MW104	10/18/2012	1/30/2013	4/25/2013	7/25/2013	10/30/2013	1/15/2014
PFBA	36.0	36.3	74.7	54.3	53.6	202
PFOA	42.0	44.8	60.7	74.7	72.5	35.1
PFBS	12.6	11.4	9.75	8.16	7.95	7.50
PFHS	8.69	9.07	13.4	11.8	10.1	14.2
PFOS	127	201	185	176	165	4.25

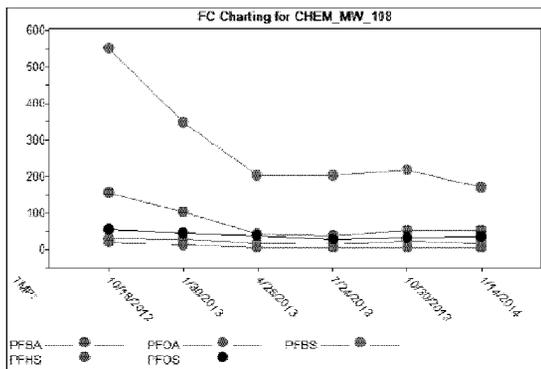
Appendix A: Target Analyte Historical Trend Data

Cottage Grove MW105 – units are ng/mL



MW105	10/19/2012	1/30/2013	4/25/2013	7/25/2013	10/30/2013	1/14/2014
PFBA	52.7	54.4	57.0	71.6	38.9	55.6
PFOA	98.3	119	104	70.6	87.2	68.3
PFBS	9.46	8.05	5.72	6.49	4.98	6.15
PFHS	18.9	21.1	16.0	6.35	6.01	12.6
PFOS	120	129	95.0	92.3	467	152

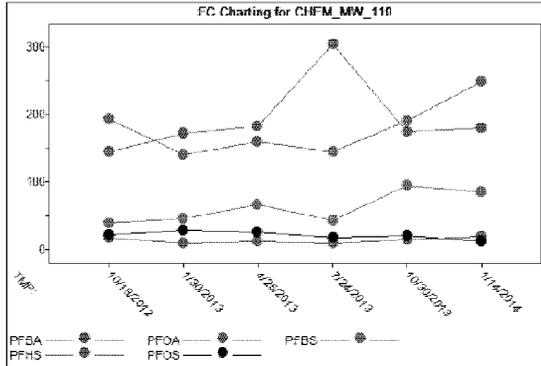
Cottage Grove MW108 – units are ng/mL



MW108	10/18/2012	1/30/2013	4/25/2013	7/24/2013	10/30/2013	1/14/2014
PFBA	155	101	42.5	38.0	51.7	51.6
PFOA	550	348	201	202	218	169
PFBS	30.5	26.5	16.1	14.6	21.6	17.0
PFHS	20.7	11.1	4.95	4.94	4.47	4.13
PFOS	55.2	45.5	37.3	26.8	33.2	33.5

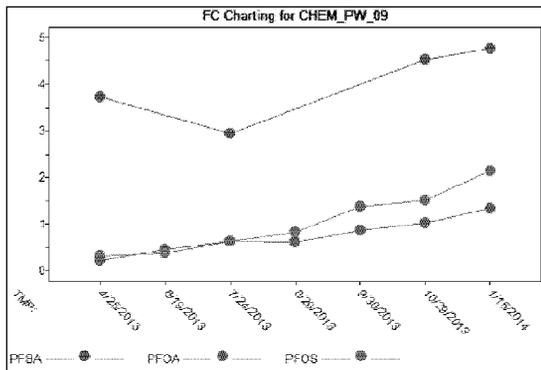
Appendix A: Target Analyte Historical Trend Data

Cottage Grove MW110 – units are ng/mL



MW110	10/18/2012	1/30/2013	4/25/2013	7/24/2013	10/30/2013	1/14/2014
PFBA	144	171	183	304	174	179
PFOA	193	141	160	144	190	248
PFBS	39.6	46.6	66.7	43.8	94.8	85.1
PFHS	17.6	10.3	11.8	9.41	15.3	19.4
PFOS	21.6	28.6	26.4	17.8	21.1	12.1

Cottage Grove PW09 – units are ng/mL

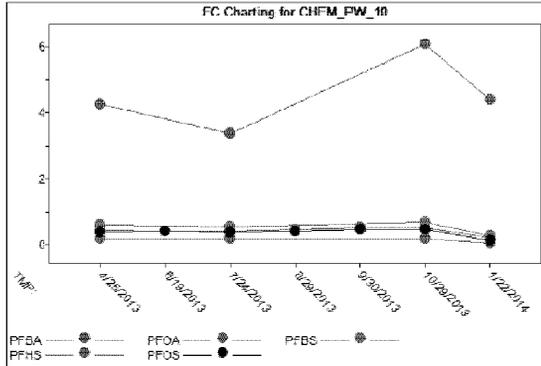


PW09	4/25/2013	6/19/2013	7/24/2013	8/29/2013	9/30/2013	10/29/2013	1/15/2014
PFBA	3.72	NA	2.95	NA	NA	4.53	4.76
PFOA	0.223	0.462	0.624	0.605	0.873	1.02	1.33
PFOS	0.324	0.376	0.622	0.818	1.38	1.52	2.13

NA = Not Applicable; analyte not requested for the sampling event.

Appendix A: Target Analyte Historical Trend Data

Cottage Grove PW10 – units are ng/mL



PW10	4/25/2013	6/19/2013	7/24/2013	8/29/2013	9/30/2013	10/29/2013	1/22/2014
PFBA	4.25	NA	3.37	NA	NA	6.09	4.40
PFOA	0.428	0.415	0.420	0.471	0.528	0.524	0.212
PFBS	0.594	NA	0.540	NA	NA	0.703	0.261
PFHS	0.197	NA	0.185	NA	NA	0.193	0.0485
PFOS	0.396	0.401	0.38	0.415	0.465	0.458	0.124

NA = Not Applicable; analyte not requested for the sampling event.

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-13

Requester: Hohenstein, Gary Allan (MAPLEWOOD)
Department: 452090 Site Source: 01J9C020
Project Number: 0069837001
Date Created: 1/10/2014
Project Description: 3M Cottage Grove Site; 1st quarter 2014 sampling
Copy List: Gaetz, Mark Anthony (MAPLEWOOD-3MUS-MN 3M CENTER)

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Comments:

3M Sample Number Sample Description Date/Time Sampled Matrix Comment

ISO11-01-03-13-001	CGMN-MW07-0- 140113	1-13-2014/14:40	GW	
ISO11-01-03-13-002	CGMN-MW07-DB- 140113	↓		
ISO11-01-03-13-003	CGMN-MW07-FMS- 140113			
ISO11-01-03-13-004	CGMN-MW12-0- 140116		1-16-2014 12:30	
ISO11-01-03-13-005	CGMN-MW12-DB- 140116	1-16-2014 12:30		
ISO11-01-03-13-006	CGMN-MW12-FMS- 140116	1-16-2014 12:30		
ISO11-01-03-13-007	CGMN-MW13-0- 140114	1-14-2014/09:00		
ISO11-01-03-13-008	CGMN-MW13-DB- 140114	1-14-2014/09:00		
ISO11-01-03-13-009	CGMN-MW13-FMS- 140114	1-14-2014/09:00		
ISO11-01-03-13-010	CGMN-MW16-0- 140114	1-14-2014/11:15		
ISO11-01-03-13-011	CGMN-MW16-DB- 140114	1-14-2014/11:15		
ISO11-01-03-13-012	CGMN-MW16-FMS- 140114	1-14-2014/11:15		
ISO11-01-03-13-013	CGMN-MW101-0- 140115	1-15-2014/10:50	✓	
ISO11-01-03-13-014	CGMN-MW101-DB- 140115	1-15-2014/10:50	GW	

Sample Condition Upon Receipt Acceptable All items accounted for

Temperature: _____ Deg C Received on Ice Other:

Collected by (print): JOHN HUNTER

Collector's signature: *[Signature]*

Relinquished by: _____ Date _____ Time _____ Shipped Via _____ Received by: _____ Date _____ Time _____

<i>[Signature]</i>	WESTON	1/17/14					

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-13 (cont.)

Requester: Hohenstein, Gary Allan (MAPLEWOOD)
Department: 452090 Site Source: 0119C020
Project Number: 0069837001
Date Created: 1/10/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Project Description: 3M Cottage Grove Site; 1st quarter 2014 sampling

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-13-015	CGMN-MW101-FMS- 140115	1-15-2014/10:50	GW	
ISO11-01-03-13-016	CGMN-MW104-0- 140115	1-15-2014/09:45		
ISO11-01-03-13-017	CGMN-MW104-DB- 140115	1-15-2014/09:45		
ISO11-01-03-13-018	CGMN-MW104-FMS- 140115	1-15-2014/09:45		
ISO11-01-03-13-019	CGMN-MW105-0- 140114	1-14-2014/13:50		
ISO11-01-03-13-020	CGMN-MW105-DB- 140114	1-14-2014/13:50		
ISO11-01-03-13-021	CGMN-MW105-FMS- 140114	1-14-2014/13:50		
ISO11-01-03-13-022	CGMN-MW108-0- 140114	1-14-2014/15:10		
ISO11-01-03-13-023	CGMN-MW108-DB- 140114	1-14-2014/15:10		
ISO11-01-03-13-024	CGMN-MW108-FMS- 140114	1-14-2014/15:10		
ISO11-01-03-13-025	CGMN-MW110-0- 140114	1-14-2014/12:20		
ISO11-01-03-13-026	CGMN-MW110-DB- 140114	1-14-2014/12:20	✓	
ISO11-01-03-13-027	CGMN-MW110-FMS- 140114	1-14-2014/12:20	GW	
ISO11-01-03-13-028	CGMN-PW06-0-			
ISO11-01-03-13-029	CGMN-PW06-DB-	NOT SAMPLED		
ISO11-01-03-13-030	CGMN-PW06-FMS-			
ISO11-01-03-13-031	CGMN-PW09-0- 140115	1-15-2014/07:40	GW	

Sample Condition Upon Receipt Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): JOHN HUNTER Collector's signature: *John Hunter*

Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
<i>John Hunter</i>	1/17/14					

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-13 (cont.)

Requester: Hohenstein, Gary Allan (MAPLEWOOD)
Department: 452090 Site Source: 01J9C020
Project Number: 0069837001
Date Created: 1/10/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Project Description: 3M Cottage Grove Site; 1st quarter 2014 sampling

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-13-032	CGMN-PW09-DB- 140115	1-15-2014/07:40	GW	SAMPLES DELIVERED TO LAB ON 1/17/14
ISO11-01-03-13-033	CGMN-PW09-FMS- 140115	1-15-2014/07:40	GW	Sample delivered
ISO11-01-03-13-034	CGMN-PW10-0- 140122	1-22-2014/1500	GW	to lab on 1/23/14
ISO11-01-03-13-035	CGMN-PW10-DB- 140122	1-22-2014/1500	GW	
ISO11-01-03-13-036	CGMN-PW10-FMS- 140122	1-22-2014/1500	GW	
ISO11-01-03-13-037	CGMN-MW14R-0- 140113	1-13-14/15:45	GW	SAMPLES DELIVERED TO LAB ON 1/17/14
ISO11-01-03-13-038	CGMN-MW14R-DB- 140113	1-13-14/15:45		
ISO11-01-03-13-039	CGMN-MW14R-FMS- 140113	1-13-14/15:45		
ISO11-01-03-13-040	CGMN-TRIP-0- 140113	1-13-14/12:00		
ISO11-01-03-13-041	CGMN-TRIP-LS- 140113	1-13-14/12:00		
ISO11-01-03-13-042	CGMN-TRIP-HS- 140113	1-13-14/12:00	✓	
ISO11-01-03-13-043	CGMN CGMN CGMN-GW-MW14-RB- 140115	1-15-14/07:45	GW	

Sample Condition Upon Receipt Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): JOAN HUNTER Collector's signature: *Gal*
Relinquished by: _____ Date _____ Time _____ Shipped Via _____ Received by: _____ Date _____ Time _____



APRIL 2014

3M_MN01596074

3630.0125

Final Report

Fluorochemical Characterization of Aqueous Samples, Cottage Grove Groundwater Sampling – 2nd Quarter 2014

Laboratory Request Number: ISO11-01-03-14

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Jim Kotsmith
3M EHS Operations
3M Building 224-5W-17
Saint Paul, MN 55144-1000
Phone: (651) 737-3635



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Testing Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.

3M Principal Analytical Investigator: Susan Wolf

Report Author: Chelsie Grochow

Analytical Report ISO11-01-03-14

Fluorochemical Characterization of Aqueous Samples, Cottage Grove Groundwater
Sampling – 2nd Quarter 2014

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater samples collected by Weston Solutions personnel at the 3M Cottage Grove facility. Samples were collected on April 23-25, 2014. Samples were returned to the 3M Environmental Laboratory on April 28, 2014 on ice for the analysis of twelve fluorochemical compounds under laboratory project number ISO11-01-03-14.

The 3M Environmental Laboratory prepared sample containers for fourteen sampling locations. Each sample set consisted of a field sample, field sample duplicate, and field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing all analytes prior to being sent to the field for sample collection. All sample bottles were fortified with internal standards and surrogate recovery standards prior to being sent to the field for sample collection.

Samples were prepared and analyzed for PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnA, PFDoA, PFBS, PFHS, PFOS and the surrogate recovery standards (SRSs) ¹³C₃-PFBA, ¹³C₄-PFOA, ¹³C₂-PFUnA, and ¹³C₄-PFOS, using method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Testing Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFPeA Concentration (ng/mL)	PFHxA Concentration (ng/mL)	PFHpA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFNA Concentration (ng/mL)	PFDA Concentration (ng/mL)
ISO11-01-03-14-001	CGMN-GW-MW07-0-140423	2.48	0.159	0.0806	<0.0250	0.351	<0.0250	<0.0250
ISO11-01-03-14-002	CGMN-GW-MW07-DB-140423	2.48	0.147	0.0749	<0.0250	0.336	<0.0250	<0.0250
Average		2.48	0.153	0.0778	<0.0250	0.344	<0.0250	<0.0250
%RPD Sample/Sample Dup		0.0	7.8	7.3	NA	4.4	NA	NA
ISO11-01-03-14-004	CGMN-GW-MW12-0-140425	218	24.8	26.4	11.7	545	1.77	1.82
ISO11-01-03-14-005	CGMN-GW-MW12-DB-140425	222	24.8	27.1	11.7	550	1.75	1.87
Average		220 ⁽²⁾	24.8	26.8	11.7	548 ⁽²⁾	1.76	1.85
%RPD Sample/Sample Dup		1.8	0.0	2.6	0.0	0.91	1.1	2.7
ISO11-01-03-14-008	CGMN-GW-MW13-0-140424	7.45	0.477	0.265	0.0883	8.87	0.0503	<0.0250
ISO11-01-03-14-009	CGMN-GW-MW13-DB-140424	6.83	0.452	0.249	0.0674	8.39	0.0387	<0.0250
Average		7.14	0.465	0.257	0.0779	8.63	0.0445	<0.0250
%RPD Sample/Sample Dup		8.7	5.4	6.2	27 ⁽³⁾	5.6	26 ⁽³⁾	NA
ISO11-01-03-14-011	CGMN-GW-MW14R-0-140423	645	280	195	25.4	542	1.70	2.11
ISO11-01-03-14-012	CGMN-GW-MW14R-DB-140423	654	275	199	26.7	543	1.74	2.08
Average		650 ⁽²⁾	278 ⁽²⁾	197	26.1	543 ⁽²⁾	1.72	2.10
%RPD Sample/Sample Dup		1.4	1.8	2.0	5.0	0.18	2.3	1.4
ISO11-01-03-14-015	CGMN-GW-MW16-0-140424	41.1	6.91	6.57	3.44	89.1	0.546	0.329
ISO11-01-03-14-016	CGMN-GW-MW16-DB-140424	40.3	6.52	6.42	3.15	82.3	0.523	0.339
Average		40.7	6.72	6.50	3.30	85.7	0.535	0.334
%RPD Sample/Sample Dup		2.0	5.8	2.3	8.8	7.9	4.3	3.0

NA = Not Applicable

- (1) Samples were analyzed using internal standard calibration unless noted otherwise. The analytical method uncertainties associated with the reported results using internal calibration are as follows: PFBA ± 18%, PFPeA ± 18%, PFHxA ± 16%, PFHpA ± 18%, PFOA ± 12%, PFNA ± 19%, PFDA ± 19%, PFUnA ± 19%, PFDoA ± 18%, PFBS ± 10%, PFHS ± 9.3%, PFOS ± 16%.
- (2) Sample set was analyzed using external standard calibration. The analytical method uncertainties associated with the reported samples using external calibration are as follows: PFBA ± 21%, PFPeA ± 17%, PFOA ± 14%, PFBS ± 18%, PFHS ± 11%, and PFOS ± 9.9%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) NR = Not reportable; sample area counts below the LOQ area counts and did not meet method blank criteria.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFPeA Concentration (ng/mL)	PFHxA Concentration (ng/mL)	PFHpA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFNA Concentration (ng/mL)	PFDA Concentration (ng/mL)
ISO11-01-03-14-019	CGMN-GW-MW101-0-140425	1330	71.8	60.9	63.2	76.1	7.08	0.248
ISO11-01-03-14-020	CGMN-GW-MW101-DB-140425	1330	72.6	61.3	61.4	75.8	6.84	0.233
Average		1330 ⁽²⁾	72.2	61.1	62.3	76.0 ⁽²⁾	6.96	0.241
%RPD Sample/Sample Dup		0.0	1.1	0.65	2.9	0.39	3.4	6.2
ISO11-01-03-14-050	CGMN-GW-MW102-0-140425	924	30.7	29.1	6.1	58.6	1.84	0.143
ISO11-01-03-14-051	CGMN-GW-MW102-DB-140425	914	30.6	28.5	5.8	57.6	1.90	0.129
Average		919 ⁽²⁾	30.7	28.8	16.0	58.1	1.87	0.136
%RPD Sample/Sample Dup		1.1	0.33	2.1	1.9	1.7	3.2	10
ISO11-01-03-14-054	CGMN-GW-MW103-0-140425	238	27.3	57.0	31.4	117	0.0487	<0.0250
ISO11-01-03-14-055	CGMN-GW-MW103-DB-140425	243	29.1	57.8	30.4	125	0.0566	<0.0250
Average		241 ⁽²⁾	28.2	57.4	30.9	121 ⁽²⁾	0.0527	<0.0250
%RPD Sample/Sample Dup		2.1	6.4	1.4	3.2	6.6	15	NA
ISO11-01-03-14-023	CGMN-GW-MW104-0-140425	107	12.7	19.9	9.08	82.0	2.57	3.20
ISO11-01-03-14-024	CGMN-GW-MW104-DB-140425	107	12.4	19.5	9.13	82.1	2.60	3.32
Average		107	12.6	19.7	9.11	82.1	2.59	3.26
%RPD Sample/Sample Dup		0.0	2.4	2.0	0.55	0.12	1.2	3.7
ISO11-01-03-14-027	CGMN-GW-MW105-0-140425	70.3	5.86	11.7	8.38	42.7	0.577	0.328
ISO11-01-03-14-028	CGMN-GW-MW105-DB-140425	71.7	6.24	11.8	8.89	45.9	0.588	0.339
Average		71.0	6.05	11.8	8.64	44.3	0.583	0.334
%RPD Sample/Sample Dup		2.0	6.3	0.85	5.9	7.2	1.9	3.3

NA = Not Applicable

- (1) Samples were analyzed using internal standard calibration unless noted otherwise. The analytical method uncertainties associated with the reported results using internal calibration are as follows: PFBA ± 18%, PFPeA ± 18%, PFHxA ± 16%, PFHpA ± 18%, PFOA ± 12%, PFNA ± 19%, PFDA ± 19%, PFUnA ± 19%, PFDoA ± 18%, PFBS ± 10%, PFHS ± 9.3%, PFOS ± 16%.
- (2) Sample set was analyzed using external standard calibration. The analytical method uncertainties associated with the reported samples using external calibration are as follows: PFBA ± 21%, PFPeA ± 17%, PFOA ± 14%, PFBS ± 18%, PFHS ± 11%, and PFOS ± 9.9%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) NR = Not reportable; sample area counts below the LOQ area counts and did not meet method blank criteria.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFPeA Concentration (ng/mL)	PFHxA Concentration (ng/mL)	PFHpA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFNA Concentration (ng/mL)	PFDA Concentration (ng/mL)
ISO11-01-03-14-031	CGMN-GW-MW108-0-140425	59.1	10.6	9.39	3.84	166	1.15	0.516
ISO11-01-03-14-032	CGMN-GW-MW108-DB-140425	71.6	12.3	11.0	4.45	162	1.36	0.607
Average		65.4	11.5	10.2	4.15	164 ⁽²⁾	1.26	0.562
%RPD Sample/Sample Dup		19	15	16	15	2.4	17	16
ISO11-01-03-14-035	CGMN-GW-MW110-0-140424	186	24.6	34.0	2.5	310	0.191	<0.0250
ISO11-01-03-14-036	CGMN-GW-MW110-DB-140424	191	23.4	31.7	2.8	310	0.197	<0.0250
Average		189 ⁽²⁾	24.0	32.9	12.7	310 ⁽²⁾	0.194	<0.0250
%RPD Sample/Sample Dup		2.6	5.0	7.0	2.4	0.0	3.1	NA
ISO11-01-03-14-039	CGMN-GW-PW09-0-140425	4.18	0.268	0.127	0.0443	1.03	0.0353	<0.0250
ISO11-01-03-14-040	CGMN-GW-PW09-DB-140425	4.10	0.275	0.143	0.0498	1.07	0.0377	<0.0250
Average		4.14	0.272	0.135	0.0471	1.05	0.0365	<0.0250
%RPD Sample/Sample Dup		1.9	2.6	12	12	3.8	6.6	NA
ISO11-01-03-14-042	CGMN-GW-PW10-0-140425	4.99	0.250	0.134	0.0420	0.421	<0.0250	<0.0250
ISO11-01-03-14-043	CGMN-GW-PW10-DB-140425	5.15	0.254	0.167	0.0517	0.420	<0.0250	<0.0250
Average		5.07	0.252	0.151	0.0469	0.421	<0.0250	<0.0250
%RPD Sample/Sample Dup		3.2	1.6	22 ⁽³⁾	21 ⁽³⁾	0.24	NA	NA
ISO11-01-03-14-045	CGMN-GW-MW105-RB-140425	<0.0500	<0.0250	<0.0250	<0.0250	<0.0480	<0.0250	<0.0250
ISO11-01-03-14-046	CGMN-GW-TRIP-0-140423	<0.0500	<0.0250	<0.0250	<0.0250	<0.0480	<0.0250	<0.0250

NA = Not Applicable

- (1) Samples were analyzed using internal standard calibration unless noted otherwise. The analytical method uncertainties associated with the reported results using internal calibration are as follows: PFBA ± 18%, PFPeA ± 18%, PFHxA ± 16%, PFHpA ± 19%, PFOA ± 12%, PFNA ± 19%, PFDA ± 19%, PFUnA ± 19%, PFDoA ± 18%, PFBS ± 10%, PFHS ± 9.3%, PFOS ± 16%.
- (2) Sample set was analyzed using external standard calibration. The analytical method uncertainties associated with the reported samples using external calibration are as follows: PFBA ± 21%, PFPeA ± 17%, PFOA ± 14%, PFBS ± 18%, PFHS ± 11%, and PFOS ± 9.9%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) NR = Not reportable; sample area counts below the LOQ area counts and did not meet method blank criteria.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	PFUnA Concentration (ng/mL)	PFDoA Concentration (ng/mL)	PFBS Concentration (ng/mL)	PFHS Concentration (ng/mL)	PFOS Concentration (ng/mL)
ISO11-01-03-14-001	CGMN-GW-MW07-0-140423	<0.0250	NR ⁽⁴⁾	0.0357	<0.0250	0.0495
ISO11-01-03-14-002	CGMN-GW-MW07-DB-140423	<0.0250	<0.0250	0.0363	<0.0250	0.0491
Average		<0.0250	<0.0250	0.0360	<0.0250	0.0493
%RPD Sample/Sample Dup		NA	NA	1.7	NA	0.81
ISO11-01-03-14-004	CGMN-GW-MW12-0-140425	0.433	0.154	67.5	9.97	115
ISO11-01-03-14-005	CGMN-GW-MW12-DB-140425	0.415	0.169	70.3	9.67	118
Average		0.424	0.162	68.9 ⁽²⁾	9.82	117 ⁽²⁾
%RPD Sample/Sample Dup		4.2	9.3	4.1	3.1	2.6
ISO11-01-03-14-008	CGMN-GW-MW13-0-140424	<0.0250	<0.0250	0.791	0.594	2.46
ISO11-01-03-14-009	CGMN-GW-MW13-DB-140424	<0.0250	<0.0250	0.712	0.517	2.20
Average		<0.0250	<0.0250	0.752	0.556	2.33
%RPD Sample/Sample Dup		NA	NA	11	14	11
ISO11-01-03-14-011	CGMN-GW-MW14R-0-140423	<0.0250	<0.0250	18.2	19.7	348
ISO11-01-03-14-012	CGMN-GW-MW14R-DB-140423	<0.0250	<0.0250	18.2	20.3	358
Average		<0.0250	<0.0250	18.2	20.0	353 ⁽²⁾
%RPD Sample/Sample Dup		NA	NA	0.0	3.0	2.8
ISO11-01-03-14-015	CGMN-GW-MW16-0-140424	<0.0250	<0.0250	37.7	4.43	51.4
ISO11-01-03-14-016	CGMN-GW-MW16-DB-140424	<0.0250	<0.0250	38.1	4.55	52.4
Average		<0.0250	<0.0250	37.9 ⁽²⁾	4.49	51.9
%RPD Sample/Sample Dup		NA	NA	1.1	2.7	1.9

NA = Not Applicable

- (1) Samples were analyzed using internal standard calibration unless noted otherwise. The analytical method uncertainties associated with the reported results using internal calibration are as follows: PFBA ± 18%, PFPeA ± 18%, PFHxA ± 16%, PFHpA ± 18%, PFOA ± 12%, PFNA ± 19%, PFDA ± 19%, PFUnA ± 19%, PFDoA ± 18%, PFBS ± 10%, PFHS ± 9.3%, PFOS ± 16%.
- (2) Sample set was analyzed using external standard calibration. The analytical method uncertainties associated with the reported samples using external calibration are as follows: PFBA ± 21%, PFPeA ± 17%, PFOA ± 14%, PFBS ± 18%, PFHS ± 11%, and PFOS ± 9.9%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) NR = Not reportable; sample area counts below the LOQ area counts and did not meet method blank criteria.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	PFUnA Concentration (ng/mL)	PFDoA Concentration (ng/mL)	PFBS Concentration (ng/mL)	PFHS Concentration (ng/mL)	PFOS Concentration (ng/mL)
ISO11-01-03-14-019	CGMN-GW-MW101-0-140425	<0.0250	<0.0250	37.9	364	136
ISO11-01-03-14-020	CGMN-GW-MW101-DB-140425	<0.0250	<0.0250	37.6	364	134
Average		<0.0250	<0.0250	37.8 ⁽²⁾	364 ⁽²⁾	135 ⁽²⁾
%RPD Sample/Sample Dup		NA	NA	0.79	0.0	1.5
ISO11-01-03-14-050	CGMN-GW-MW102-0-140425	<0.0250	<0.0250	7.0	146	67.1
ISO11-01-03-14-051	CGMN-GW-MW102-DB-140425	<0.0250	<0.0250	7.4	147	67.6
Average		<0.0250	<0.0250	7.12	147 ⁽²⁾	67.4
%RPD Sample/Sample Dup		NA	NA	0.56	0.68	0.74
ISO11-01-03-14-054	CGMN-GW-MW103-0-140425	<0.0250	<0.0250	6.51	21.0	7.99
ISO11-01-03-14-055	CGMN-GW-MW103-DB-140425	<0.0250	<0.0250	6.51	22.5	7.64
Average		<0.0250	<0.0250	6.51	21.8 ⁽²⁾	7.82
%RPD Sample/Sample Dup		NA	NA	0.0	6.9	4.5
ISO11-01-03-14-023	CGMN-GW-MW104-0-140425	0.0489	<0.0250	7.95	12.8	238
ISO11-01-03-14-024	CGMN-GW-MW104-DB-140425	0.0625	<0.0250	8.25	12.3	229
Average		0.0557	<0.0250	8.10	12.6	234 ⁽²⁾
%RPD Sample/Sample Dup		24 ⁽³⁾	NA	3.7	4.0	3.8
ISO11-01-03-14-027	CGMN-GW-MW105-0-140425	<0.0250	<0.0250	7.56	8.48	56.6
ISO11-01-03-14-028	CGMN-GW-MW105-DB-140425	<0.0250	<0.0250	7.80	8.70	60.7
Average		<0.0250	<0.0250	7.68	8.59	58.7
%RPD Sample/Sample Dup		NA	NA	3.1	2.6	7.0

NA = Not Applicable

- (1) Samples were analyzed using internal standard calibration unless noted otherwise. The analytical method uncertainties associated with the reported results using internal calibration are as follows: PFBA ± 18%, PFPeA ± 18%, PFHxA ± 16%, PFHpA ± 18%, PFOA ± 12%, PFNA ± 19%, PFDA ± 19%, PFUnA ± 19%, PFDoA ± 18%, PFBS ± 10%, PFHS ± 9.3%, PFOS ± 16%.
- (2) Sample set was analyzed using external standard calibration. The analytical method uncertainties associated with the reported samples using external calibration are as follows: PFBA ± 21%, PFPeA ± 17%, PFOA ± 14%, PFBS ± 18%, PFHS ± 11%, and PFOS ± 9.9%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) NR = Not reportable; sample area counts below the LOQ area counts and did not meet method blank criteria.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	PFUnA Concentration (ng/mL)	PFDoA Concentration (ng/mL)	PFBS Concentration (ng/mL)	PFHS Concentration (ng/mL)	PFOS Concentration (ng/mL)
ISO11-01-03-14-031	CGMN-GW-MW108-0-140425	<0.0250	<0.0250	17.8	3.32	40.2
ISO11-01-03-14-032	CGMN-GW-MW108-DB-140425	<0.0250	<0.0250	17.0	3.84	46.4
Average		<0.0250	<0.0250	17.4 ⁽²⁾	3.58	43.3
%RPD Sample/Sample Dup		NA	NA	4.6	15	14
ISO11-01-03-14-035	CGMN-GW-MW110-0-140424	<0.0250	<0.0250	87.1	21.2	12.4
ISO11-01-03-14-036	CGMN-GW-MW110-DB-140424	<0.0250	<0.0250	85.1	20.5	12.6
Average		<0.0250	<0.0250	86.1 ⁽²⁾	20.9	12.5
%RPD Sample/Sample Dup		NA	NA	2.3	3.3	1.6
ISO11-01-03-14-039	CGMN-GW-PW09-0-140425	<0.0250	<0.0250	0.150	0.0823	1.62
ISO11-01-03-14-040	CGMN-GW-PW09-DB-140425	<0.0250	<0.0250	0.145	0.0852	1.58
Average		<0.0250	<0.0250	0.148	0.0838	1.60
%RPD Sample/Sample Dup		NA	NA	3.4	3.5	2.5
ISO11-01-03-14-042	CGMN-GW-PW10-0-140425	<0.0250	<0.0250	0.460	0.105	0.438
ISO11-01-03-14-043	CGMN-GW-PW10-DB-140425	<0.0250	<0.0250	0.481	0.115	0.435
Average		<0.0250	<0.0250	0.471	0.110	0.437
%RPD Sample/Sample Dup		NA	NA	4.5	9.1	0.69
ISO11-01-03-14-045	CGMN-GW-MW105-RB-140425	<0.0250	<0.0250	<0.0250	<0.0250	<0.185
ISO11-01-03-14-046	CGMN-GW-TRIP-0-140423	<0.0250	<0.0250	<0.0250	<0.0250	<0.185

NA = Not Applicable

- (1) Samples were analyzed using internal standard calibration unless noted otherwise. The analytical method uncertainties associated with the reported results using internal calibration are as follows: PFBA ± 18%, PFPeA ± 18%, PFHxA ± 16%, PFHpA ± 19%, PFOA ± 12%, PFNA ± 19%, PFDA ± 19%, PFUnA ± 19%, PFDoA ± 18%, PFBS ± 10%, PFHS ± 9.3%, PFOS ± 16%.
- (2) Sample set was analyzed using external standard calibration. The analytical method uncertainties associated with the reported samples using external calibration are as follows: PFBA ± 21%, PFPeA ± 17%, PFOA ± 14%, PFBS ± 18%, PFHS ± 11%, and PFOS ± 9.9%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) NR = Not reportable; sample area counts below the LOQ area counts and did not meet method blank criteria.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorobutanoic Acid (C4 Acid)	PFBA	Linear
Perfluoropentanoic Acid (C5 Acid)	PFPeA	Linear
Perfluorohexanoic Acid (C6 Acid)	PFHxA	Linear
Perfluoroheptanoic Acid (C7 Acid)	PFHpA	Linear
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorononanoic Acid (C9 Acid)	PFNA	Linear
Perfluorodecanoic Acid (C10 Acid)	PFDA	Linear
Perfluoroundecanoic Acid (C11 Acid)	PFUnA	Linear
Perfluorododecanoic Acid (C12 Acid)	PFDaA	Linear
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched

2.2 Sample Collection

Samples were collected on April 23-25, 2014 in Nalgene™ (high-density polyethylene) bottles prepared at the 3M Environmental Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample bottles were spiked with a mixture of mass-labeled internal standards at a nominal concentration of 1 ng/mL and with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL. Collected sample bottles were returned to the laboratory on ice on April 28, 2014.

2.3 Sample Preparation

All samples were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2).

Samples requiring a 1:100 dilution were prepared by diluting 0.1 mL of sample with 9.9 mLs of methanol. The sampling locations that required dilution were prepared by adding an aliquot of a surrogate standard solution at a nominal concentration of 1 ng/mL.

During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples.

2.4 Analysis

All samples and quality control samples were analyzed for twelve target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.1. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk
Liquid Chromatograph	Agilent 1200 or 1260
Analysis Method	ETS-8-044.1
Analysis Date	5/8/14, 5/12/14, 5/13/14, 5/14/14
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 μ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 μ
Injection Volume	5 or 10 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500
Ion Source	Turbo Spray
Polarity	Negative
Software	Analyst 1.6.1

Table 4. Liquid Chromatography Gradient Program.

Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
ETS-8-044.1 Analysis				
0	0.00	750	90.0	10.0
1	0.50	750	90.0	10.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	90.0	10.0
9	19.0	750	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard ⁽¹⁾	Mass Transition Q1/Q3
PFBA	213/169	[¹³ C ₄]PFBA	217/172
PFPeA	263/219	[¹³ C ₅]PFPeA	268/223
PFHxA	313/119	[¹³ C ₂]PFHxA	315/270
	313/269		
PFHpA	363/319	[¹³ C ₄]PFHpA	367/322
	363/119		
PFOA	413/369	[¹³ C ₆]PFOA	421/376
	413/219		
	413/169		
PFNA	463/419	[¹³ C ₉]PFNA	472/427
	463/219		
	463/169		
PFDA	513/469	[¹³ C ₆]PFDA	519/474
	513/219		
	513/269		
PFUnA	563/519	[¹³ C ₇]PFUnA	570/525
	563/269		
	563/219		
PFDoA	613/569	[¹³ C ₂]PFDoA	615/570
	613/319		
	613/269		
PFBS	299/80	[¹⁸ O ₂]PFBS	303/84
	299/99		
PFHS	399/99	[¹³ C ₃]PFHS	402/80
	399/80		
PFOS	499/99	[¹³ C ₆]PFOS	507/80
	499/80		
	499/130		
[¹³ C ₃]PFBA surrogate	216/172	[¹³ C ₄]PFBA	217/172
[¹³ C ₆]PFOA surrogate	417/372	[¹³ C ₆]PFOA	421/376
[¹³ C ₆]PFOS surrogate	503/80	[¹³ C ₆]PFOS	507/80
[¹³ C ₂]PFUnA surrogate	565/520	[¹³ C ₇]PFUnA	570/525

(1) Internal standard was not used for the external calibration analysis on 5/12/14 and 5/13/14.
 A scheduled MRM was used for the initial analysis on 5/8/14. Otherwise, the dwell time was 20 or 50 msec for each transition.
 The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

Internal calibration analysis:

Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed with the prepared samples. Of these fourteen calibration standards, ten contained the surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). The re-analysis of PFOS on 5/14/14 consisted of nine calibration standards ranging from 0.0125 ng/mL to 5 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

External calibration analysis:

Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. A total of ten spiked standards ranging from 0.02 ng/mL to 25 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL nominal. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of $100 \pm 25\%$ ($100 \pm 30\%$ for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD, were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.1 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the Table 6.

Table 6. LOQ

Analyte	5/8/14 LOQ, ng/mL ⁽¹⁾	5/12/14 LOQ, ng/mL ⁽²⁾	5/13/14 LOQ, ng/mL ⁽²⁾	5/14/14 LOQ, ng/mL ⁽¹⁾
PFBA	0.0500	5.00	NA	NA
PFPeA	0.0250	NA	2.00	NA
PFHxA	0.0250	NA	NA	NA
PFHpA	0.0250	NA	NA	NA
PFOA	0.0480	4.79	NA	NA
PFNA	0.0250	NA	NA	NA
PFDA	0.0250	NA	NA	NA
PFUnA	0.0250	NA	NA	NA
PFDoA	0.0250	NA	NA	NA
PFBS	0.0250	2.00	NA	NA
PFHS	0.0250	2.00	NA	NA
PFOS	0.185	1.85	NA	0.0232

NA = Not Applicable

(1) Dilution factor of 2 was applied to the LOQ.

(2) Dilution factor of 100 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±25%.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: method/solvent blanks, field/trip blanks, and sampling equipment blanks. Each blank result was reviewed and used to evaluate method performance to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate, while only low and high lab control spikes were prepared for the surrogates. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water containing calcium and magnesium to produce the desired concentration. The LCSs were then diluted with methanol or in the same manner as the samples. Method ETS-8-044.1 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCSs meet these criteria and were used to evaluate the analytical method uncertainty in section 3.7 of the report.

The following calculations were used to generate data in Table 7.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%$$

$$\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%$$

Table 7. Lab Control Spike Results.

ETS-8-044.1 Internal calibration Analyzed 5/8/14						
Lab ID	PFBA			PFPeA		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140507-1	0.198	0.210	106	0.198	0.193	97.6
LCS-140507-2	0.198	0.210	106	0.198	0.199	101
LCS-140507-3	0.198	0.215	108	0.198	0.197	99.7
Average ± %RSD	107% ± 1.1%			99.4% ± 1.7%		
LCS-140507-4	19.8	20.9	106	19.8	21.0	106
LCS-140507-5	19.8	20.6	104	19.8	20.7	105
LCS-140507-6	19.8	21.5	108	19.8	21.2	107
Average ± %RSD	106% ± 1.9%			106% ± 0.94%		
LCS-140507-7	79.0	80.5	102	79.0	79.0	100
LCS-140507-8	79.0	81.5	103	79.0	79.0	100
LCS-140507-9	79.0	82.3	104	79.0	79.7	101
Average ± %RSD	103% ± 0.97%			100% ± 0.58%		

ETS-8-044.1 Internal calibration Analyzed 5/8/14						
Lab ID	PFHxA			PFHpA		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140507-1	0.198	0.188	95.2	0.198	0.185	93.3
LCS-140507-2	0.198	0.188	94.7	0.198	0.175	88.6
LCS-140507-3	0.198	0.190	96.1	0.198	0.179	90.3
Average ± %RSD	95.3% ± 0.74%			90.7% ± 2.6%		
LCS-140507-4	19.8	20.7	105	19.8	20.9	106
LCS-140507-5	19.8	20.2	102	19.8	20.8	105
LCS-140507-6	19.8	21.1	106	19.8	21.7	109
Average ± %RSD	104% ± 2.0%			107% ± 1.9%		
LCS-140507-7	79.0	78.6	99.5	79.0	76.5	96.8
LCS-140507-8	79.0	78.0	98.7	79.0	75.0	94.9
LCS-140507-9	79.0	72.9	92.3	79.0	76.1	96.3
Average ± %RSD	96.8% ± 4.1%			96.0% ± 1.0%		

NA = Not Applicable

(1) LCSs spiked above the upper limit of quantification.

Table 7 continued. Lab Control Spike Results.

ETS-8-044.1 Internal calibration Analyzed 5/8/14						
PFOA (Linear + Branched)				PFNA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140507-1	0.190	0.196	103	0.198	0.194	97.9
LCS-140507-2	0.190	0.171	90.0	0.198	0.182	91.8
LCS-140507-3	0.190	0.179	94.3	0.198	0.192	96.9
Average ± %RSD	95.8% ± 6.9%			95.5% ± 3.4%		
LCS-140507-4	19.0	18.6	98.0	19.8	21.5	108
LCS-140507-5	19.0	18.2	96.0	19.8	20.0	101
LCS-140507-6	19.0	19.5	103	19.8	21.7	109
Average ± %RSD	99.0% ± 3.6%			106% ± 4.1%		
LCS-140507-7	75.7	73.1	96.6	79.0	77.2	97.7
LCS-140507-8	75.7	71.3	94.2	79.0	78.1	98.8
LCS-140507-9	75.7	76.7	101	79.0	80.5	102
Average ± %RSD	97.3% ± 3.5%			99.5% ± 2.2%		

ETS-8-044.1 Internal calibration Analyzed 5/8/14						
PFDA				PFUnA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140507-1	0.198	0.175	88.4	0.198	0.183	92.3
LCS-140507-2	0.198	0.198	100	0.198	0.190	95.9
LCS-140507-3	0.198	0.198	100	0.198	0.190	95.8
Average ± %RSD	96.1% ± 7.0%			94.7% ± 2.2%		
LCS-140507-4	19.8	19.8	100	19.8	20.7	105
LCS-140507-5	19.8	19.2	97.1	19.8	20.0	101
LCS-140507-6	19.8	19.6	98.9	19.8	21.3	108
Average ± %RSD	98.7% ± 1.5%			105% ± 3.3%		
LCS-140507-7	79.0	76.8	97.3	79.0	80.2	101
LCS-140507-8	79.0	77.8	98.5	79.0	81.2	103
LCS-140507-9	79.0	79.3	100	79.0	80.7	102
Average ± %RSD	98.6% ± 1.4%			102% ± 0.98%		

NA = Not Applicable

(1) LCSs spiked above the upper limit of quantification.

Table 7 continued. Lab Control Spike Results.

ETS-8-044.1 Internal calibration Analyzed 5/8/14						
Lab ID	PFDoA			PFBS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140507-1	0.198	0.199	101	0.198	0.165	83.5
LCS-140507-2	0.198	0.194	98.2	0.198	0.175	88.3
LCS-140507-3	0.198	0.190	96.1	0.198	0.180	90.7
Average ± %RSD	98.4% ± 2.5%			87.5% ± 4.2%		
LCS-140507-4	19.8	20.7	105	19.8	17.9	90.4
LCS-140507-5	19.8	20.4	103	19.8	17.1	86.5
LCS-140507-6	19.8	21.2	107	19.8	17.9	90.6
Average ± %RSD	105% ± 1.9%			89.2% ± 2.6%		
LCS-140507-7	79.0	82.5	104	79.0	>ULOQ	NA
LCS-140507-8	79.0	80.5	102	79.0	>ULOQ	NA
LCS-140507-9	79.0	81.4	103	79.0	>ULOQ	NA
Average ± %RSD	103% ± 0.97%			NA ⁽¹⁾		

ETS-8-044.1 Internal calibration Analyzed 5/8/14						
Lab ID	PFHS			PFOS (Linear + Branched)		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140507-1	0.198	0.198	99.8	0.184	0.183	99.4
LCS-140507-2	0.198	0.183	92.6	0.184	0.210	114
LCS-140507-3	0.198	0.175	88.2	0.184	0.194	106
Average ± %RSD	93.5% ± 6.3%			106% ± 6.9%		
LCS-140507-4	19.8	19.5	98.6	18.4	19.7	107
LCS-140507-5	19.8	19.6	98.9	18.4	18.8	102
LCS-140507-6	19.8	19.4	97.8	18.4	20.0	109
Average ± %RSD	98.4% ± 0.58%			106% ± 3.4%		
LCS-140507-7	79.0	70.7	89.5	73.2	74.7	102
LCS-140507-8	79.0	71.9	91.0	73.2	76.5	105
LCS-140507-9	79.0	70.6	89.3	73.2	76.6	105
Average ± %RSD	89.9% ± 1.0%			104% ± 1.7%		

NA = Not Applicable

(1) LCSs spiked above the upper limit of quantification.

Table 7 continued. Lab Control Spike Results.

ETS-8-044.1 Internal calibration Analyzed 5/8/14						
Lab ID	¹³ C ₃ -PFBA			¹³ C ₄ -PFOA		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140507-1	0.197	0.192	97.7	0.198	0.208	105
LCS-140507-2	0.197	0.207	105	0.198	0.210	106
LCS-140507-3	0.197	0.197	99.8	0.198	0.183	92.6
Average ± %RSD	101% ± 3.7%			101% ± 7.4%		
LCS-140507-4	1.97	2.02	103	1.98	1.98	100
LCS-140507-5	1.97	1.99	101	1.98	1.91	96.7
LCS-140507-6	1.97	1.98	101	1.98	1.97	99.4
Average ± %RSD	102% ± 1.1%			98.7% ± 1.8%		

ETS-8-044.1 Internal calibration Analyzed 5/8/14						
Lab ID	¹³ C ₂ -PFUnA			¹³ C ₄ -PFOS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140507-1	0.198	0.185	93.5	0.189	0.188	99.4
LCS-140507-2	0.198	0.208	105	0.189	0.189	99.8
LCS-140507-3	0.198	0.183	92.2	0.189	0.181	95.6
Average ± %RSD	96.9% ± 7.3%			98.3% ± 2.4%		
LCS-140507-4	1.98	2.05	104	1.89	1.99	106
LCS-140507-5	1.98	1.97	99.7	1.89	1.86	98.4
LCS-140507-6	1.98	2.06	104	1.89	1.92	102
Average ± %RSD	103% ± 2.4%			102% ± 3.7%		

NA = Not Applicable

(1) LCSs spiked above the upper limit of quantification.

Table 7 continued. Lab Control Spike Results.

ETS-8-044.1 External calibration Analyzed 5/12/14						
Lab ID	PFBA			PFOA (Linear + Branched)		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140512-1	0.199	0.270	136	0.191	0.221	116
LCS-140512-2	0.199	0.197	99.1	0.191	0.205	107
LCS-140512-3	0.199	0.212	107	0.191	0.210	110
Average ± %RSD	114% ± 17%			111% ± 4.1%		
LCS-140512-4	1.99	2.30	116	1.91	2.17	114
LCS-140512-5	1.99	2.30	115	1.91	2.21	116
LCS-140512-6	1.99	2.33	117	1.91	2.25	118
Average ± %RSD	116% ± 0.86%			116% ± 1.7%		
LCS-140512-7	20.0	22.5	113	19.1	21.3	112
LCS-140512-8	20.0	22.1	110	19.1	22.0	115
LCS-140512-9	20.0	22.2	111	19.1	21.6	113
Average ± %RSD	111% ± 1.4%			113% ± 1.4%		

ETS-8-044.1 External calibration Analyzed 5/12/14						
Lab ID	PFBS			PFHS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140512-1	0.199	0.248	125	0.199	0.227	114
LCS-140512-2	0.199	0.212	107	0.199	0.200	101
LCS-140512-3	0.199	0.218	109	0.199	0.212	106
Average ± %RSD	114% ± 8.7%			107% ± 6.1%		
LCS-140512-4	1.99	2.36	119	1.99	2.29	115
LCS-140512-5	1.99	2.40	120	1.99	2.32	116
LCS-140512-6	1.99	2.38	119	1.99	2.37	119
Average ± %RSD	119% ± 0.49%			117% ± 1.8%		
LCS-140512-7	20.0	22.3	111	20.0	22.0	110
LCS-140512-8	20.0	22.3	112	20.0	22.1	111
LCS-140512-9	20.0	22.7	113	20.0	22.3	111
Average ± %RSD	112% ± 0.89%			111% ± 0.52%		

NA = Not Applicable

(1) LCSs spiked above the upper limit of quantification.

Table 7 continued. Lab Control Spike Results.

ETS-8-044.1 External calibration Analyzed 5/12/14						
PFOS (Linear + Branched)				¹³ C ₄ -PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140512-1	0.185	0.188	102	0.190	0.214	112
LCS-140512-2	0.185	0.181	97.6	0.190	0.211	111
LCS-140512-3	0.185	0.188	102	0.190	0.216	113
Average ± %RSD	101% ± 2.5%			112% ± 0.89%		
LCS-140512-4	1.85	2.09	113	1.90	2.25	118
LCS-140512-5	1.85	2.15	116	1.90	2.24	118
LCS-140512-6	1.85	2.14	116	1.90	2.10	111
Average ± %RSD	115% ± 1.5%			116% ± 3.5%		
LCS-140512-7	18.5	21.0	114			
LCS-140512-8	18.5	21.3	115			
LCS-140512-9	18.5	21.5	116			
Average ± %RSD	115% ± 0.87%					

ETS-8-044.1 External calibration Analyzed 5/12/14						
¹³ C ₃ -PFBA				¹³ C ₄ -PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140512-1	0.198	0.237	120	0.199	0.227	114
LCS-140512-2	0.198	0.216	109	0.199	0.221	111
LCS-140512-3	0.198	0.234	118	0.199	0.217	109
Average ± %RSD	116% ± 5.1%			111% ± 2.3%		
LCS-140512-4	1.98	2.44	123	1.99	2.32	117
LCS-140512-5	1.98	2.40	121	1.99	2.33	117
LCS-140512-6	1.98	2.30	116	1.99	2.29	115
Average ± %RSD	120% ± 3.0%			116% ± 1.0%		

NA = Not Applicable

(1) LCSs spiked above the upper limit of quantification.

Table 7 continued. Lab Control Spike Results.

ETS-8-044.1 External calibration Analyzed 5/13/14			
PFPeA			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140512-1	0.199	0.210	106
LCS-140512-2	0.199	0.210	106
LCS-140512-3	0.199	0.218	109
Average ± %RSD	107% ± 1.6%		
LCS-140512-4	1.99	2.31	116
LCS-140512-5	1.99	2.33	117
LCS-140512-6	1.99	2.36	118
Average ± %RSD	117% ± 0.85%		
LCS-140512-7	20.0	23.1	115
LCS-140512-8	20.0	22.9	115
LCS-140512-9	20.0	22.9	115
Average ± %RSD	115% ± 0.0%		

ETS-8-044.1 Internal calibration Analyzed 5/14/14						
PFOS (Linear + Branched)				¹³ C ₄ -PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140513-1	0.0921	0.0948	103	0.0949	0.0895	94.3
LCS-140513-2	0.0921	0.0897	97.3	0.0949	0.0874	92.1
LCS-140513-3	0.0921	0.0966	105	0.0949	0.0973	103
Average ± %RSD	102% ± 3.9%			96.5% ± 6.0%		
LCS-140513-4	0.921	0.985	107	0.949	0.915	96.4
LCS-140513-5	0.921	0.973	106	0.949	0.887	93.5
LCS-140513-6	0.921	1.00	109	0.949	0.896	94.4
Average ± %RSD	107% ± 1.4%			94.8% ± 1.6%		
LCS-140513-7	3.67	3.88	106			
LCS-140513-8	3.67	3.96	108			
LCS-140513-9	3.67	3.95	108			
Average ± %RSD	107% ± 1.1%					

NA = Not Applicable

(1) LCSs spiked above the upper limit of quantification.

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.1, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFBA	Internal	8.90	±18%
PFPeA	Internal	9.09	±18%
PFHxA	Internal	7.78	±16%
PFHpA	Internal	9.35	±19%
PFOA	Internal	5.83	±12%
PFNA	Internal	9.65	±19%
PFDA	Internal	9.39	±19%
PFUnA	Internal	9.40	±19%
PFDoA	Internal	8.82	±18%
PFBS	Internal	5.21	±10%
PFHS	Internal	4.65	±9.3%
PFOS	Internal	7.82	±16%
PFBA	External	10.3	±21%
PFPeA	External	8.44	±17%
PFOA	External	6.87	±14%
PFBS	External	9.17	±18%
PFHS	External	5.62	±11%
PFOS	External	4.93	±9.9%

3.8 Field Matrix Spikes (FMS)

A field matrix spike sample was collected at each sampling point to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 0.5 times the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the extraction and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, each sample bottle contained stable isotope surrogate recovery spikes of ¹³C₃-PFBA, ¹³C₄-PFOA, ¹³C₂-PFUnA, and ¹³C₄-PFOS, which were added at a nominal concentration of 0.1 ng/mL to all sample bottles prior to sample collection. The ¹³C₃-PFBA and ¹³C₄-PFOA were selected to represent perfluorocarboxylic acids from C4-C8. The ¹³C₂-labeled PFUnA was selected to represent perfluorocarboxylic acids from C9-C12. The ¹³C₄-labeled PFOS was selected to represent the C4, C6, C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do

not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

$$\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration: Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} \times 100\%$$

Table 9. Field Matrix Spike Concentrations

Well ID	Final Spike Concentration (ng/mL)											
	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnA	PFDaA	PFBS	PFHS	PFOS
MW07, PW09, PW10	2.00	2.00	2.00	2.00	1.92	2.00	2.00	2.00	2.00	2.00	2.00	1.85
MW13	10.0	10.0	10.0	10.0	9.58	10.0	10.0	10.0	10.0	10.0	10.0	9.27
MW16	5.00	5.00	5.00	5.00	4.79	5.00	5.00	5.00	5.00	5.00	5.00	4.64
	50.0	NA	NA	NA	49.4	NA	NA	NA	NA	49.5	49.5	49.4
MW101, MW103, MW105, MW108	5.00	5.00	5.00	5.00	4.79	5.00	5.00	5.00	5.00	5.00	5.00	4.64
	100	NA	NA	NA	98.8	NA	NA	NA	NA	99.0	99.0	98.8
MW12, MW14R, MW102, MW104, MW110	10.0	10.0	10.0	10.0	9.58	10.0	10.0	10.0	10.0	10.0	10.0	9.27
	100	NA	NA	NA	98.8	NA	NA	NA	NA	99.0	99.0	98.8
Trip Blank	2.00	2.00	2.00	2.00	1.92	2.00	2.00	2.00	2.00	2.00	2.00	1.85
	10.0	10.0	10.0	10.0	9.58	10.0	10.0	10.0	10.0	10.0	10.0	9.27
	100	NA	NA	NA	98.8	NA	NA	NA	NA	99.0	99.0	98.8

NA = Not Applicable. The spiking solution did not contain all of the selected analytes.

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for the fourteen locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Relative percent difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

For those analytes where the field matrix spike level was not appropriate as compared to the sample concentration, the surrogate recovery standards were used to assess method accuracy. For all sampling locations, field matrix spikes (where applicable) and/or surrogate recovery standards met method acceptance criteria except for the following:

MW102: The low field matrix spike sample had a surrogate recovery of 69.0% for $^{13}\text{C}_4\text{-PFOS}$. Since the most appropriate field matrix spike sample met acceptance criteria and the average surrogate recovery for $^{13}\text{C}_4\text{-PFOS}$ was 73.3%, the method uncertainty was not expanded.

Trip Blank: The mid level field matrix spike recoveries were approximately 50% of the expected value. Based on other FMS spike levels at half of the concentration of the mid level, it is likely the mid field matrix spike was spiked at 5 ng/mL instead of the intended 10 ng/mL. All other field matrix spikes met acceptance criteria.

Table 10. CGMN-GW-MW07-140423 ⁽¹⁾

3M LIMS ID	Description	PFBA		PFPeA		PFHxA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-001	CGMN-GW-MW07-0-140423	2.48	NA	0.159	NA	0.0806	NA
ISO11-01-03-14-002	CGMN-GW-MW07-DB-140423	2.48	NA	0.147	NA	0.0749	NA
ISO11-01-03-14-003	CGMN-GW-MW07-FMS-140423	4.58	105	2.06	95.4	1.99	95.6
Average Concentration (ng/mL) ± %RPD		2.48 ng/mL ± 0.0%		0.153 ng/mL ± 7.8%		0.0778 ng/mL ± 7.3%	

3M LIMS ID	Description	PFHpA		PFOA		PFNA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-001	CGMN-GW-MW07-0-140423	<0.0250	NA	0.351	NA	<0.0250	NA
ISO11-01-03-14-002	CGMN-GW-MW07-DB-140423	<0.0250	NA	0.336	NA	<0.0250	NA
ISO11-01-03-14-003	CGMN-GW-MW07-FMS-140423	1.90	95.0	2.06	89.4	1.90	95.0
Average Concentration (ng/mL) ± %RPD		<0.0250 ng/mL		0.344 ng/mL ± 4.4%		<0.0250 ng/mL	

3M LIMS ID	Description	PFDA		PFUnA		PFDoA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-001	CGMN-GW-MW07-0-140423	<0.0250	NA	<0.0250	NA	NR	NA
ISO11-01-03-14-002	CGMN-GW-MW07-DB-140423	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-003	CGMN-GW-MW07-FMS-140423	1.86	93.0	1.90	95.0	1.92	96.0
Average Concentration (ng/mL) ± %RPD		<0.0250 ng/mL		<0.0250 ng/mL		<0.0250 ng/mL	

NA = Not Applicable
 NR = Not Reportable; sample area counts were below the LOQ area counts and did not meet method blank criteria
 (1) Samples were analyzed using internal standard calibration.

Table 10 continued. CGMN-GW-MW07-140423 ⁽¹⁾

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-001	CGMN-GW-MW07-0-140423	0.0357	NA	<0.0250	NA	0.0495	NA
ISO11-01-03-14-002	CGMN-GW-MW07-DB-140423	0.0363	NA	<0.0250	NA	0.0491	NA
ISO11-01-03-14-003	CGMN-GW-MW07-FMS-140423	1.66	81.2	1.77	88.5	1.87	98.4
Average Concentration (ng/mL) ± %RPD		0.0360 ng/mL ± 1.7%		<0.0250 ng/mL		0.0493 ng/mL ± 0.81%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₂ -PFUnA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-001	CGMN-GW-MW07-0-140423	98.6	90.8	95.6	99.6
ISO11-01-03-14-002	CGMN-GW-MW07-DB-140423	93.6	96.2	98.2	100
ISO11-01-03-14-003	CGMN-GW-MW07-FMS-140423	106	87.9	91.0	101
Average Recovery (%) ± %RSD		99.4% ± 6.3%	91.6% ± 4.6%	94.9% ± 3.8%	100% ± 0.72%

NA = Not Applicable
 NR = Not Reportable; sample area counts were below the LOQ area counts and did not meet method blank criteria
 (1) Samples were analyzed using internal standard calibration.

Table 11. CGMN-GW-MW12-140425

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽²⁾		PFHxA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-004	CGMN-GW-MW12-0-140425	218	NA	24.8	NA	26.4	NA
ISO11-01-03-14-005	CGMN-GW-MW12-DB-140425	222	NA	24.8	NA	27.1	NA
ISO11-01-03-14-006	CGMN-GW-MW12-LS-140425	NA ⁽³⁾	NA ⁽³⁾	35.0	NC	35.9	NC
ISO11-01-03-14-007	CGMN-GW-MW12-HS-140425	317	NC	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		220 ng/mL ± 1.8%		24.8 ng/mL ± 0.0%		26.8 ng/mL ± 2.6%	

3M LIMS ID	Description	PFHpA ⁽²⁾		PFOA ⁽¹⁾		PFNA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-004	CGMN-GW-MW12-0-140425	11.7	NA	545	NA	1.77	NA
ISO11-01-03-14-005	CGMN-GW-MW12-DB-140425	11.7	NA	550	NA	1.75	NA
ISO11-01-03-14-006	CGMN-GW-MW12-LS-140425	22.8	111	NA ⁽³⁾	NA ⁽³⁾	11.3	95.4
ISO11-01-03-14-007	CGMN-GW-MW12-HS-140425	NA ⁽⁴⁾	NA ⁽⁴⁾	652	NC	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		11.7 ng/mL ± 0.0%		548 ng/mL ± 0.91%		1.76 ng/mL ± 1.1%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 11 continued. CGMN-GW-MW12-140425

3M LIMS ID	Description	PFDA ⁽²⁾		PFUnA ⁽²⁾		PFDoA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-004	CGMN-GW-MW12-0-140425	1.82	NA	0.433	NA	0.154	NA
ISO11-01-03-14-005	CGMN-GW-MW12-DB-140425	1.87	NA	0.415	NA	0.169	NA
ISO11-01-03-14-006	CGMN-GW-MW12-LS-140425	11.5	96.5	10.7	103	10.2	100
ISO11-01-03-14-007	CGMN-GW-MW12-HS-140425	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		1.85 ng/mL ± 2.7%		0.424 ng/mL ± 4.2%		0.162 ng/mL ± 9.3%	

3M LIMS ID	Description	PFBS ⁽¹⁾		PFHS ⁽²⁾		PFOS ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-004	CGMN-GW-MW12-0-140425	67.5	NA	9.97	NA	115	NA
ISO11-01-03-14-005	CGMN-GW-MW12-DB-140425	70.3	NA	9.67	NA	118	NA
ISO11-01-03-14-006	CGMN-GW-MW12-LS-140425	NA ⁽³⁾	NA ⁽³⁾	20.3	105	NA ⁽³⁾	NA ⁽³⁾
ISO11-01-03-14-007	CGMN-GW-MW12-HS-140425	164	96.1	NA ⁽⁴⁾	NA ⁽⁴⁾	202	86.0
Average Concentration (ng/mL) ± %RPD		68.9 ng/mL ± 4.1%		9.82 ng/mL ± 3.1%		117 ng/mL ± 2.6%	

3M LIMS ID	Description	¹³ C ₃ -PFBA ⁽¹⁾	¹³ C ₄ -PFOA ⁽¹⁾	¹³ C ₂ -PFUnA ⁽²⁾	¹³ C ₄ -PFOS ⁽¹⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-004	CGMN-GW-MW12-0-140425	109	105	88.0	110
ISO11-01-03-14-005	CGMN-GW-MW12-DB-140425	113	105	96.3	108
ISO11-01-03-14-006	CGMN-GW-MW12-LS-140425	NA ⁽³⁾	NA ⁽³⁾	114	NA ⁽³⁾
ISO11-01-03-14-007	CGMN-GW-MW12-HS-140425	109	106	92.1	107
Average Recovery (%) ± %RSD		110% ± 2.1%	105% ± 0.55%	97.6% ± 12%	108% ± 1.4%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 12. CGMN-GW-MW13-140424 ⁽¹⁾

		PFBA		PFPeA		PFHxA	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-008	CGMN-GW-MW13-0-140424	7.45	NA	0.477	NA	0.265	NA
ISO11-01-03-14-009	CGMN-GW-MW13-DB-140424	6.83	NA	0.452	NA	0.249	NA
ISO11-01-03-14-010	CGMN-GW-MW13-FMS-140424	13.1	110	10.3	103	10.6	103
Average Concentration (ng/mL) ± %RPD		7.14 ng/mL ± 8.7%		0.465 ng/mL ± 5.4%		0.257 ng/mL ± 6.2%	

		PFHpA		PFOA		PFNA	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-008	CGMN-GW-MW13-0-140424	0.0883	NA	8.87	NA	0.0503	NA
ISO11-01-03-14-009	CGMN-GW-MW13-DB-140424	0.0674	NA	8.39	NA	0.0387	NA
ISO11-01-03-14-010	CGMN-GW-MW13-FMS-140424	9.47	93.9	18.6	104	9.81	97.7
Average Concentration (ng/mL) ± %RPD		0.0779 ng/mL ± 27% ⁽²⁾		8.63 ng/mL ± 5.6%		0.0445 ng/mL ± 26% ⁽²⁾	

		PFDA		PFUnA		PFDoA	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-008	CGMN-GW-MW13-0-140424	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-009	CGMN-GW-MW13-DB-140424	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-010	CGMN-GW-MW13-FMS-140424	9.66	96.6	9.91	99.1	9.94	99.4
Average Concentration (ng/mL) ± %RPD		<0.0250 ng/mL		<0.0250 ng/mL		<0.0250 ng/mL	

NA = Not Applicable

(1) Samples were analyzed using internal standard calibration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 12 continued. CGMN-GW-MW13-140424 ⁽¹⁾

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-008	CGMN-GW-MW13-0-140424	0.791	NA	0.594	NA	2.46	NA
ISO11-01-03-14-009	CGMN-GW-MW13-DB-140424	0.712	NA	0.517	NA	2.20	NA
ISO11-01-03-14-010	CGMN-GW-MW13-FMS-140424	9.12	83.7	9.76	92.0	11.9	103
Average Concentration (ng/mL) ± %RPD		0.752 ng/mL ± 11%		0.556 ng/mL ± 14%		2.33 ng/mL ± 11%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₂ -PFUnA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-008	CGMN-GW-MW13-0-140424	102	94.6	95.2	97.6
ISO11-01-03-14-009	CGMN-GW-MW13-DB-140424	101	102	85.9	94.8
ISO11-01-03-14-010	CGMN-GW-MW13-FMS-140424	91.8	98.3	109	101
Average Recovery (%) ± %RSD		98.3% ± 5.7%	98.3% ± 3.8%	96.7% ± 12%	97.8% ± 3.2%

NA = Not Applicable

(1) Samples were analyzed using internal standard calibration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 13. CGMN-GW-MW14R-140423

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽¹⁾		PFHxA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-0'1	CGMN-GW-MW14R-0-140423	645	NA	280	NA	195	NA
ISO11-01-03-14-0'2	CGMN-GW-MW14R-DB-140423	654	NA	275	NA	199	NA
ISO11-01-03-14-0'3	CGMN-GW-MW14R-LS-140423	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	193	NC
ISO11-01-03-14-0'4	CGMN-GW-MW14R-HS-140423	751	NC	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		650 ng/mL ± 1.4%		278 ng/mL ± 1.8%		197 ng/mL ± 2.0%	

3M LIMS ID	Description	PFHpA ⁽²⁾		PFOA ⁽¹⁾		PFNA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-0'1	CGMN-GW-MW14R-0-140423	25.4	NA	542	NA	1.70	NA
ISO11-01-03-14-0'2	CGMN-GW-MW14R-DB-140423	26.7	NA	543	NA	1.74	NA
ISO11-01-03-14-0'3	CGMN-GW-MW14R-LS-140423	34.8	NC	NA ⁽³⁾	NA ⁽³⁾	11.6	98.8
ISO11-01-03-14-0'4	CGMN-GW-MW14R-HS-140423	NA ⁽⁴⁾	NA ⁽⁴⁾	646	NC	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		26.1 ng/mL ± 5.0%		543 ng/mL ± 0.18%		1.72 ng/mL ± 2.3%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 13 continued. CGMN-GW-MW14R-140423

3M LIMS ID	Description	PFDA ⁽²⁾		PFUnA ⁽²⁾		PFDoA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-0' 1	CGMN-GW-MW14R-0-140423	2.11	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-0' 2	CGMN-GW-MW14R-DB-140423	2.08	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-0' 3	CGMN-GW-MW14R-LS-140423	11.4	93.0	9.51	95.1	9.81	98.1
ISO11-01-03-14-0' 4	CGMN-GW-MW14R-HS-140423	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		2.10 ng/mL ± 1.4%		<0.0250 ng/mL		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS ⁽²⁾		PFHS ⁽²⁾		PFOS ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-0' 1	CGMN-GW-MW14R-0-140423	18.2	NA	19.7	NA	348	NA
ISO11-01-03-14-0' 2	CGMN-GW-MW14R-DB-140423	18.2	NA	20.3	NA	358	NA
ISO11-01-03-14-0' 3	CGMN-GW-MW14R-LS-140423	26.0	78.0	29.9	99.0	NA ⁽³⁾	NA ⁽³⁾
ISO11-01-03-14-0' 4	CGMN-GW-MW14R-HS-140423	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	461	NC
Average Concentration (ng/mL) ± %RPD		18.2 ng/mL ± 0.0%		20.0 ng/mL ± 3.0%		353 ng/mL ± 2.8%	

3M LIMS ID	Description	¹³ C ₂ -PFBA ⁽¹⁾	¹³ C ₂ -PFOA ⁽¹⁾	¹³ C ₂ -PFUnA ⁽²⁾	¹³ C ₂ -PFOS ⁽¹⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-0' 1	CGMN-GW-MW14R-0-140423	107	108	99.8	108
ISO11-01-03-14-0' 2	CGMN-GW-MW14R-DB-140423	112	109	96.0	111
ISO11-01-03-14-0' 3	CGMN-GW-MW14R-LS-140423	NA ⁽³⁾	NA ⁽³⁾	95.8	NA ⁽³⁾
ISO11-01-03-14-0' 4	CGMN-GW-MW14R-HS-140423	111	106	102	113
Average Recovery (%) ± %RSD		110% ± 2.4%	108% ± 1.4%	98.4% ± 3.1%	111% ± 2.3%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 14. CGMN-GW-MW16-140424

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽¹⁾		PFHxA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-0'5	CGMN-GW-MW16-0-140424	41.1	NA	6.91	NA	6.57	NA
ISO11-01-03-14-0'6	CGMN-GW-MW16-DB-140424	40.3	NA	6.52	NA	6.42	NA
ISO11-01-03-14-0'7	CGMN-GW-MW16-LS-140424	45.7	NC	11.7	99.6	11.5	100
ISO11-01-03-14-0'8	CGMN-GW-MW16-HS-140424	91.0	101	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		40.7 ng/mL ± 2.0%		6.72 ng/mL ± 5.8%		6.50 ng/mL ± 2.3%	

3M LIMS ID	Description	PFHpA ⁽¹⁾		PFOA ⁽¹⁾		PFNA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-0'5	CGMN-GW-MW16-0-140424	3.44	NA	89.1	NA	0.546	NA
ISO11-01-03-14-0'6	CGMN-GW-MW16-DB-140424	3.15	NA	82.3	NA	0.523	NA
ISO11-01-03-14-0'7	CGMN-GW-MW16-LS-140424	8.42	102	94.3	NC	5.52	99.7
ISO11-01-03-14-0'8	CGMN-GW-MW16-HS-140424	NA ⁽²⁾	NA ⁽²⁾	123	75.5	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		3.30 ng/mL ± 8.8%		85.7 ng/mL ± 7.9%		0.535 ng/mL ± 4.3%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) The spike level did not contain the target analyte.

(3) Samples were analyzed using external standard calibration.

(4) Samples were not re-analyzed for this analyte.

Table 14 continued. CGMN-GW-MW16-140424

3M LIMS ID	Description	PFDA ⁽¹⁾		PFUnA ⁽¹⁾		PFDoA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-0'5	CGMN-GW-MW16-0-140424	0.329	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-0'6	CGMN-GW-MW16-DB-140424	0.339	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-0'7	CGMN-GW-MW16-LS-140424	5.02	93.7	4.75	95.0	4.86	97.2
ISO11-01-03-14-0'8	CGMN-GW-MW16-HS-140424	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		0.334 ng/mL ± 3.0%		<0.0250 ng/mL		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS ⁽³⁾		PFHS ⁽¹⁾		PFOS ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-0'5	CGMN-GW-MW16-0-140424	37.7	NA	4.43	NA	51.4	NA
ISO11-01-03-14-0'6	CGMN-GW-MW16-DB-140424	38.1	NA	4.55	NA	52.4	NA
ISO11-01-03-14-0'7	CGMN-GW-MW16-LS-140424	NA ⁽⁴⁾	NA ⁽⁴⁾	9.30	96.2	59.2	NC
ISO11-01-03-14-0'8	CGMN-GW-MW16-HS-140424	89.0	103	48.6	89.1	108	114
Average Concentration (ng/mL) ± %RPD		37.9 ng/mL ± 1.1%		4.49 ng/mL ± 2.7%		51.9 ng/mL ± 1.9%	

3M LIMS ID	Description	¹³ C ₃ -PFBA ⁽¹⁾	¹³ C ₄ -PFOA ⁽¹⁾	¹³ C ₂ -PFUnA ⁽¹⁾	¹³ C ₄ -PFOS ⁽¹⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-0'5	CGMN-GW-MW16-0-140424	101	100	93.5	96.0
ISO11-01-03-14-0'6	CGMN-GW-MW16-DB-140424	97.2	87.2	92.2	104
ISO11-01-03-14-0'7	CGMN-GW-MW16-LS-140424	100	108	98.5	98.3
ISO11-01-03-14-0'8	CGMN-GW-MW16-HS-140424	100	92.7	107	104
Average Recovery (%) ± %RSD		99.6% ± 1.6%	97.0% ± 9.3%	97.8% ± 6.9%	101% ± 4.0%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) The spike level did not contain the target analyte.

(3) Samples were analyzed using external standard calibration.

(4) Samples were not re-analyzed for this analyte.

Table 15. CGMN-GW-MW101-140425

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽²⁾		PFHxA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-019	CGMN-GW-MW101-0-140425	1330	NA	71.8	NA	60.9	NA
ISO11-01-03-14-020	CGMN-GW-MW101-DB-140425	1330	NA	72.6	NA	61.3	NA
ISO11-01-03-14-021	CGMN-GW-MW101-LS-140425	NA ⁽³⁾	NA ⁽³⁾	75.1	NC	68.4	NC
ISO11-01-03-14-022	CGMN-GW-MW101-HS-140425	1410	NC	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		1330 ng/mL ± 0.0%		72.2 ng/mL ± 1.1%		61.1 ng/mL ± 0.65%	

3M LIMS ID	Description	PFHpA ⁽²⁾		PFOA ⁽¹⁾		PFNA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-019	CGMN-GW-MW101-0-140425	63.2	NA	76.1	NA	7.08	NA
ISO11-01-03-14-020	CGMN-GW-MW101-DB-140425	61.4	NA	75.6	NA	6.84	NA
ISO11-01-03-14-021	CGMN-GW-MW101-LS-140425	67.7	NC	NA ⁽³⁾	NA ⁽³⁾	11.9	98.8
ISO11-01-03-14-022	CGMN-GW-MW101-HS-140425	NA ⁽⁴⁾	NA ⁽⁴⁾	177	102	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		62.3 ng/mL ± 2.9%		76.0 ng/mL ± 0.39%		6.96 ng/mL ± 3.4%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 15 continued. CGMN-GW-MW101-140425

3M LIMS ID	Description	PFDA ⁽²⁾		PFUnA ⁽²⁾		PFDoA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-019	CGMN-GW-MW101-0-140425	0.248	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-020	CGMN-GW-MW101-DB-140425	0.233	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-021	CGMN-GW-MW101-LS-140425	5.11	97.4	4.78	95.6	4.94	98.8
ISO11-01-03-14-022	CGMN-GW-MW101-HS-140425	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.241 ng/mL ± 6.2%		<0.0250 ng/mL		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS ⁽¹⁾		PFHS ⁽¹⁾		PFOS ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-019	CGMN-GW-MW101-0-140425	37.9	NA	364	NA	136	NA
ISO11-01-03-14-020	CGMN-GW-MW101-DB-140425	37.6	NA	364	NA	134	NA
ISO11-01-03-14-021	CGMN-GW-MW101-LS-140425	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾
ISO11-01-03-14-022	CGMN-GW-MW101-HS-140425	142	105	461	NC	231	97.2
Average Concentration (ng/mL) ± %RPD		37.8 ng/mL ± 0.79%		364 ng/mL ± 0.0%		135 ng/mL ± 1.5%	

3M LIMS ID	Description	¹³ C ₃ -PFBA ⁽¹⁾	¹³ C ₄ -PFOA ⁽¹⁾	¹³ C ₂ -PFUnA ⁽²⁾	¹³ C ₄ -PFOS ⁽¹⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-019	CGMN-GW-MW101-0-140425	107	107	93.1	110
ISO11-01-03-14-020	CGMN-GW-MW101-DB-140425	106	106	96.1	111
ISO11-01-03-14-021	CGMN-GW-MW101-LS-140425	NA ⁽³⁾	NA ⁽³⁾	96.8	NA ⁽³⁾
ISO11-01-03-14-022	CGMN-GW-MW101-HS-140425	106	104	91.3	109
Average Recovery (%) ± %RSD		106% ± 0.54%	106% ± 1.4%	94.3% ± 2.7%	110% ± 0.91%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 16. CGMN-GW-MW102-140425

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽²⁾		PFHxA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-050	CGMN-GW-MW102-0-140425	924	NA	30.7	NA	29.1	NA
ISO11-01-03-14-051	CGMN-GW-MW102-DB-140425	914	NA	30.6	NA	28.5	NA
ISO11-01-03-14-052	CGMN-GW-MW102-LS-140425	NA ⁽³⁾	NA ⁽³⁾	40.3	NC	39.8	NC
ISO11-01-03-14-053	CGMN-GW-MW102-HS-140425	995	NC	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		919 ng/mL ± 1.1%		30.7 ng/mL ± 0.33%		28.8 ng/mL ± 2.1%	

3M LIMS ID	Description	PFHpA ⁽²⁾		PFOA ⁽²⁾		PFNA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-050	CGMN-GW-MW102-0-140425	16.1	NA	58.6	NA	1.84	NA
ISO11-01-03-14-051	CGMN-GW-MW102-DB-140425	15.8	NA	57.6	NA	1.90	NA
ISO11-01-03-14-052	CGMN-GW-MW102-LS-140425	26.9	109	64.6	NC	11.7	98.3
ISO11-01-03-14-053	CGMN-GW-MW102-HS-140425	NA ⁽⁴⁾	NA ⁽⁴⁾	140	82.9	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		16.0 ng/mL ± 1.9%		58.1 ng/mL ± 1.7%		1.87 ng/mL ± 3.2%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

- (1) Samples were analyzed using external standard calibration.
- (2) Samples were analyzed using internal standard calibration.
- (3) Samples were not re-analyzed for this analyte.
- (4) The spike level did not contain the target analyte.
- (5) Surrogate recovery did not meet acceptance criteria of 100 ± 30%.

Table 16 continued. CGMN-GW-MW102-140425

3M LIMS ID	Description	PFDA ⁽²⁾		PFUnA ⁽²⁾		PFDoA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-050	CGMN-GW-MW102-0-140425	0.143	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-051	CGMN-GW-MW102-DB-140425	0.129	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-052	CGMN-GW-MW102-LS-140425	9.41	92.7	9.44	94.4	9.74	97.4
ISO11-01-03-14-053	CGMN-GW-MW102-HS-140425	NA ⁽⁵⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		0.136 ng/mL ± 10%		<0.0250 ng/mL		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS ⁽²⁾		PFHS ⁽¹⁾		PFOS ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-050	CGMN-GW-MW102-0-140425	7.10	NA	146	NA	67.1	NA
ISO11-01-03-14-051	CGMN-GW-MW102-DB-140425	7.14	NA	147	NA	67.6	NA
ISO11-01-03-14-052	CGMN-GW-MW102-LS-140425	15.3	81.8	NA ⁽³⁾	NA ⁽³⁾	71.5	NC
ISO11-01-03-14-053	CGMN-GW-MW102-HS-140425	NA ⁽⁵⁾	NA ⁽⁵⁾	244	98.0	143	76.5
Average Concentration (ng/mL) ± %RPD		7.12 ng/mL ± 0.56%		147 ng/mL ± 0.68%		67.4 ng/mL ± 0.74%	

3M LIMS ID	Description	¹³ C ₃ -PFBA ⁽¹⁾	¹³ C ₄ -PFOA ⁽²⁾	¹³ C ₂ -PFUnA ⁽²⁾	¹³ C ₄ -PFOS ⁽²⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-050	CGMN-GW-MW102-0-140425	110	103	92.2	73.9
ISO11-01-03-14-051	CGMN-GW-MW102-DB-140425	110	104	93.3	76.6
ISO11-01-03-14-052	CGMN-GW-MW102-LS-140425	NA ⁽³⁾	95.3	95.1	69.0 ⁽⁵⁾
ISO11-01-03-14-053	CGMN-GW-MW102-HS-140425	107	103	97.0	73.5
Average Recovery (%) ± %RSD		109% ± 1.6%	101% ± 4.0%	94.4% ± 2.2%	73.3% ± 4.3%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

- (1) Samples were analyzed using external standard calibration.
- (2) Samples were analyzed using internal standard calibration.
- (3) Samples were not re-analyzed for this analyte.
- (4) The spike level did not contain the target analyte.
- (5) Surrogate recovery did not meet acceptance criteria of 100 ± 30%.

Table 17. CGMN-GW-MW103-140425

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽²⁾		PFHxA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-054	CGMN-GW-MW103-0-140425	238	NA	27.3	NA	57.0	NA
ISO11-01-03-14-055	CGMN-GW-MW103-DB-140425	243	NA	29.1	NA	57.8	NA
ISO11-01-03-14-056	CGMN-GW-MW103-LS-140425	NA ⁽³⁾	NA ⁽³⁾	32.0	NC	57.7	NC
ISO11-01-03-14-057	CGMN-GW-MW103-HS-140425	331	NC	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		241 ng/mL ± 2.1%		28.2 ng/mL ± 6.4%		57.4 ng/mL ± 1.4%	

3M LIMS ID	Description	PFHpA ⁽²⁾		PFOA ⁽¹⁾		PFNA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-054	CGMN-GW-MW103-0-140425	31.4	NA	117	NA	0.0487	NA
ISO11-01-03-14-055	CGMN-GW-MW103-DB-140425	30.4	NA	125	NA	0.0566	NA
ISO11-01-03-14-056	CGMN-GW-MW103-LS-140425	36.0	02	NA ⁽³⁾	NA ⁽³⁾	4.76	94.1
ISO11-01-03-14-057	CGMN-GW-MW103-HS-140425	NA ⁽⁴⁾	NA ⁽⁴⁾	227	107	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		30.9 ng/mL ± 3.2%		121 ng/mL ± 6.6%		0.0527 ng/mL ± 15%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 17 continued. CGMN-GW-MW103-140425

3M LIMS ID	Description	PFDA ⁽²⁾		PFUnA ⁽²⁾		PFDoA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-054	CGMN-GW-MW103-0-140425	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-055	CGMN-GW-MW103-DB-140425	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-056	CGMN-GW-MW103-LS-140425	4.75	95.0	4.90	98.0	4.85	97.0
ISO11-01-03-14-057	CGMN-GW-MW103-HS-140425	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		<0.0250 ng/mL		<0.0250 ng/mL		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS ⁽²⁾		PFHS ⁽¹⁾		PFOS ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-054	CGMN-GW-MW103-0-140425	6.51	NA	21.0	NA	7.99	NA
ISO11-01-03-14-055	CGMN-GW-MW103-DB-140425	6.51	NA	22.5	NA	7.64	NA
ISO11-01-03-14-056	CGMN-GW-MW103-LS-140425	10.3	75.8	NA ⁽³⁾	NA ⁽³⁾	11.9	87.9
ISO11-01-03-14-057	CGMN-GW-MW103-HS-140425	NA ⁽³⁾	NA ⁽³⁾	124	103	108	101
Average Concentration (ng/mL) ± %RPD		6.51 ng/mL ± 0.0%		21.8 ng/mL ± 6.9%		7.82 ng/mL ± 4.5%	

3M LIMS ID	Description	¹³ C ₃ -PFBA ⁽¹⁾	¹³ C ₄ -PFOA ⁽¹⁾	¹³ C ₂ -PFUnA ⁽²⁾	¹³ C ₂ -PFOS ⁽²⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-054	CGMN-GW-MW103-0-140425	108	109	92.9	92.0
ISO11-01-03-14-055	CGMN-GW-MW103-DB-140425	110	109	97.6	98.1
ISO11-01-03-14-056	CGMN-GW-MW103-LS-140425	NA ⁽³⁾	NA ⁽³⁾	99.0	89.8
ISO11-01-03-14-057	CGMN-GW-MW103-HS-140425	107	110	95.4	97.0
Average Recovery (%) ± %RSD		108% ± 1.4%	109% ± 0.53%	96.2% ± 2.8%	94.2% ± 4.2%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 18. CGMN-GW-MW104-140425

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽¹⁾		PFHxA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-023	CGMN-GW-MW104-0-140425	107	NA	12.7	NA	19.9	NA
ISO11-01-03-14-024	CGMN-GW-MW104-DB-140425	107	NA	12.4	NA	19.5	NA
ISO11-01-03-14-025	CGMN-GW-MW104-LS-140425	114	NC	22.6	100	28.8	91.0
ISO11-01-03-14-026	CGMN-GW-MW104-HS-140425	197	90.0	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		107 ng/mL ± 0.0%		12.6 ng/mL ± 2.4%		19.7 ng/mL ± 2.0%	

3M LIMS ID	Description	PFHpA ⁽¹⁾		PFOA ⁽¹⁾		PFNA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-023	CGMN-GW-MW104-0-140425	9.08	NA	82.0	NA	2.57	NA
ISO11-01-03-14-024	CGMN-GW-MW104-DB-140425	9.13	NA	82.1	NA	2.60	NA
ISO11-01-03-14-025	CGMN-GW-MW104-LS-140425	18.4	92.9	92.1	NC	12.3	97.1
ISO11-01-03-14-026	CGMN-GW-MW104-HS-140425	NA ⁽²⁾	NA ⁽²⁾	158	79.2	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		9.11 ng/mL ± 0.55%		82.1 ng/mL ± 0.12%		2.59 ng/mL ± 1.2%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) The spike level did not contain the target analyte.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) Samples were not re-analyzed for this analyte.

(5) Samples were analyzed using external standard calibration.

Table 18 continued. CGMN-GW-MW104-140425

3M LIMS ID	Description	PFDA ⁽¹⁾		PFUnA ⁽¹⁾		PFDoA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-023	CGMN-GW-MW104-0-140425	3.20	NA	0.0489	NA	<0.0250	NA
ISO11-01-03-14-024	CGMN-GW-MW104-DB-140425	3.32	NA	0.0625	NA	<0.0250	NA
ISO11-01-03-14-025	CGMN-GW-MW104-LS-140425	13.3	100	10.0	99.4	9.83	98.3
ISO11-01-03-14-026	CGMN-GW-MW104-HS-140425	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		3.26 ng/mL ± 3.7%		0.0557 ng/mL ± 24% ⁽³⁾		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS ⁽¹⁾		PFHS ⁽¹⁾		PFOS ⁽⁵⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-023	CGMN-GW-MW104-0-140425	7.95	NA	12.8	NA	238	NA
ISO11-01-03-14-024	CGMN-GW-MW104-DB-140425	8.25	NA	12.3	NA	229	NA
ISO11-01-03-14-025	CGMN-GW-MW104-LS-140425	15.6	75.0	21.9	93.0	NA ⁽⁴⁾	NA ⁽⁴⁾
ISO11-01-03-14-026	CGMN-GW-MW104-HS-140425	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	332	NC
Average Concentration (ng/mL) ± %RPD		8.10 ng/mL ± 3.7%		12.6 ng/mL ± 4.0%		234 ng/mL ± 3.8%	

3M LIMS ID	Description	¹³ C ₃ -PFBA ⁽¹⁾	¹³ C ₄ -PFOA ⁽¹⁾	¹³ C ₂ -PFUnA ⁽¹⁾	¹³ C ₂ -PFOS ⁽⁵⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-023	CGMN-GW-MW104-0-140425	99.2	86.7	101	111
ISO11-01-03-14-024	CGMN-GW-MW104-DB-140425	100	96.9	102	111
ISO11-01-03-14-025	CGMN-GW-MW104-LS-140425	99.1	96.1	113	NA ⁽⁴⁾
ISO11-01-03-14-026	CGMN-GW-MW104-HS-140425	96.1	98.7	98.6	101
Average Recovery (%) ± %RSD		98.6% ± 1.7%	94.6% ± 5.7%	104% ± 6.1%	108% ± 5.3%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) The spike level did not contain the target analyte.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) Samples were not re-analyzed for this analyte.

(5) Samples were analyzed using external standard calibration.

Table 19. CGMN-GW-MW105-140425 ⁽¹⁾

3M LIMS ID	Description	PFBA		PFPeA		PFHxA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-027	CGMN-GW-MW105-0-140425	70.3	NA	5.86	NA	11.7	NA
ISO11-01-03-14-028	CGMN-GW-MW105-DB-140425	71.7	NA	6.24	NA	11.8	NA
ISO11-01-03-14-029	CGMN-GW-MW105-LS-140425	74.1	NC	10.5	89.0	16.6	NC
ISO11-01-03-14-030	CGMN-GW-MW105-HS-140425	164	93.0	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		71.0 ng/mL ± 2.0%		6.05 ng/mL ± 6.3%		11.8 ng/mL ± 0.85%	

3M LIMS ID	Description	PFHpA		PFOA		PFNA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-027	CGMN-GW-MW105-0-140425	8.38	NA	42.7	NA	0.577	NA
ISO11-01-03-14-028	CGMN-GW-MW105-DB-140425	8.89	NA	45.9	NA	0.588	NA
ISO11-01-03-14-029	CGMN-GW-MW105-LS-140425	13.5	97.2	47.2	NC	5.51	98.5
ISO11-01-03-14-030	CGMN-GW-MW105-HS-140425	NA ⁽²⁾	NA ⁽²⁾	122	78.6	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		8.64 ng/mL ± 5.9%		44.3 ng/mL ± 7.2%		0.583 ng/mL ± 1.9%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) The spike level did not contain the target analyte.

(3) Samples were not re-analyzed for this analyte.

Table 19 continued. CGMN-GW-MW105-140425 ⁽¹⁾

3M LIMS ID	Description	PFDA		PFUnA		PFDoA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-027	CGMN-GW-MW105-0-140425	0.328	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-028	CGMN-GW-MW105-DB-140425	0.339	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-029	CGMN-GW-MW105-LS-140425	5.10	95.3	4.91	98.2	4.83	96.6
ISO11-01-03-14-030	CGMN-GW-MW105-HS-140425	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		0.334 ng/mL ± 3.3%		<0.0250 ng/mL		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-027	CGMN-GW-MW105-0-140425	7.56	NA	8.48	NA	56.6	NA
ISO11-01-03-14-028	CGMN-GW-MW105-DB-140425	7.80	NA	8.70	NA	60.7	NA
ISO11-01-03-14-029	CGMN-GW-MW105-LS-140425	12.0	86.4	12.9	86.2	66.1	NC
ISO11-01-03-14-030	CGMN-GW-MW105-HS-140425	NA ⁽³⁾	NA ⁽³⁾	97.0	89.3	169	112
Average Concentration (ng/mL) ± %RPD		7.68 ng/mL ± 3.1%		8.59 ng/mL ± 2.6%		58.7 ng/mL ± 7.0%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₂ -PFUnA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-027	CGMN-GW-MW105-0-140425	96.4	96.6	92.9	88.0
ISO11-01-03-14-028	CGMN-GW-MW105-DB-140425	102	99.3	98.4	95.5
ISO11-01-03-14-029	CGMN-GW-MW105-LS-140425	100	101	94.4	90.6
ISO11-01-03-14-030	CGMN-GW-MW105-HS-140425	99.5	104	95.8	96.9
Average Recovery (%) ± %RSD		99.5% ± 2.3%	100% ± 3.5%	95.4% ± 2.5%	92.8% ± 4.5%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) The spike level did not contain the target analyte.

(3) Samples were not re-analyzed for this analyte.

Table 20. CGMN-GW-MW108-140425

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽¹⁾		PFHxA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-031	CGMN-GW-MW108-0-140425	59.1	NA	10.6	NA	9.39	NA
ISO11-01-03-14-032	CGMN-GW-MW108-DB-140425	71.6	NA	12.3	NA	11.0	NA
ISO11-01-03-14-033	CGMN-GW-MW108-LS-140425	74.3	NC	17.0	NC	16.3	NC
ISO11-01-03-14-034	CGMN-GW-MW108-HS-140425	156	90.6	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		65.4 ng/mL ± 19%		11.5 ng/mL ± 15%		10.2 ng/mL ± 16%	

3M LIMS ID	Description	PFHpA ⁽¹⁾		PFOA ⁽²⁾		PFNA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-031	CGMN-GW-MW108-0-140425	3.84	NA	166	NA	1.15	NA
ISO11-01-03-14-032	CGMN-GW-MW108-DB-140425	4.45	NA	162	NA	1.36	NA
ISO11-01-03-14-033	CGMN-GW-MW108-LS-140425	10.1	19	NA ⁽⁴⁾	NA ⁽⁴⁾	6.43	103
ISO11-01-03-14-034	CGMN-GW-MW108-HS-140425	NA ⁽²⁾	NA ⁽²⁾	265	102	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		4.15 ng/mL ± 15%		164 ng/mL ± 2.4%		1.26 ng/mL ± 17%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) The spike level did not contain the target analyte.

(3) Samples were analyzed using external standard calibration.

(4) Samples were not re-analyzed for this analyte.

Table 20 continued. CGMN-GW-MW108-140425

3M LIMS ID	Description	PFDA ⁽¹⁾		PFUnA ⁽¹⁾		PFDoA ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-031	CGMN-GW-MW108-0-140425	0.516	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-032	CGMN-GW-MW108-DB-140425	0.607	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-033	CGMN-GW-MW108-LS-140425	5.34	95.6	4.95	99.0	4.98	99.6
ISO11-01-03-14-034	CGMN-GW-MW108-HS-140425	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾	NA ⁽²⁾
Average Concentration (ng/mL) ± %RPD		0.562 ng/mL ± 16%		<0.0250 ng/mL		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS ⁽³⁾		PFHS ⁽¹⁾		PFOS ⁽¹⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-031	CGMN-GW-MW108-0-140425	17.8	NA	3.32	NA	40.2	NA
ISO11-01-03-14-032	CGMN-GW-MW108-DB-140425	17.0	NA	3.84	NA	46.4	NA
ISO11-01-03-14-033	CGMN-GW-MW108-LS-140425	NA ⁽⁵⁾	NA ⁽⁵⁾	8.85	105	52.2	NC
ISO11-01-03-14-034	CGMN-GW-MW108-HS-140425	124	108	93.0	90.3	153	111
Average Concentration (ng/mL) ± %RPD		17.4 ng/mL ± 4.6%		3.58 ng/mL ± 15%		43.3 ng/mL ± 14%	

3M LIMS ID	Description	¹³ C ₃ -PFBA ⁽¹⁾	¹³ C ₄ -PFOA ⁽³⁾	¹³ C ₂ -PFUnA ⁽¹⁾	¹³ C ₄ -PFOS ⁽¹⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-031	CGMN-GW-MW108-0-140425	97.6	98.8	101	98.6
ISO11-01-03-14-032	CGMN-GW-MW108-DB-140425	96.6	97.4	99.2	89.5
ISO11-01-03-14-033	CGMN-GW-MW108-LS-140425	99.3	NA ⁽⁴⁾	102	103
ISO11-01-03-14-034	CGMN-GW-MW108-HS-140425	98.8	99.4	94.6	94.1
Average Recovery (%) ± %RSD		98.1% ± 1.2%	98.5% ± 1.0%	99.2% ± 3.3%	96.3% ± 6.0%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) The spike level did not contain the target analyte.

(3) Samples were analyzed using external standard calibration.

(4) Samples were not re-analyzed for this analyte.

Table 21. CGMN-GW-MW110-140424

3M LIMS ID	Description	PFBA ⁽¹⁾		PFPeA ⁽²⁾		PFHxA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-035	CGMN-GW-MW110-0-140424	186	NA	24.6	NA	34.0	NA
ISO11-01-03-14-036	CGMN-GW-MW110-DB-140424	191	NA	23.4	NA	31.7	NA
ISO11-01-03-14-037	CGMN-GW-MW110-LS-140424	NA ⁽³⁾	NA ⁽³⁾	38.4	90.6	47.6	NC
ISO11-01-03-14-038	CGMN-GW-MW110-HS-140424	306	~17	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		189 ng/mL ± 2.6%		24.0 ng/mL ± 5.0%		32.9 ng/mL ± 7.0%	

3M LIMS ID	Description	PFHpA ⁽²⁾		PFOA ⁽¹⁾		PFNA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-035	CGMN-GW-MW110-0-140424	12.5	NA	310	NA	0.191	NA
ISO11-01-03-14-036	CGMN-GW-MW110-DB-140424	12.8	NA	310	NA	0.197	NA
ISO11-01-03-14-037	CGMN-GW-MW110-LS-140424	28.6	~00	NA ⁽³⁾	NA ⁽³⁾	15.8	98.2
ISO11-01-03-14-038	CGMN-GW-MW110-HS-140424	NA ⁽⁴⁾	NA ⁽⁴⁾	410	NC	NA ⁽⁴⁾	NA ⁽⁴⁾
Average Concentration (ng/mL) ± %RPD		12.7 ng/mL ± 2.4%		310 ng/mL ± 0.0%		0.194 ng/mL ± 3.1%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 21 continued. CGMN-GW-MW110-140424

3M LIMS ID	Description	PFDA ⁽²⁾		PFUnA ⁽²⁾		PFDoA ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-035	CGMN-GW-MW110-0-140424	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-036	CGMN-GW-MW110-DB-140424	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-037	CGMN-GW-MW110-LS-140424	15.3	96.2	15.5	97.5	15.3	96.2
ISO11-01-03-14-038	CGMN-GW-MW110-HS-140424	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		<0.0250 ng/mL		<0.0250 ng/mL		<0.0250 ng/mL	

3M LIMS ID	Description	PFBS ⁽¹⁾		PFHS ⁽²⁾		PFOS ⁽²⁾	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-035	CGMN-GW-MW110-0-140424	87.1	NA	21.2	NA	12.4	NA
ISO11-01-03-14-036	CGMN-GW-MW110-DB-140424	85.1	NA	20.5	NA	12.6	NA
ISO11-01-03-14-037	CGMN-GW-MW110-LS-140424	NA ⁽³⁾	NA ⁽³⁾	33.7	80.5	26.3	93.9
ISO11-01-03-14-038	CGMN-GW-MW110-HS-140424	185	99.9	NA	NA	114	103
Average Concentration (ng/mL) ± %RPD		86.1 ng/mL ± 2.3%		20.9 ng/mL ± 3.3%		12.5 ng/mL ± 1.6%	

3M LIMS ID	Description	¹³ C ₃ -PFBA ⁽¹⁾	¹³ C ₄ -PFOA ⁽¹⁾	¹³ C ₂ -PFUnA ⁽²⁾	¹³ C ₂ -PFOS ⁽²⁾
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-035	CGMN-GW-MW110-0-140424	101	102	100	93.6
ISO11-01-03-14-036	CGMN-GW-MW110-DB-140424	96.6	97.3	95.3	91.7
ISO11-01-03-14-037	CGMN-GW-MW110-LS-140424	NA ⁽³⁾	NA ⁽³⁾	106	92.8
ISO11-01-03-14-038	CGMN-GW-MW110-HS-140424	104	99.8	99.8	96.4
Average Recovery (%) ± %RSD		101% ± 3.7%	99.7% ± 2.4%	100% ± 4.4%	93.6% ± 2.1%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using external standard calibration.

(2) Samples were analyzed using internal standard calibration.

(3) Samples were not re-analyzed for this analyte.

(4) The spike level did not contain the target analyte.

Table 22. CGMN-GW-PW09-140425 ⁽¹⁾

3M LIMS ID	Description	PFBA		PFPeA		PFHxA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-039	CGMN-GW-PW09-0-140425	4.18	NA	0.268	NA	0.127	NA
ISO11-01-03-14-040	CGMN-GW-PW09-DB-140425	4.10	NA	0.275	NA	0.143	NA
ISO11-01-03-14-041	CGMN-GW-PW09-FMS-140425	6.14	NC	2.17	94.9	2.08	97.3
Average Concentration (ng/mL) ± %RPD		4.14 ng/mL ± 1.9%		0.272 ng/mL ± 2.6%		0.135 ng/mL ± 12%	

3M LIMS ID	Description	PFHpA		PFOA		PFNA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-039	CGMN-GW-PW09-0-140425	0.0443	NA	1.03	NA	0.0353	NA
ISO11-01-03-14-040	CGMN-GW-PW09-DB-140425	0.0498	NA	1.07	NA	0.0377	NA
ISO11-01-03-14-041	CGMN-GW-PW09-FMS-140425	2.14	105	2.69	85.4	1.89	92.7
Average Concentration (ng/mL) ± %RPD		0.0471 ng/mL ± 12%		1.05 ng/mL ± 3.8%		0.0365 ng/mL ± 6.6%	

3M LIMS ID	Description	PFDA		PFUnA		PFDoA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-039	CGMN-GW-PW09-0-140425	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-040	CGMN-GW-PW09-DB-140425	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-041	CGMN-GW-PW09-FMS-140425	1.82	91.0	1.90	95.0	1.99	99.5
Average Concentration (ng/mL) ± %RPD		<0.0250 ng/mL		<0.0250 ng/mL		<0.0250 ng/mL	

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 (1) Samples were analyzed using internal standard calibration.

Table 22 continued. CGMN-GW-PW09-140425 ⁽¹⁾

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-039	CGMN-GW-PW09-0-140425	0.150	NA	0.0823	NA	1.62	NA
ISO11-01-03-14-040	CGMN-GW-PW09-DB-140425	0.145	NA	0.0852	NA	1.58	NA
ISO11-01-03-14-041	CGMN-GW-PW09-FMS-140425	1.79	82.1	1.87	89.3	3.44	99.5
Average Concentration (ng/mL) ± %RPD		0.148 ng/mL ± 3.4%		0.0838 ng/mL ± 3.5%		1.60 ng/mL ± 2.5%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₂ -PFUnA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-039	CGMN-GW-PW09-0-140425	93.0	95.4	96.2	96.6
ISO11-01-03-14-040	CGMN-GW-PW09-DB-140425	102	99.4	90.9	97.9
ISO11-01-03-14-041	CGMN-GW-PW09-FMS-140425	99.9	87.5	91.6	99.7
Average Recovery (%) ± %RSD		100% ± 2.0%	94.1% ± 6.4%	92.9% ± 3.1%	98.1% ± 1.6%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

Table 23. CGMN-GW-PW10-140425 ⁽¹⁾

3M LIMS ID	Description	PFBA		PFPeA		PFHxA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-042	CGMN-GW-PW10-0-140425	4.99	NA	0.250	NA	0.134	NA
ISO11-01-03-14-043	CGMN-GW-PW10-DB-140425	5.15	NA	0.254	NA	0.167	NA
ISO11-01-03-14-044	CGMN-GW-PW10-FMS-140425	7.16	NC	2.22	98.4	2.13	99.0
Average Concentration (ng/mL) ± %RPD		5.07 ng/mL ± 3.2%		0.252 ng/mL ± 1.6%		0.151 ng/mL ± 22% ⁽²⁾	

3M LIMS ID	Description	PFHpA		PFOA		PFNA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-042	CGMN-GW-PW10-0-140425	0.0420	NA	0.421	NA	<0.0250	NA
ISO11-01-03-14-043	CGMN-GW-PW10-DB-140425	0.0517	NA	0.420	NA	<0.0250	NA
ISO11-01-03-14-044	CGMN-GW-PW10-FMS-140425	1.92	93.7	2.32	98.9	1.88	94.0
Average Concentration (ng/mL) ± %RPD		0.0469 ng/mL ± 21% ⁽²⁾		0.421 ng/mL ± 0.24%		<0.0250 ng/mL	

3M LIMS ID	Description	PFDA		PFUnA		PFDoA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-042	CGMN-GW-PW10-0-140425	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-043	CGMN-GW-PW10-DB-140425	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-044	CGMN-GW-PW10-FMS-140425	1.83	91.5	1.93	96.5	1.97	98.5
Average Concentration (ng/mL) ± %RPD		<0.0250 ng/mL		<0.0250 ng/mL		<0.0250 ng/mL	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 23 continued. CGMN-GW-PW10-140425 ⁽¹⁾

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-042	CGMN-GW-PW10-0-140425	0.460	NA	0.105	NA	0.438	NA
ISO11-01-03-14-043	CGMN-GW-PW10-DB-140425	0.481	NA	0.115	NA	0.435	NA
ISO11-01-03-14-044	CGMN-GW-PW10-FMS-140425	2.11	82.0	1.92	90.5	2.31	101
Average Concentration (ng/mL) ± %RPD		0.471 ng/mL ± 4.5%		0.110 ng/mL ± 9.1%		0.437 ng/mL ± 0.69%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₂ -PFUnA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-042	CGMN-GW-PW10-0-140425	99.2	87.6	95.9	94.1
ISO11-01-03-14-043	CGMN-GW-PW10-DB-140425	97.7	101	99.8	88.4
ISO11-01-03-14-044	CGMN-GW-PW10-FMS-140425	98.5	95.5	97.6	93.4
Average Recovery (%) ± %RSD		98.5% ± 0.76%	94.7% ± 7.1%	97.8% ± 2.0%	92.0% ± 3.4%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Samples were analyzed using internal standard calibration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 24. CGMN-GW-TRIP-140423 ⁽¹⁾

3M LIMS ID	Description	PFBA		PFPeA		PFHxA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-046	CGMN-GW-TRIP-0-140423	<0.0500	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-047	CGMN-GW-TRIP-LS-140423	2.00	100	1.86	93.0	1.82	91.0
ISO11-01-03-14-048	CGMN-GW-TRIP-MS-140423	5.19	51.9 ⁽²⁾	4.96	49.6 ⁽²⁾	4.79	47.9 ⁽²⁾
ISO11-01-03-14-049	CGMN-GW-TRIP-HS-140423	99.0	99.0	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾

3M LIMS ID	Description	PFHpA		PFOA		PFNA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-046	CGMN-GW-TRIP-0-140423	<0.0250	NA	<0.0480	NA	<0.0250	NA
ISO11-01-03-14-047	CGMN-GW-TRIP-LS-140423	1.94	97.0	1.79	93.2	1.81	90.5
ISO11-01-03-14-048	CGMN-GW-TRIP-MS-140423	4.88	48.8 ⁽²⁾	4.91	51.3 ⁽²⁾	4.69	46.9 ⁽²⁾
ISO11-01-03-14-049	CGMN-GW-TRIP-HS-140423	NA ⁽³⁾	NA ⁽³⁾	85.4	86.4	NA ⁽³⁾	NA ⁽³⁾

3M LIMS ID	Description	PFDA		PFUnA		PFDoA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-046	CGMN-GW-TRIP-0-140423	<0.0250	NA	<0.0250	NA	<0.0250	NA
ISO11-01-03-14-047	CGMN-GW-TRIP-LS-140423	1.82	91.0	1.88	94.0	1.89	94.5
ISO11-01-03-14-048	CGMN-GW-TRIP-MS-140423	4.74	47.4 ⁽²⁾	4.99	49.9 ⁽²⁾	4.93	49.3 ⁽²⁾
ISO11-01-03-14-049	CGMN-GW-TRIP-HS-140423	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾	NA ⁽³⁾

NA = Not Applicable

(1) Samples were analyzed using internal standard calibration.

(2) Field matrix spike recovery did not meet acceptance criteria of 100 ± 30%.

(3) The spike level did not contain the target analyte.

Table 24 continued. CGMN-GW-TRIP-140423 ⁽¹⁾

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-14-046	CGMN-GW-TRIP-0-140423	<0.0250	NA	<0.0250	NA	<0.185	NA
ISO11-01-03-14-047	CGMN-GW-TRIP-LS-140423	1.67	83.5	1.78	89.0	1.77	95.7
ISO11-01-03-14-048	CGMN-GW-TRIP-MS-140423	4.19	41.9 ⁽²⁾	4.63	46.3 ⁽²⁾	4.50	48.5 ⁽²⁾
ISO11-01-03-14-049	CGMN-GW-TRIP-HS-140423	71.8	72.5	83.9	84.7	102	103

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₂ -PFOA	¹³ C ₂ -PFUnA	¹³ C ₁ -PFOS
		%Recovery	%Recovery	%Recovery	%Recovery
ISO11-01-03-14-046	CGMN-GW-TRIP-0-140423	100	104	97.2	99.1
ISO11-01-03-14-047	CGMN-GW-TRIP-LS-140423	97.3	90.5	95.2	99.7
ISO11-01-03-14-048	CGMN-GW-TRIP-MS-140423	104	109	100	99.0
ISO11-01-03-14-049	CGMN-GW-TRIP-HS-140423	98.3	103	97.0	89.2

NA = Not Applicable

(1) Samples were analyzed using internal standard calibration.

(2) Field matrix spike recovery did not meet acceptance criteria of 100 ± 30%.

(3) The spike level did not contain the target analyte.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Table 1 and 10-24 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Attachments

Attachment A: Select target analyte historical trend data for select Cottage Grove locations.

8 Signatures



Digitally signed by Susan T. Wolf
DN: c=US, st=MN, l=St. Paul, ou=3M
Environmental Laboratory - authenticated
by LRA, email=stwolf@mmm.com, o=3M,
cn=Susan T. Wolf
Reason: I have reviewed this document
Date: 2014.05.27 12:17:23 -05'00'

Susan T. Wolf, 3M Principal Analytical Investigator



Digitally signed by William K. Reagen
DN: c=US, st=MN, l=St. Paul,
ou=Laboratory Director, ou=3M
Environmental Laboratory - authenticated
by LRA, email=wkreagen@mmm.com,
o=3M, cn=William K. Reagen
Reason: I am approving this document
Date: 2014.05.27 12:57:44 -05'00'

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.



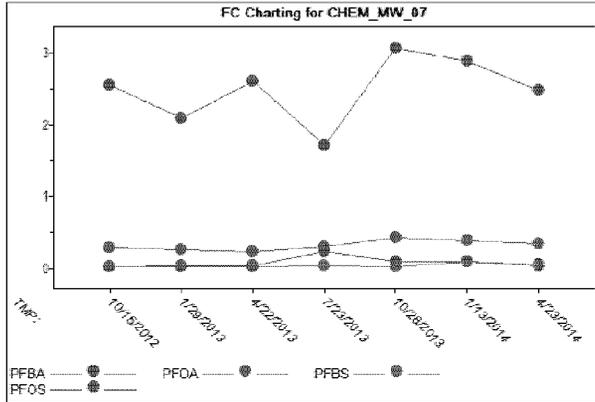
Digitally signed by Darcy K. Peterson
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory -
authenticated by LRA, email=dpeterson@mmm.com, o=3M,
cn=Darcy K. Peterson
Reason: I have reviewed this document
Date: 2014.05.28 07:45:21 -05'00'

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M Environmental Laboratory.

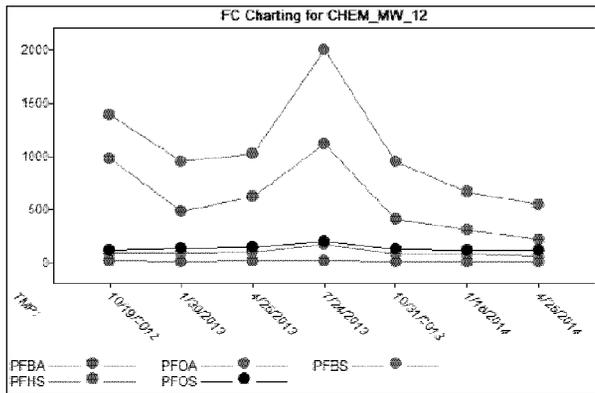
Attachment A: Select Target Analyte Historical Trend Data; units of ng/mL

MW07



MW07	10/15/2012	1/29/2013	4/22/2013	7/23/2013	10/28/2013	1/13/2014	4/23/2014
PFBA	2.55	2.09	2.61	1.71	3.07	2.88	2.48
PFOA	0.298	0.259	0.240	0.303	0.428	0.390	0.344
PFBS	<0.0250	<0.0250	0.0268	0.0396	0.0291	<0.100	0.0360
PFOS	0.0257	0.0439	0.0459	0.241	0.104	0.104	0.0493

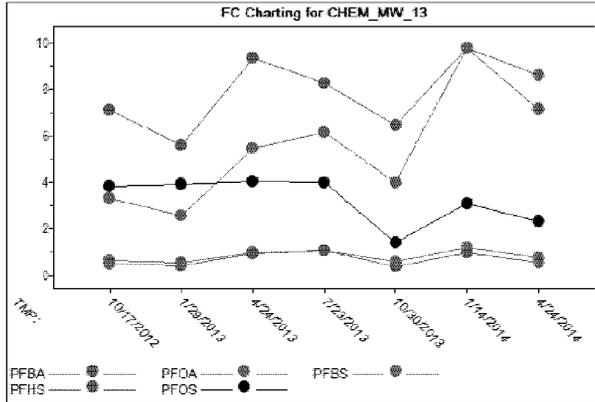
MW12



MW12	10/19/2012	1/30/2013	4/25/2013	7/24/2013	10/31/2013	1/16/2014	4/25/2014
PFBA	975	484	622	1110	408	312	220
PFOA	1390	946	1020	2000	954	668	548
PFBS	94.3	91.4	105	175	87.7	82.1	68.9
PFHS	20.9	13.7	16.4	23.9	13	13.1	9.82
PFOS	122	143	145	204	131	121	117

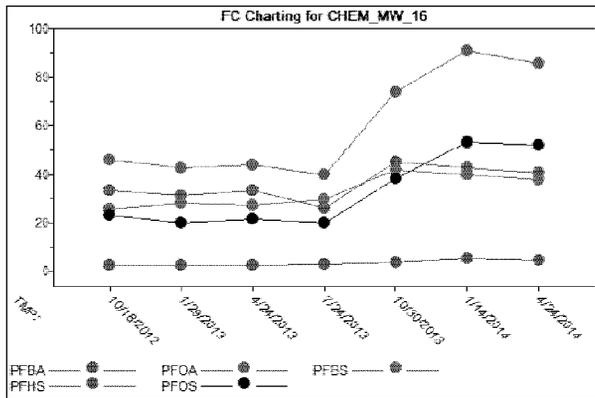
Attachment A continued: Select Target Analyte Historical Trend Data; units of ng/mL

MW13



MW13	10/17/2012	1/29/2013	4/24/2013	7/23/2013	10/30/2013	1/14/2014	4/24/2014
PFBA	7.10	5.60	9.34	8.30	6.48	9.80	7.14
PFOA	3.33	2.56	5.46	6.17	4.01	9.78	8.63
PFBS	0.619	0.560	0.972	1.07	0.571	1.20	0.752
PFHS	0.487	0.396	0.931	1.05	0.366	0.969	0.556
PFOS	3.85	3.92	4.05	4.02	1.40	3.08	2.33

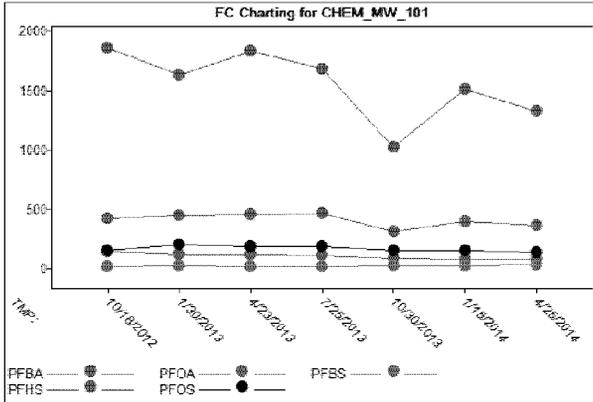
MW16



MW16	10/18/2012	1/29/2013	4/24/2013	7/24/2013	10/30/2013	1/14/2014	4/24/2014
PFBA	33.6	31.5	33.2	26.3	45.0	42.9	40.7
PFOA	45.9	42.6	43.8	39.9	73.9	91.0	85.7
PFBS	25.7	28.3	27.5	29.6	42.1	40.0	37.9
PFHS	2.71	2.54	2.74	2.83	3.95	5.52	4.49
PFOS	23.1	20.2	21.8	20.1	38.2	53.1	51.9

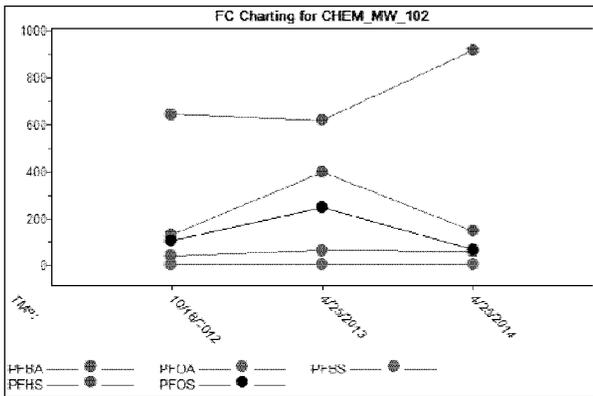
Attachment A continued: Select Target Analyte Historical Trend Data; units of ng/mL

MW101



MW101	10/18/2012	1/30/2013	4/23/2013	7/25/2013	10/30/2013	1/15/2014	4/25/2014
PFBA	1860	1630	1830	1680	1020	1510	1330
PFOA	147	124	121	112	86.7	79.4	76.0
PFBS	23.9	25.2	19.5	24.0	25.5	25.4	37.8
PFHS	427	455	459	464	314	398	364
PFOS	154	206	188	189	158	159	135

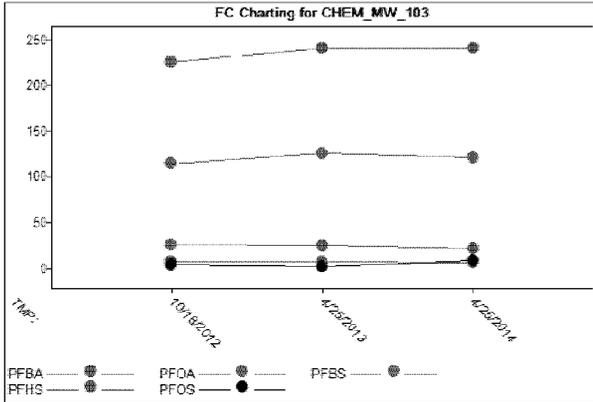
MW102



MW102	10/18/2012	4/25/2013	4/25/2014
PFBA	648	621	919
PFOA	42.2	62.0	58.1
PFBS	5.52	4.57	7.12
PFHS	129	401	147
PFOS	107	250	67.4

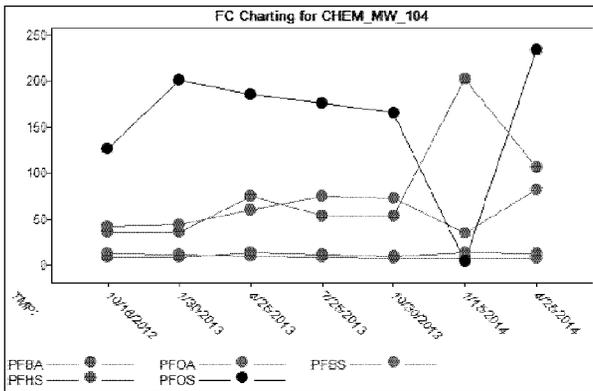
Attachment A continued: Select Target Analyte Historical Trend Data; units of ng/mL

MW103



MW103	10/18/2012	4/25/2013	4/25/2014
PFBA	226	241	241
PFOA	115	126	121
PFBS	7.16	7.01	6.51
PFHS	26.2	24.7	21.8
PFOS	4.02	1.55	7.82

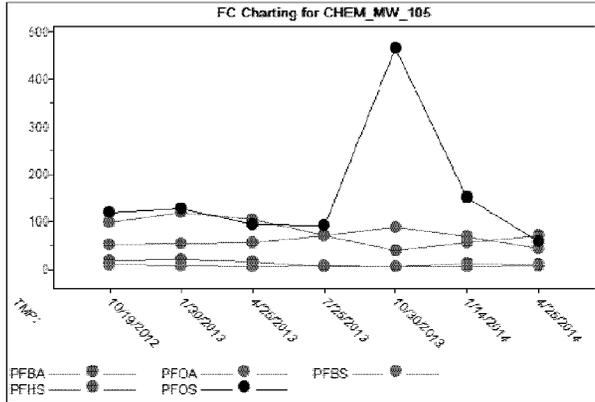
MW104



MW104	10/18/2012	1/30/2013	4/25/2013	7/25/2013	10/30/2013	1/15/2014	4/25/2014
PFBA	36.0	36.3	74.7	54.3	53.6	202	107
PFOA	42.0	44.8	60.7	74.7	72.5	35.1	82.1
PFBS	12.6	11.4	9.75	8.16	7.95	7.50	8.10
PFHS	8.69	9.07	13.4	11.8	10.1	14.2	12.6
PFOS	127	201	185	176	165	4.25	234

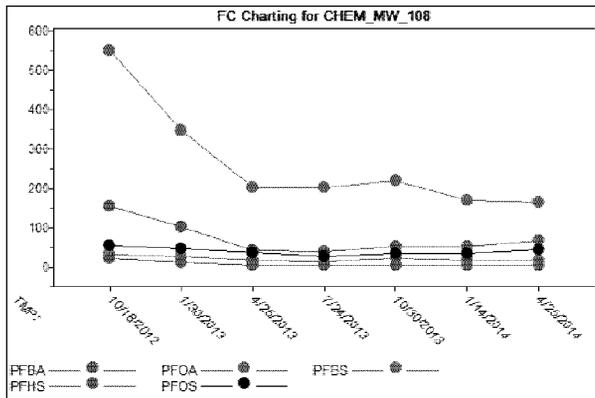
Attachment A continued: Select Target Analyte Historical Trend Data; units of ng/mL

MW105



MW105	10/19/2012	1/30/2013	4/25/2013	7/25/2013	10/30/2013	1/14/2014	4/25/2014
PFBA	52.7	54.4	57.0	71.6	38.9	55.6	71.0
PFOA	98.3	119	104	70.6	87.2	68.3	44.3
PFBS	9.46	8.03	5.72	6.49	4.98	6.15	7.68
PFHS	18.9	21.1	16.0	6.35	6.01	12.6	8.59
PFOS	120	129	95.0	92.3	467	152	58.7

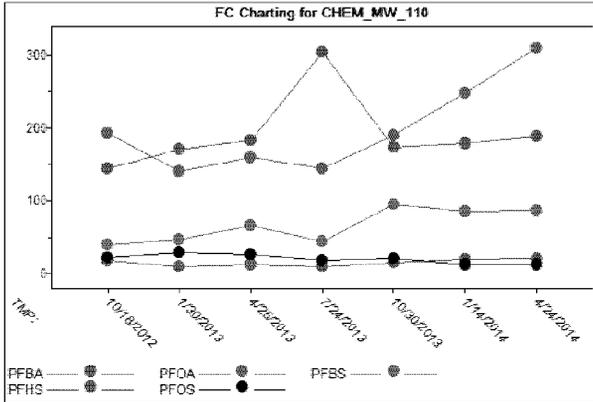
MW108



MW108	10/18/2012	1/30/2013	4/25/2013	7/24/2013	10/30/2013	1/14/2014	4/25/2014
PFBA	155	101	42.5	38.0	51.7	51.6	65.4
PFOA	550	348	201	202	218	169	164
PFBS	30.5	26.5	16.1	14.6	21.6	17.0	17.4
PFHS	20.7	11.1	4.95	4.94	4.47	4.13	3.58
PFOS	55.2	45.5	37.3	26.8	33.2	33.5	43.3

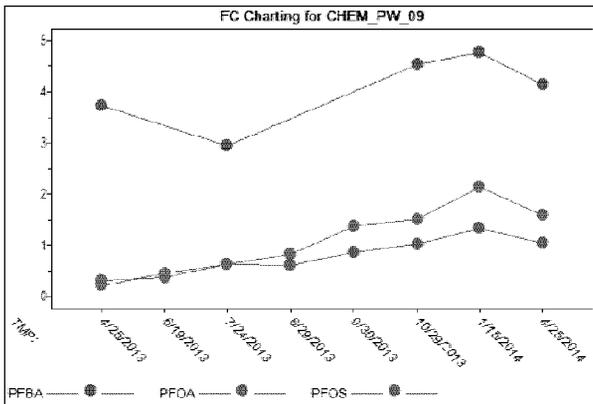
Attachment A continued: Select Target Analyte Historical Trend Data; units of ng/mL

MW110



MW110	10/18/2012	1/30/2013	4/25/2013	7/24/2013	10/30/2013	1/14/2014	4/24/2014
PFBA	144	171	183	304	174	179	189
PFOA	193	141	160	144	190	248	310
PFBS	39.6	46.6	66.7	43.8	94.8	85.1	86.1
PFHS	17.6	10.3	11.8	9.41	15.3	19.4	20.9
PFOS	21.6	28.6	26.4	17.8	21.1	12.1	12.5

PW09

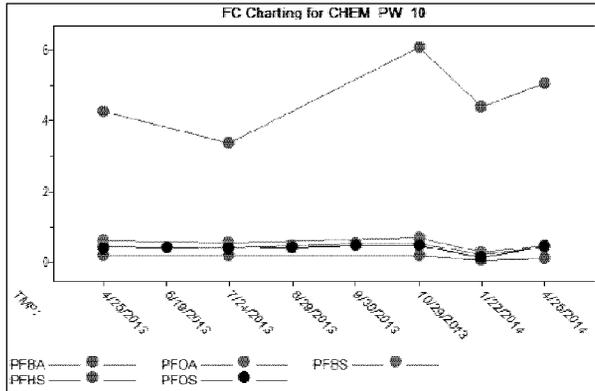


PW09	4/25/2013	6/19/2013	7/24/2013	8/29/2013	9/30/2013	10/29/2013	1/15/2014	4/25/2014
PFBA	3.72	NA	2.95	NA	NA	4.53	4.76	4.14
PFOA	0.223	0.462	0.624	0.605	0.873	1.02	1.33	1.05
PFOS	0.324	0.376	0.622	0.818	1.38	1.52	2.13	1.60

NA=Not applicable; analyte was not requested for this sampling event.

Attachment A continued: Select Target Analyte Historical Trend Data; units of ng/mL

PW10



PW10	4/25/2013	6/19/2013	7/24/2013	8/29/2013	9/30/2013	10/29/2013	1/22/2014	4/25/2014
PFBA	4.25	NA	3.37	NA	NA	6.09	4.40	5.07
PFOA	0.428	0.415	0.420	0.471	0.528	0.524	0.212	0.421
PFBS	0.594	NA	0.540	NA	NA	0.703	0.261	0.471
PFHS	0.197	NA	0.185	NA	NA	0.193	0.0485	0.110
PFOS	0.396	0.401	0.38	0.415	0.465	0.458	0.124	0.437

NA=Not applicable; analyte was not requested for this sampling event.

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-14

Requester: Kotsmith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 4/14/2014
Project Description: Cottage Grove GW Sampling; 2nd Quarter 2014
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-14-001	CGMN-GW-GW-MW07-0- 14 04 23	4/23/14 1425	GW	
ISO11-01-03-14-002	CGMN-GW-GW-MW07-DB- 14 04 23	4/23/14 1425		
ISO11-01-03-14-003	CGMN-GW-GW-MW07-FMS- 14 04 23	4/23/14 1425		
ISO11-01-03-14-004	CGMN-GW-MW12-0- 14 04 25	4/25/14 1530		
ISO11-01-03-14-005	CGMN-GW-MW12-DB- ↓	↓ 1530		
ISO11-01-03-14-006	CGMN-GW-MW12-LS- ↓	↓ 1530		
ISO11-01-03-14-007	CGMN-GW-MW12-HS- 14 04 25	4/25/14 1530		
ISO11-01-03-14-008	CGMN-GW-MW13-0- 14 04 24	4/24/14 1215		
ISO11-01-03-14-009	CGMN-GW-MW13-DB- ↓	4/24/14 1215		
ISO11-01-03-14-010	CGMN-GW-MW13-FMS- 14 04 24	4/24/14 1215		
ISO11-01-03-14-011	CGMN-GW-MW14R-0- 14 04 23	4/23/14 1505		
ISO11-01-03-14-012	CGMN-GW-MW14R-DB- ↓	4/23/14 1505		
ISO11-01-03-14-013	CGMN-GW-MW14R-LS- ↓	4/23/14 1505	↓	
ISO11-01-03-14-014	CGMN-GW-MW14R-HS- 14 04 23	4/23/14 1505	GW	

Sample Condition Upon Receipt: Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): JOHN HUNTER Collector's signature: 

Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-14 (cont.)

Requester: Kotsmith, James Ronald (MAPLEWC)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 4/14/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Project Description: Cottage Grove GW Sampling; 2nd Quarter 2014

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-14-015	CGMN-GW-MW16-0- 140424	4/24/14 1350	GW	
ISO11-01-03-14-016	CGMN-GW-MW16-DB-	4/24/14 1350		
ISO11-01-03-14-017	CGMN-GW-MW16-LS- ↓	4/24/14 1350		
ISO11-01-03-14-018	CGMN-GW-MW16-HS- 140424	4/24/14 1350		
ISO11-01-03-14-019	CGMN-GW-MW101-0- 140425	4/25/14 1245		
ISO11-01-03-14-020	CGMN-GW-MW101-DB-	1245		
ISO11-01-03-14-021	CGMN-GW-MW101-LS-	1245		
ISO11-01-03-14-022	CGMN-GW-MW101-HS-	1245		
ISO11-01-03-14-023	CGMN-GW-MW104-0-	1325		
ISO11-01-03-14-024	CGMN-GW-MW104-DB-	1325		
ISO11-01-03-14-025	CGMN-GW-MW104-LS-	1325		
ISO11-01-03-14-026	CGMN-GW-MW104-HS-	1325		
ISO11-01-03-14-027	CGMN-GW-MW105-0-	1435		
ISO11-01-03-14-028	CGMN-GW-MW105-DB-	1435		
ISO11-01-03-14-029	CGMN-GW-MW105-LS-	1435		
ISO11-01-03-14-030	CGMN-GW-MW105-HS- ✓	↓ 1435 ✓	✓	
ISO11-01-03-14-031	CGMN-GW-MW108-0- 140425	4/25/14 1515	GW	

Sample Condition Upon Receipt: Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): JOHN HUNTER Collector's signature: [Signature]

Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4887

Project: ISO11-01-03-14 (cont.)

Requester: Kotsmith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 4/14/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mnn.com

Project Description: Cottage Grove GW Sampling; 2nd Quarter 2014

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-14-032	CGMN-GW-MW108-DB- 140425	4/23/14 1515	GW	
ISO11-01-03-14-033	CGMN-GW-MW108-LS- ↓	↓ 1515		
ISO11-01-03-14-034	CGMN-GW-MW108-HS- 140425	4/25/14 1515		
ISO11-01-03-14-035	CGMN-GW-MW110-0- 140424	4/24/14 1500		
ISO11-01-03-14-036	CGMN-GW-MW110-DB- ↓	↓ 1500		
ISO11-01-03-14-037	CGMN-GW-MW110-LS- ↓	↓ 1500		
ISO11-01-03-14-038	CGMN-GW-MW110-HS- 140424	4/24/14 1500		
ISO11-01-03-14-039	CGMN-GW-PW09-0- 140425	4/25/14 1000		
ISO11-01-03-14-040	CGMN-GW-PW09-DB-		1000	
ISO11-01-03-14-041	CGMN-GW-PW09-FMS-		1000	
ISO11-01-03-14-042	CGMN-GW-PW10-0-		0945	
ISO11-01-03-14-043	CGMN-GW-PW10-DB- ↓		0945	
ISO11-01-03-14-044	CGMN-GW-PW10-FMS- 140425	↓	0945	
ISO11-01-03-14-045	CGMN-GW-GW-MW108-RB- 140425	4/25/14 1110		
ISO11-01-03-14-046	CGMN-GW-TRIP-0- 140423	4/23/14 1300		
ISO11-01-03-14-047	CGMN-GW-TRIP-LS- 140423	4/23/14 1300	✓	
ISO11-01-03-14-048	CGMN-GW-TRIP-MS- 140423	4/23/14 1300	GW	

Sample Condition Upon Receipt: Acceptable All items accounted for
 Temperature: _____ Deg C Received on Ice Other:

Collected by (print): JOHN HUNTER Collector's signature: [Signature]

Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-14 (cont.)

Requester: Koismith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 4/14/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Project Description: Cottage Grove GW Sampling; 2nd Quarter 2014

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-14-049	CGMN-GW-TRIP-HS- 140423	4/23/14 1100	GW	
ISO11-01-03-14-050	CGMN-GW-MW102-0- 140425	4/25/14 1200		
ISO11-01-03-14-051	CGMN-GW-MW102-DB-	1200		
ISO11-01-03-14-052	CGMN-GW-MW102-LS-	1200		
ISO11-01-03-14-053	CGMN-GW-MW102-HS-	1200		
ISO11-01-03-14-054	CGMN-GW-MW103-0-	1345		
ISO11-01-03-14-055	CGMN-GW-MW103-DB-	1345		
ISO11-01-03-14-056	CGMN-GW-MW103-LS- ↓	↓ 1345	✓	
ISO11-01-03-14-057	CGMN-GW-MW103-HS- 140425	4/25/14 1345	GW	

Sample Condition Upon Receipt: Acceptable All items accounted for
Temperature: _____ Deg C: Received on Ice Other:

Collected by (print): JOHN HUNTER Collector's signature: [Signature]

Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time



JULY 2014

Final Report

Analysis of PFBA, PFOA, PFBS, PFHS, and PFOS in Aqueous Samples, Cottage Grove Groundwater Sampling 3rd Quarter 2014

Laboratory Request Number: ISO11-01-03-16

Report Date – Date of Last Signature

Testing Laboratory

3M EHS&S Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Gary Hohenstein
3M EHS&S Operations
3M Building 224-5W-03
Saint Paul, MN 55144-1000
Phone: (651) 737-3570



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.

3M Principal Analytical Investigator: Susan Wolf

Report Author: Chelsie Grochow

Analytical Report ISO11-01-03-16

Analysis of PFBA, PFOA, PFBS, PFHS, and PFOS in Aqueous Samples,
Cottage Grove Groundwater Sampling – 3rd Quarter 2014

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater samples collected by Weston Solutions personnel at the 3M Cottage Grove facility. Samples were collected on July 15-17, 2014. Samples were returned to the 3M Environmental Laboratory on July 18, 2014 on ice for the analysis of Perfluorobutanoic acid (PFBA), Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS) and Perfluorooctane sulfonate (PFOS) under laboratory project number ISO11-01-03-16.

The 3M Environmental Laboratory prepared sample containers for twelve sampling locations. Each sample set consisted of a field sample, field sample duplicate, and a target analyte field matrix spike. Each empty container was marked with a “fill to here” line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. Select sample bottles were fortified with internal standards and surrogate recovery standards prior to being sent to the field for sample collection.

Samples were prepared and analyzed for PFBA, PFOA, PFBS, PFHS, and PFOS using method ETS-8-044.1 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”. Internal standards were used to aid in the data quality objectives for the analysis of select samples, were applicable.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 “General Requirements for the Competence of Testing and Calibration Laboratories”, in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory’s quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
ISO11-01-03-16-001	CGMN-MW07-0-140716	3.16	0.606	0.0759	0.0434	0.237
ISO11-01-03-16-002	CGMN-MW07-DB-140716	2.93	0.598	0.0765	0.0398	0.236
Average		3.05	0.602	0.0762	0.0416	0.237
%RPD Sample/Sample Dup		7.6	1.3	0.79	8.7	0.42
ISO11-01-03-16-004	CGMN-MW12-0-140717	157	395	49.3	7.87	94.2
ISO11-01-03-16-005	CGMN-MW12-DB-140717	150	409	51.4	8.34	99.5
Average		154⁽²⁾	402⁽²⁾	50.4⁽²⁾	8.11⁽²⁾	96.9⁽²⁾
%RPD Sample/Sample Dup		4.6	3.5	4.2	5.8	5.5
ISO11-01-03-16-007	CGMN-MW13-0-140716	7.86	13.9	0.970	0.700	4.37
ISO11-01-03-16-008	CGMN-MW13-DB-140716	8.29	14.8	0.975	0.726	4.58
Average		8.08	14.4	0.973	0.713	4.48
%RPD Sample/Sample Dup		5.3	6.3	0.51	3.6	4.7
ISO11-01-03-16-010	CGMN-MW14R-0-140716	685	430	29.8	20.7 ⁽²⁾	155 ⁽²⁾
ISO11-01-03-16-011	CGMN-MW14R-DB-140716	657	421	29.3	19.4 ⁽²⁾	159 ⁽²⁾
Average		671⁽²⁾	426⁽²⁾	29.6⁽²⁾	20.1⁽²⁾	158⁽²⁾
%RPD Sample/Sample Dup		4.2	2.1	1.7	6.5	1.9
ISO11-01-03-16-013	CGMN-MW16-0-140716	46.7	99.1	37.4	5.39	57.8
ISO11-01-03-16-014	CGMN-MW16-DB-140716	44.7	97.4	38.3	5.52	56.1
Average		45.7	98.3	37.9	5.46	57.0
%RPD Sample/Sample Dup		4.4	1.7	2.4	2.4	3.0
ISO11-01-03-16-016	CGMN-MW101-0-140717	1460	83.5	29.5	483	191
ISO11-01-03-16-017	CGMN-MW101-DB-140717	1450	84.2	29.0	477	194
Average		1460⁽²⁾	83.9⁽²⁾	29.3⁽²⁾	480⁽²⁾	193⁽²⁾
%RPD Sample/Sample Dup		0.69	0.83	1.7	1.3	1.6
ISO11-01-03-16-019	CGMN-MW104-0-140717	47.4	74.4	8.62	8.90	228
ISO11-01-03-16-020	CGMN-MW104-DB-140717	48.4	78.5	8.74	8.99	238
Average		47.9⁽²⁾	76.5⁽²⁾	8.68⁽²⁾	8.95⁽²⁾	233⁽²⁾
%RPD Sample/Sample Dup		2.1	5.4	1.4	1.0	4.3
ISO11-01-03-16-022	CGMN-MW105-0-140717	40.9	27.3	4.87	6.34	37.5
ISO11-01-03-16-023	CGMN-MW105-DB-140717	37.4	28.0	4.91	6.96	34.9
Average		39.2⁽²⁾	27.7⁽²⁾	4.89⁽²⁾	6.65⁽²⁾	36.2⁽²⁾
%RPD Sample/Sample Dup		8.9	2.5	0.82	9.3	7.2
ISO11-01-03-16-025	CGMN-MW108-0-140717	224	526	60.5	11.4	58.2
ISO11-01-03-16-026	CGMN-MW108-DB-140717	219	518	60.9	11.1	57.7
Average		222⁽²⁾	522⁽²⁾	60.7⁽²⁾	11.3⁽²⁾	58.0⁽²⁾
%RPD Sample/Sample Dup		2.3	1.5	0.66	2.7	0.86

NA = Not Applicable

- (1) Sample set was analyzed by internal standard calibration, except where noted. The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFBA ± 17%, PFOA ± 15%, PFBS ± 18%, PFHS ± 17%, and PFOS ± 20%.
- (2) Sample set was analyzed by external standard calibration. The analytical method uncertainties associated with the reported results by external standard calibration are as follows: PFBA ± 18%, PFOA ± 13%, PFBS ± 15%, PFHS ± 13%, and PFOS ± 11%.
- (3) The sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
ISO11-01-03-16-028	CGMN-MW110-0-140716	187	344	81.1	21.4	16.4
ISO11-01-03-16-029	CGMN-MW110-DB-140716	183	435	83.4	26.7	14.4
Average		185 ⁽²⁾	390 ⁽²⁾	82.3 ⁽²⁾	24.1 ⁽²⁾	15.4 ⁽²⁾
%RPD Sample/Sample Dup		2.2	23 ⁽³⁾	2.8	22 ⁽³⁾	13
ISO11-01-03-16-031	CGMN-PW09-0-140717	4.32	1.17	0.152	0.0810	1.79
ISO11-01-03-16-032	CGMN-PW09-DB-140717	4.32	1.15	0.158	0.0806	1.78
Average		4.32	1.16	0.155	0.0808	1.79
%RPD Sample/Sample Dup		0.0	1.7	3.9	0.50	0.56
ISO11-01-03-16-034	CGMN-PW10-0-140717	4.79	0.479	0.522	0.101	0.498
ISO11-01-03-16-035	CGMN-PW10-DB-140717	5.01	0.489	0.558	0.106	0.507
Average		4.90	0.484	0.540	0.104	0.503
%RPD Sample/Sample Dup		4.5	2.1	6.7	4.8	1.8

NA = Not Applicable

- (1) Sample set was analyzed by internal standard calibration, except where noted. The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFBA ± 17%, PFOA ± 15%, PFBS ± 18%, PFHS ± 17%, and PFOS ± 20%.
- (2) Sample set was analyzed by external standard calibration. The analytical method uncertainties associated with the reported results by external standard calibration are as follows: PFBA ± 18%, PFOA ± 13%, PFBS ± 15%, PFHS ± 13%, and PFOS ± 11%.
- (3) The sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorobutanoic Acid (C4 Acid)	PFBA	Linear
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched

2.2 Sample Collection

Samples were collected on July 15-17, 2014 in Nalgene™ (high-density polyethylene) bottles prepared at the 3M Environmental Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. Collected sample bottles were returned to the laboratory on ice on July 18, 2014.

2.3 Sample Preparation

Sample concentrations were expected to range from <0.025 ng/mL to >1000 ng/mL. Sampling locations that were expected to have concentration <100 ng/mL were analyzed by internal standard calibration analysis. Sampling locations that were expected to have concentration >100 ng/mL were analyzed by external standard calibration analysis. The following sample preparation procedures were followed for each type of analysis.

Internal standard calibration analysis: Samples analyzed by internal standard calibration were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2).

During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples.

External standard calibration analysis: Samples analyzed by external standard calibration required dilution prior to analysis. Samples were prepared by diluting 0.1 mL of a well-mixed sample with 9.8 mL of methanol (dilution factor of 100). An aliquot of surrogate spiking solution was added to the diluted samples at a nominal concentration of 1 ng/mL.

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk
Liquid Chromatograph	Agilent 1260
Analysis Method	ETS-8-044.1
Analysis Date	7/22/14, 7/23/14, 7/28/14
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 μ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 μ
Injection Volume	5 μL
Mass Spectrometer	AB Sciex TripleQuad 5500
Ion Source	Turbo Spray
Polarity	Negative
Software	Analyst 1.6.1

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.1 Analysis				
Step Number	Total Time (min)	Flow Rate (μL/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	750	90.0	10.0
1	0.50	750	90.0	10.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	90.0	10.0
9	19.0	750	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard ⁽¹⁾	Mass Transition Q1/Q3
PFBA	213/169	[¹³ C ₄]-PFBA	217/172
PFOA	413/369	[¹³ C ₈]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/80	[¹⁸ O ₂]-PFBS	303/84
	299/99		
PFHS	399/99	[¹³ C ₃]-PFHS	402/80
	399/80		
PFOS	499/99	[¹³ C ₈]-PFOS	507/80
	499/80		
	499/130		
[¹³ C ₃]-PFBA	216/172	[¹³ C ₄]-PFBA	217/172
[¹³ C ₄]-PFOA	417/372	[¹³ C ₈]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₈]-PFOS	507/80

Dwell time was 20 msec for each transition. The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standard was not used for the samples analyzed by solvent dilution external standard calibration.

3 Data Analysis

3.1 Calibration

Solvent dilution analysis using internal standard calibration: Samples were analyzed for all analytes against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 methanol:laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. Calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed (0.0125 ng/mL to 10 ng/mL (nominal) for the SRSs). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

Solvent dilution analysis using external standard calibration: Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution into 50 mL of 90:10 methanol:laboratory Milli-Q™ water. Calibration standards ranging from 0.02 ng/mL to 25 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. Low or high points were disabled to meet method criteria. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

For both methods of analysis, each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area counts or peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.1 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the Table 6 below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 7/22/14 Analysis	LOQ, ng/mL ⁽²⁾ 7/23/14, 7/28/14 Analysis
PFBA	0.0500	5.00
PFOA	0.0240	1.92
PFBS	0.0250	2.00
PFHS	0.0250	2.00
PFOS	0.0232	1.85

(1) A dilution factor of 2 was applied to the LOQ.

(2) A dilution factor of 100 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±25%.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: method/solvent blanks, field/trip blanks, and sampling equipment blanks. Each blank result was reviewed and used to evaluate method performance. The method/solvent blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs prepared for internal standard calibration analysis were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. LCSs prepared for external standard calibration analysis were prepared by spiking known amounts of the analytes into 1.0 mL of laboratory reagent water and 9.0 mL of methanol to produce the desired concentration. Method ETS-8-044.1 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report.

The following calculations were used to generate data in Table 7.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%$$

$$\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%$$

Table 7. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 7/22/14	PFBA			PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140722-1	0.198	0.158	79.6	0.190	0.198	104
LCS-140722-2	0.198	0.160	80.8	0.190	0.180	94.8
LCS-140722-3	0.198	0.159	80.2	0.190	0.188	98.9
Average ± %RSD	80.2% ± 0.75%			99.2% ± 4.6%		
LCS-140722-4	19.8	19.1	96.7	19.0	17.6	92.9
LCS-140722-5	19.8	19.4	98.0	19.0	18.4	96.7
LCS-140722-6	19.8	20.6	104	19.0	18.2	95.6
Average ± %RSD	99.6% ± 3.9%			95.1% ± 2.1%		
LCS-140722-7	160	163	102	153	149	97.2
LCS-140722-8	160	162	101	153	144	94.4
LCS-140722-9	160	154	96.4	153	145	94.6
Average ± %RSD	99.8% ± 3.0%			95.4% ± 1.6%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 7/22/14						
PFBS				PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140722-1	0.198	0.178	89.7	0.198	0.193	97.6
LCS-140722-2	0.198	0.171	86.4	0.198	0.199	100
LCS-140722-3	0.198	0.180	90.9	0.198	0.197	99.4
Average ± %RSD	89.0% ± 2.6%			99.0% ± 1.3%		
LCS-140722-4	19.8	17.1	86.6	19.8	18.6	94.0
LCS-140722-5	19.8	17.5	88.4	19.8	19.5	98.5
LCS-140722-6	19.8	18.2	92.0	19.8	20.2	102
Average ± %RSD	89.0% ± 3.1%			98.2% ± 4.1%		
LCS-140722-7	160	138	86.0	160	142	88.9
LCS-140722-8	160	129	80.4	160	141	88.1
LCS-140722-9	160	129	80.8	160	140	87.7
Average ± %RSD	82.4% ± 3.8%			88.2% ± 0.69%		

ETS-8-044.1 Internal standard calibration Analyzed 7/22/14			
PFOS (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140722-1	0.184	0.180	97.8
LCS-140722-2	0.184	0.171	92.8
LCS-140722-3	0.184	0.179	97.2
Average ± %RSD	95.9% ± 2.8%		
LCS-140722-4	18.4	17.2	93.3
LCS-140722-5	18.4	18.0	97.6
LCS-140722-6	18.4	17.9	97.2
Average ± %RSD	96.0% ± 2.5%		
LCS-140722-7	148	142	96.2
LCS-140722-8	148	137	92.3
LCS-140722-9	148	136	91.6
Average ± %RSD	93.4% ± 2.7%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 7/22/14						
¹³ C ₃ -PFBA				¹³ C ₄ -PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140722-1	0.197	0.195	98.9	0.198	0.212	107
LCS-140722-2	0.197	0.188	95.2	0.198	0.193	97.6
LCS-140722-3	0.197	0.185	94.1	0.198	0.208	105
Average ± %RSD	96.1% ± 2.6%			103% ± 4.8%		
LCS-140722-4	1.97	2.03	103	1.98	2.13	107
LCS-140722-5	1.97	1.91	96.9	1.98	2.01	102
LCS-140722-6	1.97	2.04	103	1.98	2.20	111
Average ± %RSD	101% ± 3.5%			107% ± 4.2%		

ETS-8-044.1 Internal standard calibration Analyzed 7/22/14			
¹³ C ₄ -PFOS			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140722-1	0.189	0.182	96.6
LCS-140722-2	0.189	0.185	97.8
LCS-140722-3	0.189	0.195	103
Average ± %RSD	99.1% ± 3.4%		
LCS-140722-4	1.89	1.89	100
LCS-140722-5	1.89	1.80	95.1
LCS-140722-6	1.89	1.94	103
Average ± %RSD	99.4% ± 4.0%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External standard calibration Analyzed 7/23/14						
PFBA				PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140723-1	0.199	0.196	98.7	0.191	0.204	107
LCS-140723-2	0.199	0.201	101	0.191	0.201	105
LCS-140723-3	0.199	0.179	89.7	0.191	0.197	103
Average ± %RSD	96.5% ± 6.2%			105% ± 1.9%		
LCS-140723-4	1.99	2.22	112	1.91	2.17	114
LCS-140723-5	1.99	2.18	110	1.91	2.09	109
LCS-140723-6	1.99	2.20	111	1.91	2.18	114
Average ± %RSD	111% ± 0.90%			112% ± 2.6%		
LCS-140723-7	20.0	22.0	110	19.1	21.9	115
LCS-140723-8	20.0	21.9	110	19.1	22.0	115
LCS-140723-9	20.0	22.2	111	19.1	21.6	113
Average ± %RSD	110% ± 0.52%			114% ± 1.0%		

ETS-8-044.1 External standard calibration Analyzed 7/23/14						
PFBS				PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140723-1	0.199	0.225	113	0.199	0.220	110
LCS-140723-2	0.199	0.221	111	0.199	0.229	115
LCS-140723-3	0.199	0.226	113	0.199	0.227	114
Average ± %RSD	112% ± 1.0%			113% ± 2.3%		
LCS-140723-4	1.99	2.32	117	1.99	2.28	115
LCS-140723-5	1.99	2.21	111	1.99	2.19	110
LCS-140723-6	1.99	2.28	115	1.99	2.29	115
Average ± %RSD	114% ± 2.7%			113% ± 2.5%		
LCS-140723-7	20.0	22.6	113	20.0	22.4	112
LCS-140723-8	20.0	22.4	112	20.0	21.8	109
LCS-140723-9	20.0	22.4	112	20.0	21.7	109
Average ± %RSD	112% ± 0.51%			110% ± 1.6%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External standard calibration Analyzed 7/23/14			
PFOS (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140723-1	0.185	0.199	107
LCS-140723-2	0.185	0.202	109
LCS-140723-3	0.185	0.197	107
Average ± %RSD	108% ± 1.1%		
LCS-140723-4	1.85	2.09	113
LCS-140723-5	1.85	2.05	111
LCS-140723-6	1.85	2.12	114
Average ± %RSD	113% ± 1.4%		
LCS-140723-7	18.5	21.3	115
LCS-140723-8	18.5	20.8	112
LCS-140723-9	18.5	20.7	112
Average ± %RSD	113% ± 1.5%		

ETS-8-044.1 Internal standard calibration Analyzed 7/23/14						
¹³ C ₃ -PFBA				¹³ C ₄ -PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140723-1	0.198	0.216	109	0.199	0.215	108
LCS-140723-2	0.198	0.214	108	0.199	0.212	107
LCS-140723-3	0.198	0.195	98.6	0.199	0.209	105
Average ± %RSD	105% ± 5.5%			107% ± 1.4%		
LCS-140723-4	1.98	2.22	112	1.99	2.23	112
LCS-140723-5	1.98	2.21	111	1.99	2.17	109
LCS-140723-6	1.98	2.21	112	1.99	2.20	110
Average ± %RSD	112% ± 0.52%			110% ± 1.4%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 7/23/14			
¹³C₄-PFOS			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140723-1	0.190	0.208	110
LCS-140723-2	0.190	0.202	107
LCS-140723-3	0.190	0.205	108
Average ± %RSD	108% ± 1.4%		
LCS-140723-4	1.90	2.11	111
LCS-140723-5	1.90	2.10	110
LCS-140723-6	1.90	2.08	110
Average ± %RSD	110% ± 0.52%		

ETS-8-044.1 External standard calibration Analyzed 7/28/14						
PFBA				PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140728-1	0.199	0.204	103	0.191	0.197	103
LCS-140728-2	0.199	0.176	88.6	0.191	0.196	103
LCS-140728-3	0.199	0.201	101	0.191	0.205	107
Average ± %RSD	97.5% ± 8.0%			104% ± 2.2%		
LCS-140728-4	1.99	2.23	112	1.91	2.01	105
LCS-140728-5	1.99	2.23	112	1.91	2.05	107
LCS-140728-6	1.99	2.20	111	1.91	2.04	107
Average ± %RSD	112% ± 0.52%			106% ± 1.1%		
LCS-140728-7	20.0	21.5	108	19.1	20.9	109
LCS-140728-8	20.0	21.1	105	19.1	20.0	105
LCS-140728-9	20.0	21.8	109	19.1	21.0	110
Average ± %RSD	107% ± 1.9%			108% ± 2.4%		

Table 7 continued. Laboratory Control Spike Results.

PFBS				PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140728-1	0.199	0.212	107	0.199	0.208	105
LCS-140728-2	0.199	0.223	112	0.199	0.222	111
LCS-140728-3	0.199	0.219	110	0.199	0.219	110
Average ± %RSD	110% ± 2.3%			109% ± 3.0%		
LCS-140728-4	1.99	2.17	109	1.99	2.16	109
LCS-140728-5	1.99	2.21	111	1.99	2.17	109
LCS-140728-6	1.99	2.15	108	1.99	2.14	108
Average ± %RSD	109% ± 1.4%			109% ± 0.53%		
LCS-140728-7	20.0	21.6	108	20.0	21.9	110
LCS-140728-8	20.0	20.7	103	20.0	21.2	106
LCS-140728-9	20.0	21.4	107	20.0	21.6	108
Average ± %RSD	106% ± 2.5%			108% ± 1.9%		

PFOS (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140728-1	0.185	0.191	103
LCS-140728-2	0.185	0.195	105
LCS-140728-3	0.185	0.197	106
Average ± %RSD	105% ± 1.5%		
LCS-140728-4	1.85	1.98	107
LCS-140728-5	1.85	2.00	108
LCS-140728-6	1.85	2.00	108
Average ± %RSD	108% ± 0.54%		
LCS-140728-7	18.5	19.9	108
LCS-140728-8	18.5	19.6	106
LCS-140728-9	18.5	20.7	112
Average ± %RSD	109% ± 2.8%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 7/28/14						
¹³ C ₃ -PFBA				¹³ C ₄ -PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140728-1	0.198	0.208	105	0.199	0.208	104
LCS-140728-2	0.198	0.196	98.9	0.199	0.211	106
LCS-140728-3	0.198	0.190	95.8	0.199	0.201	101
Average ± %RSD	99.9% ± 4.7%			104% ± 2.4%		
LCS-140728-4	1.98	2.02	102	1.99	1.99	99.8
LCS-140728-5	1.98	2.11	107	1.99	2.14	107
LCS-140728-6	1.98	2.14	108	1.99	2.11	106
Average ± %RSD	106% ± 3.0%			104% ± 3.7%		

ETS-8-044.1 Internal standard calibration Analyzed 7/28/14			
¹³ C ₄ -PFOS			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-140728-1	0.190	0.203	107
LCS-140728-2	0.190	0.195	103
LCS-140728-3	0.190	0.194	102
Average ± %RSD	104% ± 2.5%		
LCS-140728-4	1.90	1.88	99.1
LCS-140728-5	1.90	2.05	108
LCS-140728-6	1.90	2.02	106
Average ± %RSD	104% ± 4.5%		

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.1, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFBA	Internal	8.56	±17%
PFOA	Internal	7.42	±15%
PFBS	Internal	9.14	±18%
PFHS	Internal	8.69	±17%
PFOS	Internal	9.93	±20%
PFBA	External	8.93	±18%
PFOA	External	6.59	±13%
PFBS	External	7.67	±15%
PFHS	External	6.37	±13%
PFOS	External	5.33	±11%

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample was collected at each sampling point to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, each sample contained stable isotope surrogate recovery spikes of ¹³C₃-PFBA, ¹³C₄-PFOA, and ¹³C₄-PFOS, which were added at a nominal concentration of 0.1 ng/mL to select sample bottles prior to sample collection or at a nominal concentration of 1 ng/mL following sample collection. The ¹³C₃-PFBA and ¹³C₄-PFOA were selected to represent perfluorocarboxylic acids. The ¹³C₄-labeled PFOS was selected to represent the perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

$$\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%$$

Table 9. Field Matrix Spike Concentrations

Location	Spike Level	Final Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
MW07, PW09, and PW10	FMS	2.00	1.98	1.98	1.98	1.98
MW13	FMS	5.00	4.94	4.95	4.95	4.95
MW110	FMS	20.0	19.8	19.8	19.8	19.8
MW16	FMS	50.0	49.4	49.5	49.5	49.5
MW101, MW104, MW105, and MW108	FMS	100	98.8	99.0	99.0	99.0
MW12 and MW14R	FMS	500	494	495	495	495
Trip Blank	Low	2.00	1.98	1.98	1.98	1.98
	High	100	98.8	99.0	99.0	99.0

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

These sampling locations have been analyzed repeatedly and the laboratory has historical field matrix spike data demonstrating that the method is applicable to the sample matrix. While the method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample, the target analytes for each sampling location were expected to cover a wide concentration range. Therefore, field matrix spike concentrations were selected based on the expected concentration of PFOA and/or PFOS. As a result, there are instances where the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For those analytes where the field matrix spike level was not appropriate as compared to the sample concentration, the surrogate recovery standards were used to assess method accuracy. All surrogate recovery standards and field matrix spike recoveries met method acceptance criteria (where applicable).

Table 10. CGMN GW MW07 140716

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-001	CGMN-MW07-0-140716	3.16	NA	0.606	NA
ISO11-01-03-16-002	CGMN-MW07-DB-140716	2.93	NA	0.598	NA
ISO11-01-03-16-003	CGMN-MW07-FMS-140716	5.16	106	2.69	106
Average Concentration (ng/mL) ± %RPD		3.05 ng/mL ± 7.6%		0.602 ng/mL ± 1.3%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-001	CGMN-MW07-0-140716	0.0759	NA	0.0434	NA	0.237	NA
ISO11-01-03-16-002	CGMN-MW07-DB-140716	0.0765	NA	0.0398	NA	0.236	NA
ISO11-01-03-16-003	CGMN-MW07-FMS-140716	2.09	102	2.14	106	2.44	111
Average Concentration (ng/mL) ± %RPD		0.0762 ng/mL ± 0.79%		0.0416 ng/mL ± 8.7%		0.237 ng/mL ± 0.42%	

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-001	CGMN-MW07-0-140716	123	120	110
ISO11-01-03-16-002	CGMN-MW07-DB-140716	123	112	127
ISO11-01-03-16-003	CGMN-MW07-FMS-140716	105	115	111
Average Recovery (%) ± %RSD		117% ± 9.0%	116% ± 3.5%	116% ± 8.1%

NA = Not Applicable
 Samples were diluted 1:1 and analyzed by internal standard calibration.

Table 11. CGMN GW MW12 140717

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-004	CGMN-MW12-0-140717	157	NA	395	NA
ISO11-01-03-16-005	CGMN-MW12-DB-140717	150	NA	409	NA
ISO11-01-03-16-006	CGMN-MW12-FMS-140717	669	103	1010	123
Average Concentration (ng/mL) ± %RPD		154 ng/mL ± 4.6%		402 ng/mL ± 3.5%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-004	CGMN-MW12-0-140717	49.3	NA	7.87	NA	94.2	NA
ISO11-01-03-16-005	CGMN-MW12-DB-140717	51.4	NA	8.34	NA	99.5	NA
ISO11-01-03-16-006	CGMN-MW12-FMS-140717	583	108	549	109 ⁽¹⁾	700	122
Average Concentration (ng/mL) ± %RPD		50.4 ng/mL ± 4.2%		8.11 ng/mL ± 5.8%		96.9 ng/mL ± 5.5%	

3M LIMS ID	Description	¹³ C ₁ -PFBA	¹³ C ₁ -PFOA	¹³ C ₁ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-004	CGMN-MW12-0-140717	101	99.7	101
ISO11-01-03-16-005	CGMN-MW12-DB-140717	105	107	106
ISO11-01-03-16-006	CGMN-MW12-FMS-140717	105	103	103
Average Recovery (%) ± %RSD		103% ± 2.2%	103% ± 3.6%	103% ± 2.2%

NA = Not Applicable

Samples were diluted 1:100 and analyzed by external standard calibration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 12. CGMN GW MW13 140716

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-007	CGMN-MW13-0-140716	7.86	NA	13.9	NA
ISO11-01-03-16-008	CGMN-MW13-DB-140716	8.29	NA	14.8	NA
ISO11-01-03-16-009	CGMN-MW13-FMS-140716	14.0	119	19.1	NC
Average Concentration (ng/mL) ± %RPD		8.08 ng/mL ± 5.3%		14.4 ng/mL ± 6.3%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-007	CGMN-MW13-0-140716	0.970	NA	0.700	NA	4.37	NA
ISO11-01-03-16-008	CGMN-MW13-DB-140716	0.975	NA	0.726	NA	4.58	NA
ISO11-01-03-16-009	CGMN-MW13-FMS-140716	6.06	103	6.26	112	9.62	104
Average Concentration (ng/mL) ± %RPD		0.973 ng/mL ± 0.51%		0.713 ng/mL ± 3.6%		4.48 ng/mL ± 4.7%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-007	CGMN-MW13-0-140716	117	115	104
ISO11-01-03-16-008	CGMN-MW13-DB-140716	116	117	111
ISO11-01-03-16-009	CGMN-MW13-FMS-140716	115	106	105
Average Recovery (%) ± %RSD		116% ± 0.87%	113% ± 5.2%	107% ± 3.5%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 and analyzed by internal standard calibration.

Table 13. CGMN GW MW14R 140716

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-0'0	CGMN-MW14R-0-140716	685	NA	430	NA
ISO11-01-03-16-0'1	CGMN-MW14R-DB-140716	657	NA	421	NA
ISO11-01-03-16-0'2	CGMN-MW14R-FMS-140716	1190	104	962	109
Average Concentration (ng/mL) ± %RPD		671 ng/mL ± 4.2%		426 ng/mL ± 2.1%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-0'0	CGMN-MW14R-0-140716	29.8	NA	20.7	NA	156	NA
ISO11-01-03-16-0'1	CGMN-MW14R-DB-140716	29.3	NA	19.4	NA	159	NA
ISO11-01-03-16-0'2	CGMN-MW14R-FMS-140716	549	105 ⁽¹⁾	535	104 ⁽¹⁾	666	103
Average Concentration (ng/mL) ± %RPD		29.6 ng/mL ± 1.7%		20.1 ng/mL ± 6.5%		158 ng/mL ± 1.9%	

3M LIMS ID	Description	¹² C ₂ -PFBA	¹² C ₄ -PFOA	¹² C ₂ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-0'0	CGMN-MW14R-0-140716	109	105	106
ISO11-01-03-16-0'1	CGMN-MW14R-DB-140716	102	103	100
ISO11-01-03-16-0'2	CGMN-MW14R-FMS-140716	104	101	103
Average Recovery (%) ± %RSD		105% ± 3.5%	103% ± 1.9%	103% ± 2.7%

NA = Not Applicable
 Samples were diluted 1:100 and analyzed by internal standard calibration.
 (1) FMS concentration greater than 10 times the sample concentration.

Table 14. CGMN GW MW16 140716

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-0'3	CGMN-MW16-0-140716	46.7	NA	99.1	NA
ISO11-01-03-16-0'4	CGMN-MW16-DB-140716	44.7	NA	97.4	NA
ISO11-01-03-16-015	CGMN-MW16-FMS-140716	103	115	161	127
Average Concentration (ng/mL) ± %RPD		45.7 ng/mL ± 4.4%		98.3 ng/mL ± 17%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-0'3	CGMN-MW16-0-140716	37.4	NA	5.39	NA	57.8	NA
ISO11-01-03-16-0'4	CGMN-MW16-DB-140716	38.3	NA	5.52	NA	56.1	NA
ISO11-01-03-16-015	CGMN-MW16-FMS-140716	92.8	111	63.2	117	119	125
Average Concentration (ng/mL) ± %RPD		37.9 ng/mL ± 2.4%		5.46 ng/mL ± 2.4%		57.0 ng/mL ± 3.0%	

3M LIMS ID	Description	¹² C ₂ -PFBA	¹² C ₄ -PFOA	¹² C ₂ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-0'3	CGMN-MW16-0-140716	106	110	115
ISO11-01-03-16-0'4	CGMN-MW16-DB-140716	94.8	116	109
ISO11-01-03-16-015	CGMN-MW16-FMS-140716	123	120	98.3
Average Recovery (%) ± %RSD		108% ± 13%	115% ± 4.4%	107% ± 7.9%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 and analyzed by external standard calibration.

Table 15. CGMN GW MW101 140717

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-0'6	CGMN-MW101-0-140717	1460	NA	83.5	NA
ISO11-01-03-16-0'7	CGMN-MW101-DB-140717	1450	NA	84.2	NA
ISO11-01-03-16-0'8	CGMN-MW101-FMS-140717	1610	NC	177	94.3
Average Concentration (ng/mL) ± %RPD		1460 ng/mL ± 0.69%		83.9 ng/mL ± 0.83%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-0'6	CGMN-MW101-0-140717	29.5	NA	483	NA	191	NA
ISO11-01-03-16-0'7	CGMN-MW101-DB-140717	29.0	NA	477	NA	194	NA
ISO11-01-03-16-0'8	CGMN-MW101-FMS-140717	128	99.7	574	NC	281	89.4
Average Concentration (ng/mL) ± %RPD		29.3 ng/mL ± 1.7%		480 ng/mL ± 1.3%		193 ng/mL ± 1.6%	

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₄ -PFOA	¹³ C ₆ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-0'6	CGMN-MW101-0-140717	105	104	106
ISO11-01-03-16-0'7	CGMN-MW101-DB-140717	106	103	105
ISO11-01-03-16-0'8	CGMN-MW101-FMS-140717	109	105	108
Average Recovery (%) ± %RSD		106% ± 2.0%	104% ± 0.96%	106% ± 1.5%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:100 and analyzed by external standard calibration.

Table 16. CGMN GW MW104 140717

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-0'9	CGMN-MW104-0-140717	47.4	NA	74.4	NA
ISO11-01-03-16-020	CGMN-MW104-DB-140717	48.4	NA	78.5	NA
ISO11-01-03-16-021	CGMN-MW104-FMS-140717	143	95.1	176	101
Average Concentration (ng/mL) ± %RPD		47.9 ng/mL ± 2.1%		76.5 ng/mL ± 5.4%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-0'9	CGMN-MW104-0-140717	8.62	NA	8.90	NA	228	NA
ISO11-01-03-16-020	CGMN-MW104-DB-140717	8.74	NA	8.99	NA	238	NA
ISO11-01-03-16-021	CGMN-MW104-FMS-140717	106	98.3 ⁽¹⁾	105	97.0 ⁽¹⁾	328	NC
Average Concentration (ng/mL) ± %RPD		8.68 ng/mL ± 1.4%		8.95 ng/mL ± 1.0%		233 ng/mL ± 4.3%	

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-0'9	CGMN-MW104-0-140717	104	101	104
ISO11-01-03-16-020	CGMN-MW104-DB-140717	107	104	104
ISO11-01-03-16-021	CGMN-MW104-FMS-140717	105	106	104
Average Recovery (%) ± %RSD		105% ± 1.5%	104% ± 2.4%	104% ± 0.35%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:100 and analyzed by external standard calibration.
 (1) FMS concentration greater than 10 times the sample concentration.

Table 17. CGMN GW MW105 140717

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-022	CGMN-MW105-0-140717	40.9	NA	27.3	NA
ISO11-01-03-16-023	CGMN-MW105-DB-140717	37.4	NA	28.0	NA
ISO11-01-03-16-024	CGMN-MW105-FMS-140717	141	102	128	102
Average Concentration (ng/mL) ± %RPD		39.2 ng/mL ± 8.9%		27.7 ng/mL ± 2.5%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-022	CGMN-MW105-0-140717	4.87	NA	6.34	NA	37.5	NA
ISO11-01-03-16-023	CGMN-MW105-DB-140717	4.91	NA	6.96	NA	34.9	NA
ISO11-01-03-16-024	CGMN-MW105-FMS-140717	106	102 ⁽¹⁾	103	97.3 ⁽¹⁾	137	102
Average Concentration (ng/mL) ± %RPD		4.89 ng/mL ± 0.82%		6.65 ng/mL ± 9.3%		36.2 ng/mL ± 7.2%	

3M LIMS ID	Description	¹³ C ₁ -PFBA	¹³ C ₁ -PFOA	¹³ C ₁ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-022	CGMN-MW105-0-140717	103	101	103
ISO11-01-03-16-023	CGMN-MW105-DB-140717	106	105	104
ISO11-01-03-16-024	CGMN-MW105-FMS-140717	108	107	108
Average Recovery (%) ± %RSD		105% ± 2.4%	105% ± 2.9%	105% ± 2.3%

NA = Not Applicable
 Samples were diluted 1:100 and analyzed by external standard calibration.
 (1) FMS concentration greater than 10 times the sample concentration.

Table 18. CGMN GW MW108 140717

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-025	CGMN-MW108-0-140717	224	NA	526	NA
ISO11-01-03-16-026	CGMN-MW108-DB-140717	219	NA	518	NA
ISO11-01-03-16-027	CGMN-MW108-FMS-140717	320	NC	631	NC
Average Concentration (ng/mL) ± %RPD		222 ng/mL ± 2.3%		522 ng/mL ± 1.5%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-025	CGMN-MW108-0-140717	60.5	NA	11.4	NA	58.2	NA
ISO11-01-03-16-026	CGMN-MW108-DB-140717	60.9	NA	11.1	NA	57.7	NA
ISO11-01-03-16-027	CGMN-MW108-FMS-140717	159	99.3	107	96.7	155	98.0
Average Concentration (ng/mL) ± %RPD		60.7 ng/mL ± 0.66%		11.3 ng/mL ± 2.7%		58.0 ng/mL ± 0.86%	

3M LIMS ID	Description	¹³ C ₁ -PFBA	¹³ C ₁ -PFOA	¹³ C ₁ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-025	CGMN-MW108-0-140717	112	107	105
ISO11-01-03-16-026	CGMN-MW108-DB-140717	104	103	103
ISO11-01-03-16-027	CGMN-MW108-FMS-140717	106	107	104
Average Recovery (%) ± %RSD		107% ± 3.9%	106% ± 2.2%	104% ± 0.90%

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
Samples were diluted 1:100 and analyzed by external standard calibration.

Table 19. CGMN GW MW110 140716

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-028	CGMN-MW110-0-140716	187	NA	344	NA
ISO11-01-03-16-029	CGMN-MW110-DB-140716	183	NA	435	NA
ISO11-01-03-16-030	CGMN-MW110-FMS-140716	206	NC	390	NC
Average Concentration (ng/mL) ± %RPD		185 ng/mL ± 2.2%		390 ng/mL ± 23% ⁽¹⁾	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-028	CGMN-MW110-0-140716	81.1	NA	21.4	NA	16.4	NA
ISO11-01-03-16-029	CGMN-MW110-DB-140716	83.4	NA	26.7	NA	14.4	NA
ISO11-01-03-16-030	CGMN-MW110-FMS-140716	99.9	NC	43.4	97.7	32.9	88.4
Average Concentration (ng/mL) ± %RPD		82.3 ng/mL ± 2.8%		24.1 ng/mL ± 22% ⁽¹⁾		15.4 ng/mL ± 13%	

3M LIMS ID	Description	¹² C ₃ -PFBA	¹² C ₄ -PFOA	¹² C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-028	CGMN-MW110-0-140716	102	94.0	100
ISO11-01-03-16-029	CGMN-MW110-DB-140716	105	95.3	98.8
ISO11-01-03-16-030	CGMN-MW110-FMS-140716	103	98.2	99.8
Average Recovery (%) ± %RSD		103% ± 1.5%	95.8% ± 2.2%	99.7% ± 0.79%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:100 and analyzed by external standard calibration.
 (1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 20. CGMN GW PW09 140717

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-032	CGMN-PW09-0-140717	4.32	NA	1.17	NA
ISO11-01-03-16-033	CGMN-PW09-DB-140717	4.32	NA	1.15	NA
ISO11-01-03-16-034	CGMN-PW09-FMS-140717	6.69	NC	3.23	107
Average Concentration (ng/mL) ± %RPD		4.32 ng/mL ± 0.0%		1.16 ng/mL ± 1.7%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-032	CGMN-PW09-0-140717	0.152	NA	0.0810	NA	1.79	NA
ISO11-01-03-16-033	CGMN-PW09-DB-140717	0.158	NA	0.0806	NA	1.78	NA
ISO11-01-03-16-034	CGMN-PW09-FMS-140717	2.06	96.2 ⁽¹⁾	2.26	110 ⁽¹⁾	3.84	104
Average Concentration (ng/mL) ± %RPD		0.155 ng/mL ± 3.9%		0.0808 ng/mL ± 0.50%		1.79 ng/mL ± 0.56%	

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-032	CGMN-PW09-0-140717	90.6	115	108
ISO11-01-03-16-033	CGMN-PW09-DB-140717	108	112	115
ISO11-01-03-16-034	CGMN-PW09-FMS-140717	112	119	108
Average Recovery (%) ± %RSD		103% ± 11%	115% ± 3.0%	110% ± 3.8%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 and analyzed by internal standard calibration.
 (1) FMS concentration greater than 10 times the sample concentration.

Table 21. CGMN GW PW10 140717

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-034	CGMN-PW10-0-140717	4.79	NA	0.479	NA
ISO11-01-03-16-035	CGMN-PW10-DB-140717	5.01	NA	0.489	NA
ISO11-01-03-16-036	CGMN-PW10-FMS-140717	7.44	NC	2.61	108
Average Concentration (ng/mL) ± %RPD		4.90 ng/mL ± 4.5%		0.484 ng/mL ± 2.1%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-034	CGMN-PW10-0-140717	0.522	NA	0.101	NA	0.498	NA
ISO11-01-03-16-035	CGMN-PW10-DB-140717	0.558	NA	0.106	NA	0.507	NA
ISO11-01-03-16-036	CGMN-PW10-FMS-140717	2.53	101	2.32	112 ⁽¹⁾	2.81	117
Average Concentration (ng/mL) ± %RPD		0.540 ng/mL ± 6.7%		0.104 ng/mL ± 4.8%		0.503 ng/mL ± 1.8%	

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₄ -PFOA	¹³ C ₆ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-034	CGMN-PW10-0-140717	106	114	117
ISO11-01-03-16-035	CGMN-PW10-DB-140717	112	120	109
ISO11-01-03-16-036	CGMN-PW10-FMS-140717	92.0	108	112
Average Recovery (%) ± %RSD		103% ± 9.7%	114% ± 5.3%	113% ± 3.8%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 and analyzed by internal standard calibration.
 (1) FMS concentration greater than 10 times the sample concentration.

Table 22. CGMN GW MW13-RB 140716 and TRIP BLANKS

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-037	CGMN-GW-MW13-RB-140716	<0.0500	NA	<0.0240	NA
ISO11-01-03-16-038	CGMN-GW-TRIP-0-140715	<0.0500	NA	<0.0240	NA
ISO11-01-03-16-039	CGMN-GW-TRIP-LS-140715	1.91	95.5	2.14	108
ISO11-01-03-16-040	CGMN-GW-TRIP-HS-140715	97.7	97.7	96.8	98.0

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-16-037	CGMN-GW-MW13-RB-140716	<0.0250	NA	<0.0250	NA	0.0388	NA
ISO11-01-03-16-038	CGMN-GW-TRIP-0-140715	<0.0250	NA	<0.0250	NA	<0.0232	NA
ISO11-01-03-16-039	CGMN-GW-TRIP-LS-140715	1.98	100	2.22	112	2.10	106
ISO11-01-03-16-040	CGMN-GW-TRIP-HS-140715	101	102	97.7	98.7	98.7	99.7

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-16-037	CGMN-GW-MW13-RB-140716	96.4	116	116
ISO11-01-03-16-038	CGMN-GW-TRIP-0-140715	90.9	115	114
ISO11-01-03-16-039	CGMN-GW-TRIP-LS-140715	92.3	112	102
ISO11-01-03-16-040	CGMN-GW-TRIP-HS-140715	107	101	104

NA = Not Applicable

Samples were diluted 1:1 and analyzed by internal standard calibration with the exception of the TRIP HS, which was diluted 1:100 and analyzed by external standard calibration.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10-22 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Attachments

Appendix A: Target Analyte Historical Trend Data for Cottage Grove Monitoring Wells MW07, MW12, MW13, MW16, MW101, MW104, MW105, MW108, MW110, PW09, and PW10.

8 Signatures



Digitally signed by Susan T. Wolf
DN: c=US, st=MN, l=St. Paul, ou=3M
Environmental Laboratory - authenticated by LRA,
email=swolf@mnm.com, o=3M, cn=Susan T. Wolf
Reason: I have reviewed this document
Date: 2014.08.08 10:42:31 -0500

Susan T. Wolf, 3M Principal Analytical Investigator



Digitally signed by William K. Reagen
DN: c=US, st=MN, l=St. Paul, ou=Laboratory Director,
ou=3M Environmental Laboratory - authenticated by LRA,
email=wkreagen@mnm.com, o=3M, cn=William
K. Reagen
Reason: I am approving this document
Date: 2014.08.11 10:37:18 -0500

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.



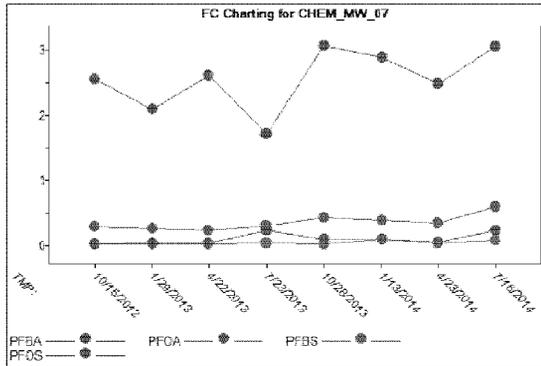
Digitally signed by Matthew D. Nauko-Stewart
DN: c=US, st=MN, l=St. Paul, ou=Quality Assurance Unit,
ou=3M Environmental Laboratory - authenticated by LRA,
email=mnstewart@mnm.com, o=3M, cn=Matthew D. Nauko-
Stewart
Reason: I agree to the terms defined by the placement of my
signature on this document
Date: 2014.08.12 09:36:39 -0500

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M Environmental Laboratory.

Appendix A: Target Analyte Historical Trend Data

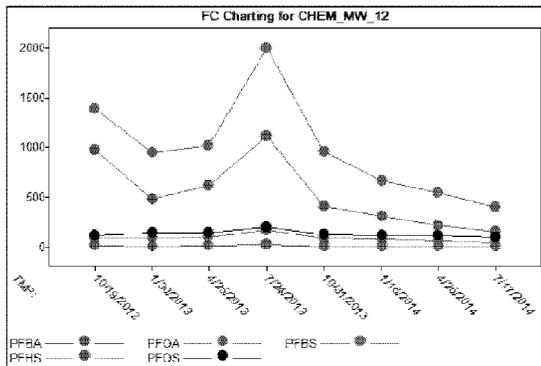
Cottage Grove MW07 – units are ng/mL



MW07	10/15/2012	1/29/2013	4/22/2013	7/23/2013	10/28/2013	1/13/2014	4/23/2014	7/16/2014
PFBA	2.55	2.09	2.61	1.71	3.07	2.88	2.48	3.05
PFOA	0.298	0.259	0.240	0.303	0.428	0.390	0.344	0.602
PFBS	<0.0250	<0.0250	0.0268	0.0396	0.0291	<0.100	0.0360	0.0762
PFOS	0.0257	0.0439	0.0459	0.241	0.104	0.104	0.0493	0.237

Samples were below the limit of quantitation for PFHS.

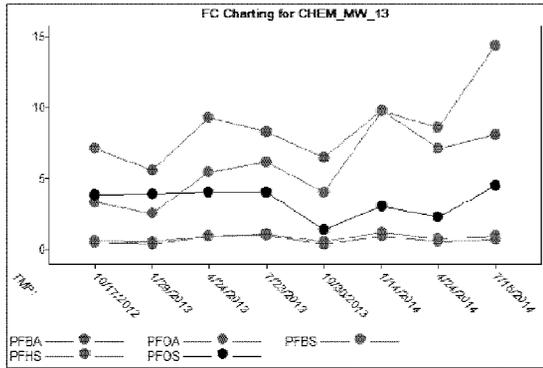
Cottage Grove MW12 – units are ng/mL



MW12	10/19/2012	1/30/2013	4/25/2013	7/24/2013	10/31/2013	1/16/2014	4/25/2014	7/17/2014
PFBA	975	484	622	1110	408	312	220	154
PFOA	1390	946	1020	2000	954	668	548	402
PFBS	94.3	91.4	105	175	87.7	82.1	68.9	50.4
PFHS	20.9	13.7	16.4	23.9	13.0	13.1	9.82	8.11
PFOS	122	143	145	204	131	121	117	96.9

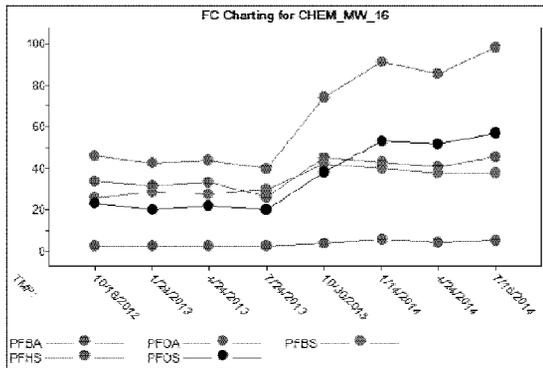
Appendix A: Target Analyte Historical Trend Data

Cottage Grove MW13 – units are ng/mL



MW13	10/17/2012	1/29/2013	4/24/2013	7/23/2013	10/30/2013	1/14/2014	4/24/2014	7/16/2014
PFBA	7.10	5.60	9.34	8.30	6.48	9.80	7.14	8.08
PFOA	3.33	2.56	5.46	6.17	4.01	9.78	8.63	14.4
PFBS	0.619	0.56	0.972	1.07	0.571	1.20	0.752	0.973
PFHS	0.487	0.396	0.931	1.05	0.366	0.969	0.556	0.713
PFOS	3.85	3.92	4.05	4.02	1.40	3.08	2.33	4.48

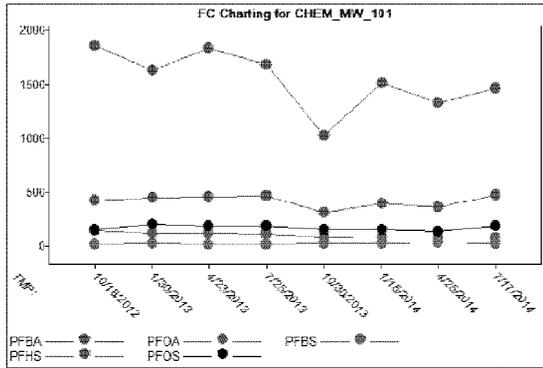
Cottage Grove MW16 – units are ng/mL



MW16	10/18/2012	1/29/2013	4/24/2013	7/24/2013	10/30/2013	1/14/2014	4/24/2014	7/16/2014
PFBA	33.6	31.5	33.2	26.3	45.0	42.9	40.7	45.7
PFOA	45.9	42.6	43.8	39.9	73.9	91.0	85.7	98.3
PFBS	25.7	28.3	27.5	29.6	42.1	40.0	37.9	37.9
PFHS	2.71	2.54	2.74	2.83	3.95	5.52	4.49	5.46
PFOS	23.1	20.2	21.8	20.1	38.2	53.1	51.9	57.0

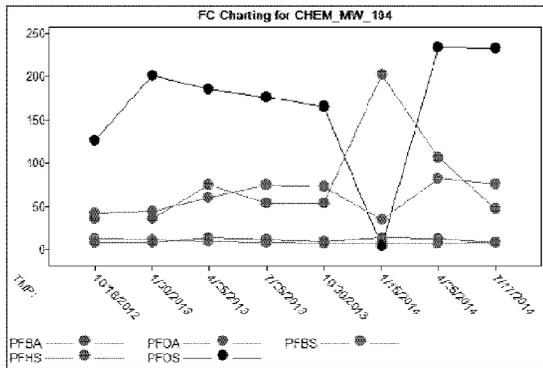
Appendix A: Target Analyte Historical Trend Data

Cottage Grove MW101 – units are ng/mL



MW101	10/18/2012	1/30/2013	4/23/2013	7/25/2013	10/30/2013	1/15/2014	4/25/2014	7/17/2014
PFBA	1860	1630	1830	1680	1020	1510	1330	1460
PFOA	147	124	121	112	86.7	79.4	76.0	83.9
PFBS	23.9	25.2	19.5	24.0	25.5	25.4	37.8	29.3
PFHS	427	455	459	464	314	398	364	480
PFOS	154	206	188	189	158	159	135	193

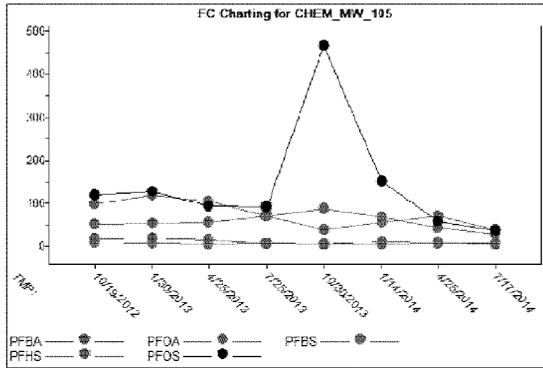
Cottage Grove MW104 – units are ng/mL



MW104	10/18/2012	1/30/2013	4/25/2013	7/25/2013	10/30/2013	1/15/2014	4/25/2014	7/17/2014
PFBA	36.0	36.3	74.7	54.3	53.6	202	107	47.9
PFOA	42.0	44.8	60.7	74.7	72.5	35.1	82.1	76.5
PFBS	12.6	11.4	9.75	8.16	7.95	7.50	8.10	8.68
PFHS	8.69	9.07	13.4	11.8	10.1	14.2	12.6	8.95
PFOS	127	201	185	176	165	4.25	234	233

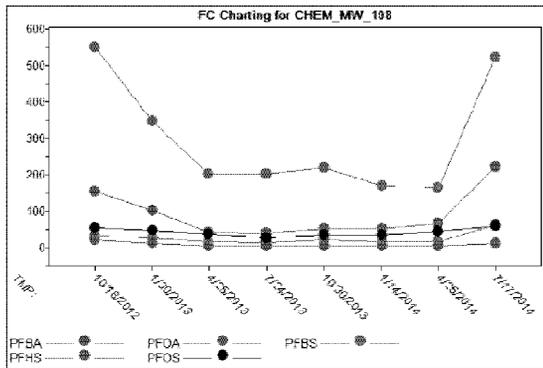
Appendix A: Target Analyte Historical Trend Data

Cottage Grove MW105 – units are ng/mL



MW105	10/19/2012	1/30/2013	4/25/2013	7/25/2013	10/30/2013	1/14/2014	4/25/2014	7/17/2014
PFBA	52.7	54.4	57.0	71.6	38.9	55.6	71.0	39.2
PFOA	98.3	119	104	70.6	87.2	68.3	44.3	27.7
PFBS	9.46	8.03	5.72	6.49	4.98	6.15	7.68	4.89
PFHS	18.9	21.1	16.0	6.35	6.01	12.6	8.59	6.65
PFOS	120	129	95.0	92.3	467	152	58.7	36.2

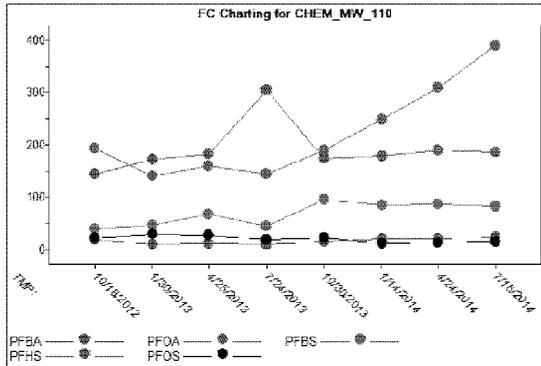
Cottage Grove MW108 – units are ng/mL



MW108	10/18/2012	1/30/2013	4/25/2013	7/24/2013	10/30/2013	1/14/2014	4/25/2014	7/17/2014
PFBA	155	101	42.5	38.0	51.7	51.6	65.4	222
PFOA	550	348	201	202	218	169	164	522
PFBS	30.5	26.5	16.1	14.6	21.6	17.0	17.4	60.7
PFHS	20.7	11.1	4.95	4.94	4.47	4.13	3.58	11.3
PFOS	55.2	45.5	37.3	26.8	33.2	33.5	43.3	58.0

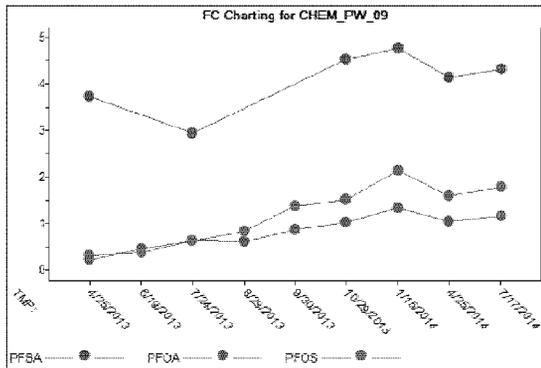
Appendix A: Target Analyte Historical Trend Data

Cottage Grove MW110 – units are ng/mL



MW110	10/18/2012	1/30/2013	4/25/2013	7/24/2013	10/30/2013	1/14/2014	4/24/2014	7/16/2014
PFBA	144	171	183	304	174	179	189	185
PFOA	193	141	160	144	190	248	310	390
PFBS	39.6	46.6	66.7	43.8	94.8	85.1	86.1	82.3
PFHS	17.6	10.3	11.8	9.41	15.3	19.4	20.9	24.1
PFOS	21.6	28.6	26.4	17.8	21.1	12.1	12.5	15.4

Cottage Grove PW09 – units are ng/mL

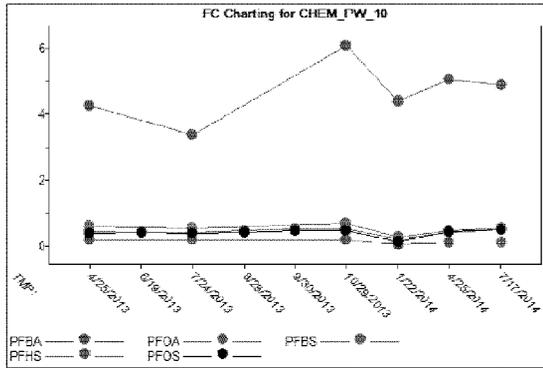


PW09	4/25/2013	6/19/2013	7/24/2013	8/29/2013	9/30/2013	10/29/2013	1/15/2014	4/25/2014	7/17/2014
PFBA	3.72	NA	2.95	NA	NA	4.53	4.76	4.14	4.32
PFOA	0.223	0.462	0.624	0.605	0.873	1.02	1.33	1.05	1.16
PFOS	0.324	0.376	0.622	0.818	1.38	1.52	2.13	1.6	1.79

NA = Not Applicable; analyte not requested for the sampling event.

Appendix A: Target Analyte Historical Trend Data

Cottage Grove PW10 – units are ng/mL



PW10	4/25/2013	6/19/2013	7/24/2013	8/29/2013	9/30/2013	10/29/2013	1/22/2014	4/25/2014	7/17/2014
PFBA	4.25	NA	3.37	NA	NA	6.09	4.40	5.07	4.90
PFOA	0.428	0.415	0.420	0.471	0.528	0.524	0.212	0.421	0.484
PFBS	0.594	NA	0.540	NA	NA	0.703	0.261	0.471	0.540
PFHS	0.197	NA	0.185	NA	NA	0.193	0.0485	0.110	0.104
PFOS	0.396	0.401	0.38	0.415	0.465	0.458	0.124	0.437	0.503

NA = Not Applicable; analyte not requested for the sampling event.



OCTOBER 2014

Final Report

Analysis of PFBA, PFOA, PFBS, PFHS, and PFOS in Cottage Grove Groundwater

4th Quarter 2014 Sampling

Laboratory Request Number: ISO11-01-03-18

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Gary Hohenstein
3M EHS Operations
3M Building 224-5W-03
Saint Paul, MN 55144-1000
Phone: (651) 737-3570



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.

3M Principal Analytical Investigator: Susan Wolf

Report Author: Kevin Eich

Analytical Report ISO11-01-03-18

Analysis of PFBA, PFOA, PFBS, PFHS, and PFOS in Cottage Grove Groundwater
4th Quarter 2014 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater samples collected by Weston Solutions personnel at the 3M Cottage Grove facility. Samples were collected on October 28-30, 2014. Samples were returned to the 3M Environmental Laboratory on October 30, 2014 on ice for the analysis of Perfluorobutanoic acid (PFBA), Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS) and Perfluorooctane sulfonate (PFOS) under laboratory project number ISO11-01-03-18.

The 3M Environmental Laboratory prepared sample containers for twelve sampling locations. Each sample set consisted of a field sample, field sample duplicate, and a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. Select sample bottles were fortified with internal standards and surrogate recovery standards prior to being sent to the field for sample collection.

Samples were prepared and analyzed for PFBA, PFOA, PFBS, PFHS, and PFOS using method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Internal standards were used to aid in the data quality objectives for the analysis of select samples, were applicable.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
ISO11-01-03-18-001	CGMN-GW-MW07-0-141028	3.15	0.615	0.0762	0.0511	0.194
ISO11-01-03-18-002	CGMN-GW-MW07-DB-141028	2.87	0.547	0.0639	0.0495	0.187
Average		3.01	0.581	0.0701	0.0503	0.191
%RPD Sample/Sample Dup		9.3	12	18	3.2	3.7
ISO11-01-03-18-004	CGMN-GW-MW12-0-141030	126	345	45.8	7.79	92.0
ISO11-01-03-18-005	CGMN-GW-MW12-DB-141030	128	350	46.4	7.76	93.0
Average		127⁽²⁾	348⁽²⁾	46.1⁽²⁾	7.78⁽²⁾	92.5⁽²⁾
%RPD Sample/Sample Dup		1.6	1.4	1.3	0.39	1.1
ISO11-01-03-18-007	CGMN-GW-MW13-0-141028	9.52	9.76	1.07	0.712	4.83
ISO11-01-03-18-008	CGMN-GW-MW13-DB-141028	9.76	10.3	1.10	0.727	4.94
Average		9.64	10.0	1.09	0.720	4.89
%RPD Sample/Sample Dup		2.5	5.4	2.8	2.1	2.3
ISO11-01-03-18-010	CGMN-GW-MW14R-0-141029	570	355	38.7	16.5	85.2
ISO11-01-03-18-011	CGMN-GW-MW14R-DB-141029	569	351	37.4	16.7	80.0
Average		570⁽²⁾	353⁽²⁾	38.1⁽²⁾	16.6⁽²⁾	82.6⁽²⁾
%RPD Sample/Sample Dup		0.18	1.1	3.4	1.2	6.3
ISO11-01-03-18-013	CGMN-GW-MW16-0-141028	38.2	73.7	32.8	4.45	41.7
ISO11-01-03-18-014	CGMN-GW-MW16-DB-141028	39.6	77.8	35.0	4.71	43.4
Average		38.9	75.8	33.9	4.58	42.6
%RPD Sample/Sample Dup		3.6	5.4	6.5	5.7	4.0
ISO11-01-03-18-016	CGMN-GW-MW101-0-141029	1410	75.2	29.6	460	165
ISO11-01-03-18-017	CGMN-GW-MW101-DB-141029	1400	74.4	29.6	443	174
Average		1410⁽²⁾	74.8⁽²⁾	29.6⁽²⁾	452⁽²⁾	170⁽²⁾
%RPD Sample/Sample Dup		0.71	1.1	0.0	3.8	5.3
ISO11-01-03-18-019	CGMN-GW-MW104-0-141029	31.0	50.3	7.73	5.81	176
ISO11-01-03-18-020	CGMN-GW-MW104-DB-141029	30.8	49.7	7.71	6.67	177
Average		30.9⁽²⁾	50.0⁽²⁾	7.72⁽²⁾	6.24⁽²⁾	177⁽²⁾
%RPD Sample/Sample Dup		0.65	1.2	0.26	14	0.57
ISO11-01-03-18-022	CGMN-GW-MW105-0-141029	45.6	93.8	3.88	18.7	73.9
ISO11-01-03-18-023	CGMN-GW-MW105-DB-141029	47.0	91.8	4.13	19.0	73.0
Average		46.3⁽²⁾	92.8⁽²⁾	4.01⁽²⁾	18.9⁽²⁾	73.5⁽²⁾
%RPD Sample/Sample Dup		3.0	2.2	6.2	1.6	1.2
ISO11-01-03-18-025	CGMN-GW-MW108-0-141029	88.1	268	23.0	8.20	42.3
ISO11-01-03-18-026	CGMN-GW-MW108-DB-141029	85.9	267	22.4	8.79	41.6
Average		87.0⁽²⁾	268⁽²⁾	22.7⁽²⁾	8.50⁽²⁾	42.0⁽²⁾
%RPD Sample/Sample Dup		2.5	0.37	2.6	6.9	1.7

- (1) Sample set was analyzed by internal standard calibration, except where noted. The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFBA ± 14%, PFOA ± 22%, PFBS ± 16%, PFHS ± 12%, and PFOS ± 16%.
- (2) Sample set was analyzed by external standard calibration. The analytical method uncertainties associated with the reported results by external standard calibration are as follows: PFBA ± 24%, PFOA ± 14%, PFBS ± 8.6%, PFHS ± 11%, and PFOS ± 15%.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
ISO11-01-03-18-028	CGMN-GW-MW110-0-141028	290	415	57.9	23.4	12.9
ISO11-01-03-18-029	CGMN-GW-MW110-DB-141028	297	419	57.3	23.6	12.8
Average		294⁽²⁾	417⁽²⁾	57.6⁽²⁾	23.5⁽²⁾	12.9⁽²⁾
%RPD Sample/Sample Dup		2.4	0.96	1.0	0.85	0.78
ISO11-01-03-18-031	CGMN-GW-PW09-0-141030	3.84	0.721	0.101	0.0560	0.919
ISO11-01-03-18-032	CGMN-GW-PW09-DB-141030	3.90	0.681	0.110	0.0522	0.757
Average		3.87	0.701	0.106	0.0541	0.838
%RPD Sample/Sample Dup		1.6	5.7	8.5	7.0	19
ISO11-01-03-18-034	CGMN-GW-PW10-0-141030	4.48	0.379	0.412	0.0795	0.345
ISO11-01-03-18-035	CGMN-GW-PW10-DB-141030	4.38	0.372	0.387	0.0759	0.329
Average		4.43	0.376	0.400	0.0777	0.337
%RPD Sample/Sample Dup		2.3	1.9	6.3	4.6	4.7

- (1) Sample set was analyzed by internal standard calibration, except where noted. The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFBA ± 14%, PFOA ± 22%, PFBS ± 16%, PFHS ± 12%, and PFOS ± 16%.
- (2) Sample set was analyzed by external standard calibration. The analytical method uncertainties associated with the reported results by external standard calibration are as follows: PFBA ± 24%, PFOA ± 14%, PFBS ± 8.6%, PFHS ± 11%, and PFOS ± 15%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorobutanoic Acid (C4 Acid)	PFBA	Linear
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched

2.2 Sample Collection

Samples were collected on October 28-30, 2014 in Nalgene™ (high-density polyethylene) bottles prepared at the 3M Environmental Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. Collected sample bottles were returned to the laboratory on ice on October 30, 2014.

2.3 Sample Preparation

Sample concentrations were expected to range from <0.025 ng/mL to >1000 ng/mL. Sampling locations that were expected to have concentration <100 ng/mL were analyzed by internal standard calibration analysis. Sampling locations that were expected to have concentration >100 ng/mL were analyzed by external standard calibration analysis. The following sample preparation procedures were followed for each type of analysis.

Internal standard calibration analysis: Samples analyzed by internal standard calibration were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2).

During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples.

External standard calibration analysis: Samples analyzed by external standard calibration required dilution prior to analysis. Samples were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol (dilution factor of 100). An aliquot of surrogate spiking solution was added to the diluted samples at a nominal concentration of 1 ng/mL.

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

Table 3. Instrument Parameters.

Instrument Name	ETS Buster
Liquid Chromatograph	Agilent 1100
Analysis Method	ETS-8-044.1
Analysis Date	11/18/14 and 11/20/14
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 µ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 µ
Injection Volume	10 µL, 30 µL
Mass Spectrometer	AB Sciex Triple Quad 4000
Ion Source	Turbo Spray
Polarity	Negative
Software	Analyst 1.6.2

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.1 Analysis				
Step Number	Total Time (min)	Flow Rate (μL/min)	Percent A (Methanol)	Percent B (2 mM ammonium acetate)
0	0.00	750	3.0	97.0
1	0.50	750	3.0	97.0
2	4.00	750	30.0	70.0
3	6.00	750	30.0	70.0
4	11.0	750	80.0	20.0
5	13.0	750	80.0	20.0
6	13.5	750	90.0	10.0
7	16.0	750	90.0	10.0
8	16.5	750	3.0	97.0
9	19.0	750	3.0	97.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard ⁽¹⁾	Mass Transition Q1/Q3
PFBA	213/169	[¹³ C ₃]-PFBA	217/172
PFOA	413/369	[¹³ C ₈]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/80	[¹⁸ O ₂]-PFBS	303/84
	299/99		
PFHS	399/99	[¹³ C ₃]-PFHS	402/80
	399/80		
PFOS	499/99	[¹³ C ₈]-PFOS	507/80
	499/80		
	499/130		
[¹³ C ₃]-PFBA	216/172	[¹³ C ₃]-PFBA	217/172
[¹³ C ₄]-PFOA	417/372	[¹³ C ₈]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₈]-PFOS	507/80

Dwell time was 50 to 100 msec for each transition. The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standard was not used for the samples analyzed by solvent dilution external standard calibration.

3 Data Analysis

3.1 Calibration

Solvent dilution analysis using internal standard calibration: Samples were analyzed for all analytes against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 methanol:laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. Calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed (0.0125 ng/mL to 10 ng/mL (nominal) for the SRSSs). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

Solvent dilution analysis using external standard calibration: Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution into 50 mL of 90:10 methanol: laboratory Milli-Q™ water. Calibration standards ranging from 0.02 ng/mL to 50 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. Low or high points were disabled to meet method criteria. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

For both methods of analysis, each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area counts or peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.1 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the Table 6 below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾	LOQ, ng/mL ⁽²⁾
	11/18/14 Analysis	11/20/14 Analysis
PFBA	0.0250	2.00
PFOA	0.0240	1.92
PFBS	0.0250	2.00
PFHS	0.0250	2.00
PFOS	0.0232	1.85

(1) A dilution factor of 2 was applied to the LOQ.

(2) A dilution factor of 100 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±25%.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: method/solvent blanks, field/trip blanks, and sampling equipment blanks. Each blank result was reviewed and used to evaluate method performance. The method/solvent blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs prepared for internal standard calibration analysis were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. LCSs prepared for external standard calibration analysis were prepared by spiking known amounts of the analytes into 1.0 mL of laboratory reagent water and 9.0 mL of methanol to produce the desired concentration. Method ETS-8-044.1 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report.

The following calculations were used to generate data in Table 7.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} \times 100\%$$

$$\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} \times 100\%$$

Table 7. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 11/18/14	PFBA			PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141118-1	0.198	0.181	91.6	0.190	0.189	99.2
LCS-141118-2	0.198	0.172	86.8	0.190	0.184	96.6
LCS-141118-3	0.198	0.187	94.5	0.190	0.191	100
Average ± %RSD	91.0% ± 4.3%			98.6% ± 1.8%		
LCS-141118-4	9.91	9.72	98.1	9.50	8.90	93.6
LCS-141118-5	9.91	9.81	98.9	9.50	8.97	94.4
LCS-141118-6	9.91	9.63	97.1	9.50	8.85	93.2
Average ± %RSD	98.0% ± 0.92%			93.7% ± 0.65%		
LCS-141118-7	157	149	94.9	150	126	83.8
LCS-141118-8	157	144	91.7	150	127	84.5
LCS-141118-9	157	142	90.8	150	124	82.9
Average ± %RSD	92.5% ± 2.3%			83.7% ± 0.96%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 11/18/14						
Lab ID	PFBS			PFHS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141118-1	0.198	0.211	107	0.198	0.211	107
LCS-141118-2	0.198	0.220	111	0.198	0.208	105
LCS-141118-3	0.198	0.220	111	0.198	0.207	104
Average ± %RSD	110% ± 2.1%			105% ± 1.5%		
LCS-141118-4	9.92	10.2	102	9.92	9.21	92.8
LCS-141118-5	9.92	10.3	104	9.92	9.48	95.5
LCS-141118-6	9.92	10.2	102	9.92	9.18	92.6
Average ± %RSD	103% ± 1.1%			93.6% ± 1.7%		
LCS-141118-7	157	155	99.0	157	153	97.6
LCS-141118-8	157	148	94.3	157	147	93.6
LCS-141118-9	157	147	93.4	157	147	93.6
Average ± %RSD	95.6% ± 3.1%			94.9% ± 2.4%		

ETS-8-044.1 Internal standard calibration Analyzed 11/18/14			
PFOS (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141118-1	0.184	0.176	95.8
LCS-141118-2	0.184	0.176	95.6
LCS-141118-3	0.184	0.180	98.1
Average ± %RSD	96.5% ± 1.4%		
LCS-141118-4	9.20	8.40	91.3
LCS-141118-5	9.20	8.64	94.0
LCS-141118-6	9.20	8.18	88.9
Average ± %RSD	91.4% ± 2.8%		
LCS-141118-7	145	133	92.0
LCS-141118-8	145	124	85.3
LCS-141118-9	145	126	86.6
Average ± %RSD	88.0% ± 4.0%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal standard calibration Analyzed 11/18/14						
¹³ C ₃ -PFBA				¹³ C ₄ -PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141118-1	0.197	0.208	105	0.198	0.207	105
LCS-141118-2	0.197	0.215	109	0.198	0.210	106
LCS-141118-3	0.197	0.218	110	0.198	0.212	107
Average ± %RSD	108% ± 2.4%			106% ± 0.94%		
LCS-141118-4	1.97	1.94	98.7	1.98	1.96	99.1
LCS-141118-5	1.97	1.90	96.6	1.98	1.90	96.0
LCS-141118-6	1.97	1.94	98.6	1.98	1.92	96.7
Average ± %RSD	98.0% ± 1.2%			97.3% ± 1.7%		

ETS-8-044.1 Internal standard calibration Analyzed 11/18/14			
¹³ C ₄ -PFOS			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141118-1	0.189	0.188	99.5
LCS-141118-2	0.189	0.196	104
LCS-141118-3	0.189	0.212	112
Average ± %RSD	105% ± 6.0%		
LCS-141118-4	1.90	1.89	99.6
LCS-141118-5	1.90	1.89	99.7
LCS-141118-6	1.90	1.85	97.2
Average ± %RSD	98.8% ± 1.4%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External standard calibration Analyzed 11/20/14						
Lab ID	PFBA			PFOA (Linear + Branched)		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141120-1	20.2	21.6	107	19.9	19.3	97.1
LCS-141120-2	20.2	21.0	104	19.9	19.0	95.3
LCS-141120-3	20.2	19.9	98.6	19.9	18.7	94.1
Average ± %RSD	103% ± 4.1%			95.5% ± 1.6%		
LCS-141120-4	502	488	97.1	496	480	96.8
LCS-141120-5	502	487	97.0	496	481	96.9
LCS-141120-6	502	505	101	496	493	99.4
Average ± %RSD	98.4% ± 2.3%			97.7% ± 1.5%		
LCS-141120-7	4040	4160	103	3980	3620	90.8
LCS-141120-8	4040	4080	101	3980	3460	86.9
LCS-141120-9	4040	4090	101	3980	3600	90.4
Average ± %RSD	102% ± 1.1%			89.4% ± 2.4%		

ETS-8-044.1 External standard calibration Analyzed 11/20/14						
Lab ID	PFBS			PFHS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141120-1	19.8	21.3	108	20.0	22.0	110
LCS-141120-2	19.8	21.2	107	20.0	20.6	103
LCS-141120-3	19.8	21.0	106	20.0	20.6	103
Average ± %RSD	107% ± 0.93%			105% ± 3.8%		
LCS-141120-4	494	495	100	498	498	99.9
LCS-141120-5	494	492	99.6	498	512	103
LCS-141120-6	494	503	102	498	513	103
Average ± %RSD	101% ± 1.3%			102% ± 1.8%		
LCS-141120-7	3970	4090	103	4000	4480	112
LCS-141120-8	3970	3940	99.3	4000	4400	110
LCS-141120-9	3970	3960	99.7	4000	4470	112
Average ± %RSD	101% ± 2.0%			111% ± 1.0%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External standard calibration Analyzed 11/20/14			
PFOS (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141120-1	19.9	16.6	83.7
LCS-141120-2	19.9	16.2	81.2
LCS-141120-3	19.9	15.5	78.0
Average ± %RSD	81.0% ± 3.5%		
LCS-141120-4	496	442	89.1
LCS-141120-5	496	440	88.7
LCS-141120-6	496	469	94.6
Average ± %RSD	90.8% ± 3.6%		
LCS-141120-7	3980	3990	100
LCS-141120-8	3980	3930	98.6
LCS-141120-9	3980	3940	98.8
Average ± %RSD	99.1% ± 0.76%		

ETS-8-044.1 External standard calibration Analyzed 11/20/14						
¹³ C ₃ -PFBA				¹³ C ₄ -PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141120-1	19.9	22.8	115	19.9	23.2	117
LCS-141120-2	19.9	23.2	117	19.9	23.4	117
LCS-141120-3	19.9	22.6	113	19.9	22.9	115
Average ± %RSD	115% ± 1.7%			116% ± 1.0%		
LCS-141120-4	199	216	109	199	221	111
LCS-141120-5	199	215	108	199	213	107
LCS-141120-6	199	215	108	199	209	105
Average ± %RSD	108% ± 0.53%			108% ± 2.8%		

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External standard calibration Analyzed 11/20/14	¹³ C ₄ -PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141120-1	19.1	22.6	118
LCS-141120-2	19.1	21.0	110
LCS-141120-3	19.1	22.5	118
Average ± %RSD	115% ± 4.0%		
LCS-141120-4	191	205	107
LCS-141120-5	191	201	105
LCS-141120-6	191	204	107
Average ± %RSD	106% ± 1.1%		

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.1, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFBA	Internal	6.78	±14%
PFOA	Internal	10.8	±22%
PFBS	Internal	7.91	±16%
PFHS	Internal	6.13	±12%
PFOS	Internal	8.24	±16%
PFBA	External	12.1	±24%
PFOA	External	7.23	±14%
PFBS	External	4.31	±8.6%
PFHS	External	5.39	±11%
PFOS	External	7.55	±15%

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample was collected at each sampling point to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, each sample contained stable isotope surrogate recovery spikes of ¹³C₃-PFBA, ¹³C₄-PFOA, and ¹³C₄-PFOS, which were added at a nominal concentration of 0.1 ng/mL to select sample bottles prior to sample collection or at a nominal concentration of 1 ng/mL following sample collection. The ¹³C₃-PFBA and ¹³C₄-PFOA were selected to represent perfluorocarboxylic acids. The ¹³C₄-labeled PFOS was selected to represent the perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

$$\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%$$

Table 9. Field Matrix Spike Concentrations

Location	Spike Level	Final Concentration (ng/mL)				
		PFBA	PFOA	PFBS	PFHS	PFOS
MW07, PW09, and PW10	FMS	2.02	1.99	1.98	2.00	1.99
MW13	FMS	10.1	9.96	9.92	10.0	9.96
MW110	FMS	20.2	19.9	19.8	20.0	19.9
MW16 and MW105	FMS	50.5	49.8	49.6	50.0	49.8
MW101, MW104, and MW108	FMS	101	99.6	99.2	100	99.6
MW12 and MW14R	FMS	252	249	248	251	249
Trip Blank	Low	2.02	1.99	1.98	2.00	1.99
	High	101	99.6	99.2	100	99.6

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of ±30%, demonstrate that the method is appropriate for the given matrix.

For those analytes where the field matrix spike level was not appropriate as compared to the sample concentration, the surrogate recovery standards were used to assess method accuracy. All surrogate recovery standards and field matrix spike recoveries met method acceptance criteria (where applicable).

Table 10. CGMN GW MW07 141028

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-001	CGMN-GW-MW07-0-141028	3.15	NA	0.615	NA
ISO11-01-03-18-002	CGMN-GW-MW07-DB-141028	2.87	NA	0.547	NA
ISO11-01-03-18-003	CGMN-GW-MW07-FMS-141028	4.95	96.0	2.50	96.3
Average Concentration (ng/mL) ± %RPD		3.01 ng/mL ± 9.3%		0.581 ng/mL ± 12%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-001	CGMN-GW-MW07-0-141028	0.0762	NA	0.0511	NA	0.194	NA
ISO11-01-03-18-002	CGMN-GW-MW07-DB-141028	0.0639	NA	0.0495	NA	0.187	NA
ISO11-01-03-18-003	CGMN-GW-MW07-FMS-141028	1.98	96.3	2.00	97.5	1.99	90.3
Average Concentration (ng/mL) ± %RPD		0.0701 ng/mL ± 18%		0.0503 ng/mL ± 3.2%		0.191 ng/mL ± 3.7%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₂ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-001	CGMN-GW-MW07-0-141028	120	116	115
ISO11-01-03-18-002	CGMN-GW-MW07-DB-141028	103	97.3	108
ISO11-01-03-18-003	CGMN-GW-MW07-FMS-141028	103	103	102
Average Recovery (%) ± %RSD		108% ± 9.1%	105% ± 9.1%	108% ± 6.1%

NA = Not Applicable
 Samples were diluted 1:1 and analyzed by internal standard calibration.

Table 11. CGMN GW MW12 141030

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-004	CGMN-GW-MW12-0-141030	126	NA	345	NA
ISO11-01-03-18-005	CGMN-GW-MW12-DB-141030	128	NA	350	NA
ISO11-01-03-18-006	CGMN-GW-MW12-FMS-141030	367	95.4	576	91.8
Average Concentration (ng/mL) ± %RPD		127 ng/mL ± 1.6%		348 ng/mL ± 1.4%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-004	CGMN-GW-MW12-0-141030	45.8	NA	7.79	NA	92.0	NA
ISO11-01-03-18-005	CGMN-GW-MW12-DB-141030	46.4	NA	7.76	NA	93.0	NA
ISO11-01-03-18-006	CGMN-GW-MW12-FMS-141030	292	99.2	250	96.5	322	92.2
Average Concentration (ng/mL) ± %RPD		46.1 ng/mL ± 1.3%		7.78 ng/mL ± 0.39%		92.5 ng/mL ± 1.1%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-004	CGMN-GW-MW12-0-141030	118	113	113
ISO11-01-03-18-005	CGMN-GW-MW12-DB-141030	111	109	115
ISO11-01-03-18-006	CGMN-GW-MW12-FMS-141030	101	97.7	97.4
Average Recovery (%) ± %RSD		110% ± 7.9%	107% ± 7.6%	109% ± 9.0%

NA = Not Applicable
 Samples were diluted 1:100 and analyzed by external standard calibration.

Table 12. CGMN GW MW13 141028

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-007	CGMN-GW-MW13-0-141028	9.52	NA	9.76	NA
ISO11-01-03-18-008	CGMN-GW-MW13-DB-141028	9.76	NA	10.3	NA
ISO11-01-03-18-009	CGMN-GW-MW13-FMS-141028	19.4	96.6	19.0	90.1
Average Concentration (ng/mL) ± %RPD		9.64 ng/mL ± 2.5%		10.0 ng/mL ± 5.4%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-007	CGMN-GW-MW13-0-141028	1.07	NA	0.712	NA	4.83	NA
ISO11-01-03-18-008	CGMN-GW-MW13-DB-141028	1.10	NA	0.727	NA	4.94	NA
ISO11-01-03-18-009	CGMN-GW-MW13-FMS-141028	10.9	98.9	10.0	92.8	14.0	91.5
Average Concentration (ng/mL) ± %RPD		1.09 ng/mL ± 2.8%		0.720 ng/mL ± 2.1%		4.89 ng/mL ± 2.3%	

3M LIMS ID	Description	¹² C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-007	CGMN-GW-MW13-0-141028	104	99.8	103
ISO11-01-03-18-008	CGMN-GW-MW13-DB-141028	99.0	103	99.9
ISO11-01-03-18-009	CGMN-GW-MW13-FMS-141028	101	102	108
Average Recovery (%) ± %RSD		101% ± 2.3%	102% ± 1.6%	103% ± 3.9%

NA = Not Applicable
 Samples were diluted 1:1 and analyzed by internal standard calibration.

Table 13. CGMN GW MW14R 141029

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-010	CGMN-GW-MW14R-0-141029	570	NA	355	NA
ISO11-01-03-18-011	CGMN-GW-MW14R-DB-141029	569	NA	351	NA
ISO11-01-03-18-012	CGMN-GW-MW14R-FMS-141029	623	NC	597	98.0
Average Concentration (ng/mL) ± %RPD		570 ng/mL ± 0.18%		353 ng/mL ± 1.1%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-010	CGMN-GW-MW14R-0-141029	38.7	NA	16.5	NA	85.2	NA
ISO11-01-03-18-011	CGMN-GW-MW14R-DB-141029	37.4	NA	16.7	NA	80.0	NA
ISO11-01-03-18-012	CGMN-GW-MW14R-FMS-141029	282	98.4	273	102	318	94.5
Average Concentration (ng/mL) ± %RPD		38.1 ng/mL ± 3.4%		16.6 ng/mL ± 1.2%		82.6 ng/mL ± 6.3%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-010	CGMN-GW-MW14R-0-141029	111	112	109
ISO11-01-03-18-011	CGMN-GW-MW14R-DB-141029	109	108	108
ISO11-01-03-18-012	CGMN-GW-MW14R-FMS-141029	120	117	113
Average Recovery (%) ± %RSD		113% ± 5.2%	113% ± 4.0%	110% ± 2.5%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:100 and analyzed by external standard calibration.

Table 14. CGMN GW MW16 141028

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-013	CGMN-GW-MW16-0-141028	38.2	NA	73.7	NA
ISO11-01-03-18-014	CGMN-GW-MW16-DB-141028	39.6	NA	77.8	NA
ISO11-01-03-18-015	CGMN-GW-MW16-FMS-141028	84.2	89.7	111	79.8
Average Concentration (ng/mL) ± %RPD		38.9 ng/mL ± 3.6%		75.8 ng/mL ± 5.4%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-013	CGMN-GW-MW16-0-141028	32.8	NA	4.45	NA	41.7	NA
ISO11-01-03-18-014	CGMN-GW-MW16-DB-141028	35.0	NA	4.71	NA	43.4	NA
ISO11-01-03-18-015	CGMN-GW-MW16-FMS-141028	78.4	89.7	50.5	91.8	83.1	81.4
Average Concentration (ng/mL) ± %RPD		33.9 ng/mL ± 6.5%		4.58 ng/mL ± 5.7%		42.6 ng/mL ± 4.0%	

3M LIMS ID	Description	¹² C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-013	CGMN-GW-MW16-0-141028	102	103	100
ISO11-01-03-18-014	CGMN-GW-MW16-DB-141028	102	101	101
ISO11-01-03-18-015	CGMN-GW-MW16-FMS-141028	104	100	103
Average Recovery (%) ± %RSD		102% ± 1.1%	101% ± 1.5%	101% ± 1.3%

NA = Not Applicable
 Samples were diluted 1:1 and analyzed by internal standard calibration.

Table 15. CGMN GW MW101 141029

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-016	CGMN-GW-MW101-0-141029	1410	NA	75.2	NA
ISO11-01-03-18-017	CGMN-GW-MW101-DB-141029	1400	NA	74.4	NA
ISO11-01-03-18-018	CGMN-GW-MW101-FMS-141029	1480	NC	172	97.6
Average Concentration (ng/mL) ± %RPD		1410 ng/mL ± 0.71%		74.8 ng/mL ± 1.1%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-016	CGMN-GW-MW101-0-141029	29.6	NA	460	NA	165	NA
ISO11-01-03-18-017	CGMN-GW-MW101-DB-141029	29.6	NA	443	NA	174	NA
ISO11-01-03-18-018	CGMN-GW-MW101-FMS-141029	129	100	558	NC	259	89.9
Average Concentration (ng/mL) ± %RPD		29.6 ng/mL ± 0.0%		452 ng/mL ± 3.8%		170 ng/mL ± 5.3%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-016	CGMN-GW-MW101-0-141029	106	107	105
ISO11-01-03-18-017	CGMN-GW-MW101-DB-141029	110	110	112
ISO11-01-03-18-018	CGMN-GW-MW101-FMS-141029	108	108	111
Average Recovery (%) ± %RSD		108% ± 1.9%	109% ± 1.4%	109% ± 3.7%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:100 and analyzed by external standard calibration.

Table 16. CGMN GW MW104 141029

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-019	CGMN-GW-MW104-0-141029	31.0	NA	50.3	NA
ISO11-01-03-18-020	CGMN-GW-MW104-DB-141029	30.8	NA	49.7	NA
ISO11-01-03-18-021	CGMN-GW-MW104-FMS-141029	125	93.2	145	95.4
Average Concentration (ng/mL) ± %RPD		30.9 ng/mL ± 0.65%		50.0 ng/mL ± 1.2%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-019	CGMN-GW-MW104-0-141029	7.73	NA	5.81	NA	176	NA
ISO11-01-03-18-020	CGMN-GW-MW104-DB-141029	7.71	NA	6.67	NA	177	NA
ISO11-01-03-18-021	CGMN-GW-MW104-FMS-141029	102	95.0	103	96.8	271	94.9
Average Concentration (ng/mL) ± %RPD		7.72 ng/mL ± 0.26%		6.24 ng/mL ± 14%		177 ng/mL ± 0.57%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-019	CGMN-GW-MW104-0-141029	112	112	111
ISO11-01-03-18-020	CGMN-GW-MW104-DB-141029	117	119	114
ISO11-01-03-18-021	CGMN-GW-MW104-FMS-141029	115	115	113
Average Recovery (%) ± %RSD		114% ± 2.2%	116% ± 3.0%	113% ± 1.4%

NA = Not Applicable
 Samples were diluted 1:100 and analyzed by external standard calibration.

Table 17. CGMN GW MW105 141029

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-022	CGMN-GW-MW105-0-141029	45.6	NA	93.8	NA
ISO11-01-03-18-023	CGMN-GW-MW105-DB-141029	47.0	NA	91.8	NA
ISO11-01-03-18-024	CGMN-GW-MW105-FMS-141029	94.3	95.0	140	94.8
Average Concentration (ng/mL) ± %RPD		46.3 ng/mL ± 3.0%		92.8 ng/mL ± 2.2%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-022	CGMN-GW-MW105-0-141029	3.88	NA	18.7	NA	73.9	NA
ISO11-01-03-18-023	CGMN-GW-MW105-DB-141029	4.13	NA	19.0	NA	73.0	NA
ISO11-01-03-18-024	CGMN-GW-MW105-FMS-141029	53.2	99.2	66.8	95.9	117	87.4
Average Concentration (ng/mL) ± %RPD		4.01 ng/mL ± 6.2%		18.9 ng/mL ± 1.6%		73.5 ng/mL ± 1.2%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₆ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-022	CGMN-GW-MW105-0-141029	111	108	110
ISO11-01-03-18-023	CGMN-GW-MW105-DB-141029	115	112	111
ISO11-01-03-18-024	CGMN-GW-MW105-FMS-141029	112	112	111
Average Recovery (%) ± %RSD		112% ± 1.9%	111% ± 2.1%	111% ± 0.55%

NA = Not Applicable
 Samples were diluted 1:100 and analyzed by external standard calibration.

Table 18. CGMN GW MW108 141029

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-025	CGMN-GW-MW108-0-141029	88.1	NA	268	NA
ISO11-01-03-18-026	CGMN-GW-MW108-DB-141029	85.9	NA	267	NA
ISO11-01-03-18-027	CGMN-GW-MW108-FMS-141029	181	93.1	352	NC
Average Concentration (ng/mL) ± %RPD		87.0 ng/mL ± 2.5%		268 ng/mL ± 0.37%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-025	CGMN-GW-MW108-0-141029	23.0	NA	8.20	NA	42.3	NA
ISO11-01-03-18-026	CGMN-GW-MW108-DB-141029	22.4	NA	8.79	NA	41.6	NA
ISO11-01-03-18-027	CGMN-GW-MW108-FMS-141029	115	93.0	107	98.5	129	87.4
Average Concentration (ng/mL) ± %RPD		22.7 ng/mL ± 2.6%		8.50 ng/mL ± 6.9%		42.0 ng/mL ± 1.7%	

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-025	CGMN-GW-MW108-0-141029	116	114	116
ISO11-01-03-18-026	CGMN-GW-MW108-DB-141029	112	110	108
ISO11-01-03-18-027	CGMN-GW-MW108-FMS-141029	110	110	111
Average Recovery (%) ± %RSD		112% ± 2.7%	112% ± 2.1%	112% ± 3.8%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:100 and analyzed by external standard calibration.

Table 19. CGMN GW MW110 141028

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-028	CGMN-GW-MW110-0-141028	290	NA	415	NA
ISO11-01-03-18-029	CGMN-GW-MW110-DB-141028	297	NA	419	NA
ISO11-01-03-18-030	CGMN-GW-MW110-FMS-141028	314	NC	426	NC
Average Concentration (ng/mL) ± %RPD		294 ng/mL ± 2.4%		417 ng/mL ± 0.96%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-028	CGMN-GW-MW110-0-141028	57.9	NA	23.4	NA	12.9	NA
ISO11-01-03-18-029	CGMN-GW-MW110-DB-141028	57.3	NA	23.6	NA	12.8	NA
ISO11-01-03-18-030	CGMN-GW-MW110-FMS-141028	75.5	NC	43.0	97.5	29.8	85.1
Average Concentration (ng/mL) ± %RPD		57.6 ng/mL ± 1.0%		23.5 ng/mL ± 0.85%		12.9 ng/mL ± 0.78%	

3M LIMS ID	Description	¹² C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-028	CGMN-GW-MW110-0-141028	109	107	108
ISO11-01-03-18-029	CGMN-GW-MW110-DB-141028	113	109	111
ISO11-01-03-18-030	CGMN-GW-MW110-FMS-141028	111	106	108
Average Recovery (%) ± %RSD		111% ± 1.8%	108% ± 1.4%	109% ± 1.7%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:100 and analyzed by external standard calibration.

Table 20. CGMN GW PW09 141030

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-031	CGMN-GW-PW09-0-141030	3.84	NA	0.721	NA
ISO11-01-03-18-032	CGMN-GW-PW09-DB-141030	3.90	NA	0.681	NA
ISO11-01-03-18-033	CGMN-GW-PW09-FMS-141030	5.85	98.0	2.57	93.8
Average Concentration (ng/mL) ± %RPD		3.87 ng/mL ± 1.6%		0.701 ng/mL ± 5.7%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-031	CGMN-GW-PW09-0-141030	0.101	NA	0.0560	NA	0.919	NA
ISO11-01-03-18-032	CGMN-GW-PW09-DB-141030	0.110	NA	0.0522	NA	0.757	NA
ISO11-01-03-18-033	CGMN-GW-PW09-FMS-141030	2.07	99.0	1.96	95.3	2.68	92.5
Average Concentration (ng/mL) ± %RPD		0.106 ng/mL ± 8.5%		0.0541 ng/mL ± 7.0%		0.838 ng/mL ± 19%	

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₂ -PFOA	¹³ C ₂ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-031	CGMN-GW-PW09-0-141030	107	110	103
ISO11-01-03-18-032	CGMN-GW-PW09-DB-141030	105	103	112
ISO11-01-03-18-033	CGMN-GW-PW09-FMS-141030	102	102	102
Average Recovery (%) ± %RSD		104% ± 2.4%	105% ± 4.2%	106% ± 5.4%

NA = Not Applicable
 Samples were diluted 1:1 and analyzed by internal standard calibration.

Table 21. CGMN GW PW10 141030

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-034	CGMN-GW-PW10-0-141030	4.48	NA	0.379	NA
ISO11-01-03-18-035	CGMN-GW-PW10-DB-141030	4.38	NA	0.372	NA
ISO11-01-03-18-036	CGMN-GW-PW10-FMS-141030	6.66	NC	2.21	92.1
Average Concentration (ng/mL) ± %RPD		4.43 ng/mL ± 2.3%		0.376 ng/mL ± 1.9%	

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-034	CGMN-GW-PW10-0-141030	0.412	NA	0.0795	NA	0.345	NA
ISO11-01-03-18-035	CGMN-GW-PW10-DB-141030	0.387	NA	0.0759	NA	0.329	NA
ISO11-01-03-18-036	CGMN-GW-PW10-FMS-141030	2.34	97.8	1.99	95.6	2.16	91.5
Average Concentration (ng/mL) ± %RPD		0.400 ng/mL ± 6.3%		0.0777 ng/mL ± 4.6%		0.337 ng/mL ± 4.7%	

3M LIMS ID	Description	¹³ C ₂ -PFBA	¹³ C ₄ -PFOA	¹³ C ₂ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-034	CGMN-GW-PW10-0-141030	106	100	102
ISO11-01-03-18-035	CGMN-GW-PW10-DB-141030	106	107	92.1
ISO11-01-03-18-036	CGMN-GW-PW10-FMS-141030	109	103	111
Average Recovery (%) ± %RSD		107% ± 1.6%	103% ± 3.4%	102% ± 9.3%

NA = Not Applicable
 NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
 Samples were diluted 1:1 and analyzed by internal standard calibration.

Table 22. CGMN GW MW108-RB 141029 and TRIP BLANKS

3M LIMS ID	Description	PFBA		PFOA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-037	CGMN-GW-RB-MW108-0-141029	0.0385	NA	0.0319	NA
ISO11-01-03-18-038	CGMN-GW-TRIP-0-141028	<0.0250	NA	<0.0240	NA
ISO11-01-03-18-039	CGMN-GW-TRIP-LS-141028	2.00	99.0	1.84	92.4
ISO11-01-03-18-040	CGMN-GW-TRIP-HS-141028	89.7	88.8	94.0	94.4

3M LIMS ID	Description	PFBS		PFHS		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-18-037	CGMN-GW-RB-MW108-0-141029	<0.0250	NA	<0.0250	NA	0.0290	NA
ISO11-01-03-18-038	CGMN-GW-TRIP-0-141028	<0.0250	NA	<0.0250	NA	<0.0232	NA
ISO11-01-03-18-039	CGMN-GW-TRIP-LS-141028	1.91	96.3	1.82	91.0	1.69	84.8
ISO11-01-03-18-040	CGMN-GW-TRIP-HS-141028	92.4	93.1	98.4	98.4	91.8	92.2

3M LIMS ID	Description	¹³ C ₃ -PFBA	¹³ C ₄ -PFOA	¹³ C ₄ -PFOS
		%Recovery	%Recovery	%Recovery
ISO11-01-03-18-037	CGMN-GW-RB-MW108-0-141029	106	102	105
ISO11-01-03-18-038	CGMN-GW-TRIP-0-141028	105	103	114
ISO11-01-03-18-039	CGMN-GW-TRIP-LS-141028	106	101	104
ISO11-01-03-18-040	CGMN-GW-TRIP-HS-141028	116	112	112

NA = Not Applicable

Samples were diluted 1:1 and analyzed by internal standard calibration with the exception of the TRIP HS, which was diluted 1:100 and analyzed by external standard calibration.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10-22 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Attachments

Appendix A: Target Analyte Historical Trend Data for Cottage Grove Monitoring Wells MW07, MW12, MW13, MW14R, MW16, MW101, MW104, MW105, MW108, MW110, PW09, and PW10.

8 Signatures



Digitally signed by Susan T. Wolf
DN: c=US, s=MN, l=St. Paul, ou=3M
Environmental Laboratory - authenticated by
LRA, email=stwolf@mmm.com, o=3M,
cn=Susan T. Wolf
Reason: I have reviewed this document
Date: 2015.01.02 14:42:01 -06'00'

Susan T. Wolf, 3M Principal Analytical Investigator



Digitally signed by William K. Reagen
DN: c=US, s=MN, l=St. Paul, ou=Laboratory
Director, ou=3M Environmental Laboratory -
authenticated by LRA,
email=wkreagen@mmm.com, o=3M, cn=William
K. Reagen
Reason: I am approving this document
Date: 2015.01.05 06:52:59 -05'00'

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.



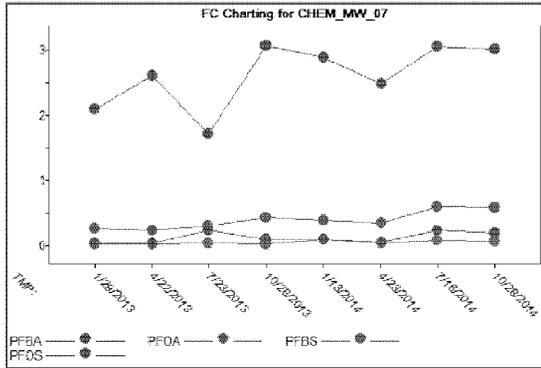
Digitally signed by Tanya K. Rude
DN: c=US, s=MN, l=St. Paul, ou=3M Environmental
Laboratory - authenticated by LRA,
email=trude@mmm.com, o=3M, cn=Tanya K. Rude
Reason: I agree to the terms defined by the
placement of my signature on this document
Date: 2015.01.02 14:49:46 -05'00'

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M Environmental Laboratory.

Appendix A: Target Analyte Historical Trend Data

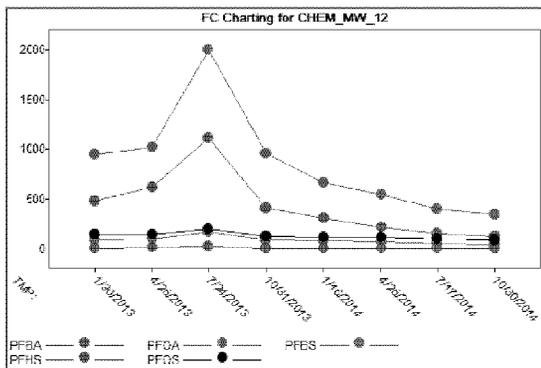
Cottage Grove MW07 – units are ng/mL



MW07	1/29/2013	4/22/2013	7/23/2013	10/28/2013	1/13/2014	4/23/2014	7/16/2014	10/28/2014
PFBA	2.09	2.61	1.71	3.07	2.88	2.48	3.05	3.01
PFOA	0.259	0.240	0.303	0.428	0.390	0.344	0.602	0.581
PFBS	<0.0250	0.0268	0.0396	0.0291	<0.100	0.0360	0.0762	0.0701
PFOS	0.0439	0.0459	0.241	0.104	0.104	0.0493	0.237	0.191

Samples were below the limit of quantitation for PFHS.

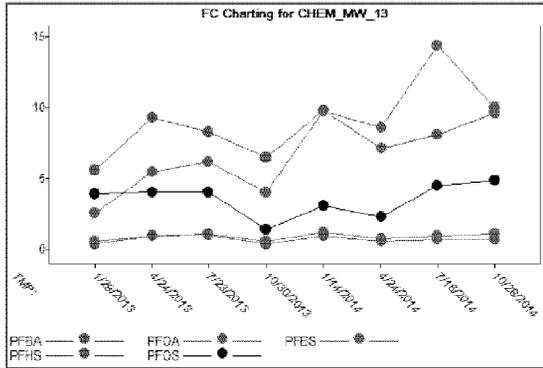
Cottage Grove MW12 – units are ng/mL



MW12	1/30/2013	4/25/2013	7/24/2013	10/31/2013	1/16/2014	4/25/2014	7/17/2014	10/30/2014
PFBA	484	622	1110	408	312	220	154	127
PFOA	946	1020	2000	954	668	548	402	348
PFBS	91.4	105	175	87.7	82.1	68.9	50.4	46.1
PFHS	13.7	16.4	23.9	13.0	13.1	9.82	8.11	7.78
PFOS	143	145	204	131	121	117	96.9	92.5

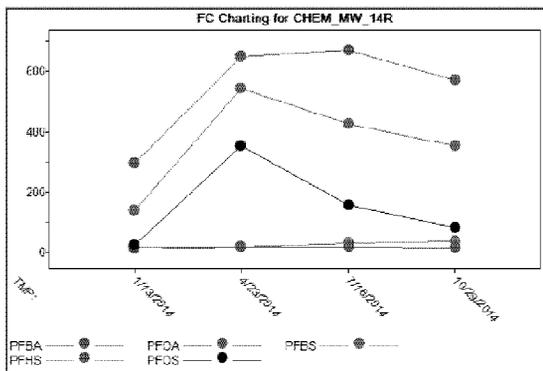
Appendix A continued: Target Analyte Historical Trend Data

Cottage Grove MW13 – units are ng/mL



MW13	1/29/2013	4/24/2013	7/23/2013	10/30/2013	1/14/2014	4/24/2014	7/16/2014	10/28/2014
PFBA	5.60	9.34	8.30	6.48	9.80	7.14	8.08	9.64
PFOA	2.56	5.46	6.17	4.01	9.78	8.63	14.4	10.0
PFBS	0.560	0.972	1.07	0.571	1.20	0.752	0.973	1.09
PFHS	0.396	0.931	1.05	0.366	0.969	0.556	0.713	0.720
PFOS	3.92	4.05	4.02	1.40	3.08	2.33	4.48	4.89

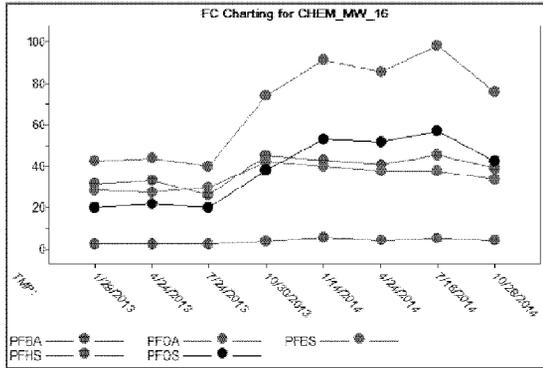
Cottage Grove MW14R – units are ng/mL



MW14R	1/13/2014	4/23/2014	7/16/2014	10/29/2014
PFBA	295	650	671	570
PFOA	139	543	426	353
PFBS	17.5	18.2	29.6	38.1
PFHS	12.3	20.0	20.1	16.6
PFOS	24.9	353	158	82.6

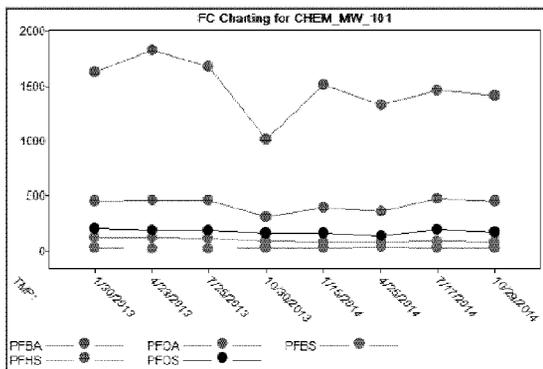
Appendix A continued: Target Analyte Historical Trend Data

Cottage Grove MW16 – units are ng/mL



MW16	1/29/2013	4/24/2013	7/24/2013	10/30/2013	1/14/2014	4/24/2014	7/16/2014	10/28/2014
PFBA	31.5	33.2	26.3	45.0	42.9	40.7	45.7	38.9
PFOA	42.6	43.8	39.9	73.9	91.0	85.7	98.3	75.8
PFBS	28.3	27.5	29.6	42.1	40.0	37.9	37.9	33.9
PFHS	2.54	2.74	2.83	3.95	5.52	4.49	5.46	4.58
PFOS	20.2	21.8	20.1	38.2	53.1	51.9	57.0	42.6

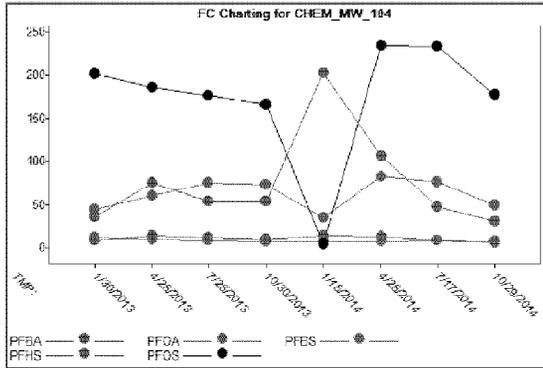
Cottage Grove MW101 – units are ng/mL



MW101	1/30/2013	4/23/2013	7/25/2013	10/30/2013	1/15/2014	4/25/2014	7/17/2014	10/29/2014
PFBA	1630	1830	1680	1020	1510	1330	1460	1410
PFOA	124	121	112	86.7	79.4	76.0	83.9	74.8
PFBS	25.2	19.5	24.0	25.5	25.4	37.8	29.3	29.6
PFHS	455	459	464	314	398	364	480	452
PFOS	206	188	189	158	159	135	193	170

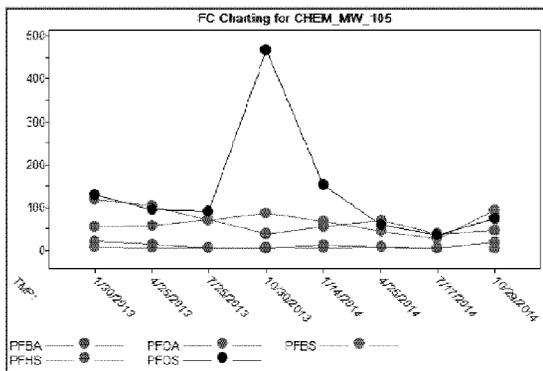
Appendix A continued: Target Analyte Historical Trend Data

Cottage Grove MW104 – units are ng/mL



MW104	1/30/2013	4/25/2013	7/25/2013	10/30/2013	1/15/2014	4/25/2014	7/17/2014	10/29/2014
PFBA	36.3	74.7	54.3	53.6	202	107	47.9	30.9
PFOA	44.8	60.7	74.7	72.5	35.1	82.1	76.5	50.00
PFBS	11.4	9.75	8.16	7.95	7.50	8.10	8.68	7.72
PFHS	9.07	13.4	11.8	10.1	14.2	12.6	8.95	6.24
PFOS	201	185	176	165	4.25	234	233	177

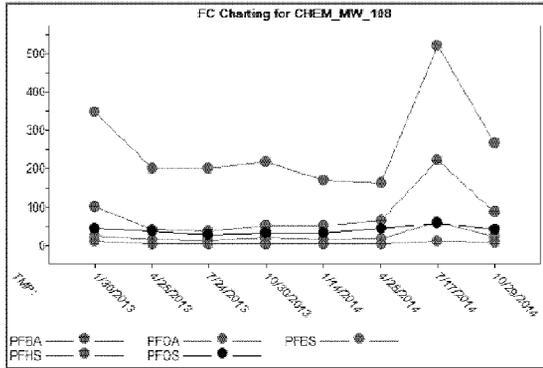
Cottage Grove MW105 – units are ng/mL



1/30/2013	4/25/2013	7/25/2013	10/30/2013	1/14/2014	4/25/2014	7/17/2014	10/29/2014	1/30/2013
54.4	57.0	71.6	38.9	55.6	71.0	39.2	46.3	54.4
119	104	70.6	87.2	68.3	44.3	27.7	92.8	119
8.03	5.72	6.49	4.98	6.15	7.68	4.89	4.01	8.03
21.1	16.0	6.35	6.01	12.6	8.59	6.65	18.9	21.1
129	95.0	92.3	467	152	58.7	36.2	73.5	129

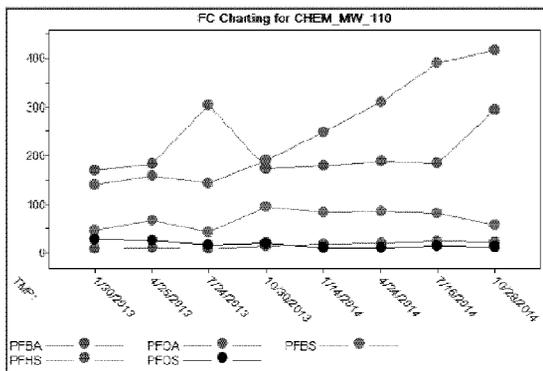
Appendix A continued: Target Analyte Historical Trend Data

Cottage Grove MW108 – units are ng/mL



MW108	1/30/2013	4/25/2013	7/24/2013	10/30/2013	1/14/2014	4/25/2014	7/17/2014	10/29/2014
PFBA	101	42.5	38.0	51.7	51.6	65.4	222	87.0
PFOA	348	201	202	218	169	164	522	268
PFBS	26.5	16.1	14.6	21.6	17.0	17.4	60.7	22.7
PFHS	11.1	4.95	4.94	4.47	4.13	3.58	11.3	8.50
PFOS	45.5	37.3	26.8	33.2	33.5	43.3	58.0	42.0

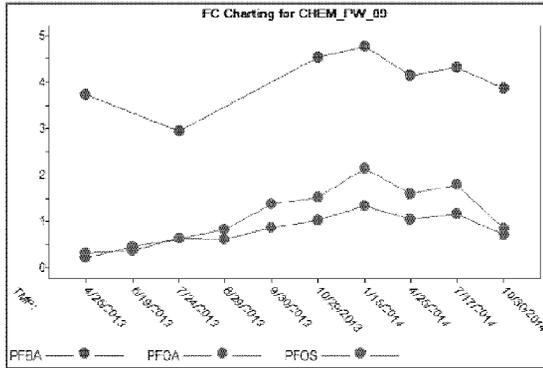
Cottage Grove MW110 – units are ng/mL



MW110	1/30/2013	4/25/2013	7/24/2013	10/30/2013	1/14/2014	4/24/2014	7/16/2014	10/28/2014
PFBA	171	183	304	174	179	189	185	294
PFOA	141	160	144	190	248	310	390	417
PFBS	46.6	66.7	43.8	94.8	85.1	86.1	82.3	57.6
PFHS	10.3	11.8	9.41	15.3	19.4	20.9	24.1	23.5
PFOS	28.6	26.4	17.8	21.1	12.1	12.5	15.4	12.9

Appendix A continued: Target Analyte Historical Trend Data

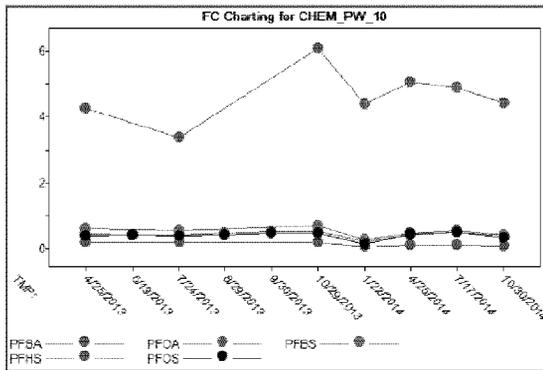
Cottage Grove PW09 – units are ng/mL



PW09	4/25/13	6/19/13	7/24/13	8/29/13	9/30/13	10/29/13	1/15/14	4/25/14	7/17/14	10/30/14
PFBA	3.72	NA	2.95	NA	NA	4.53	4.76	4.14	4.32	3.87
PFOA	0.223	0.462	0.624	0.605	0.873	1.02	1.33	1.05	1.16	0.701
PFOS	0.324	0.376	0.622	0.818	1.38	1.52	2.13	1.60	1.79	0.838

NA = Not Applicable; analyte not requested for the sampling event.

Cottage Grove PW10 – units are ng/mL



PW10	4/25/13	6/19/13	7/24/13	8/29/13	9/30/13	10/29/13	1/15/14	4/25/14	7/17/14	10/30/14
PFBA	4.25	NA	3.37	NA	NA	6.09	4.40	5.07	4.90	4.43
PFOA	0.428	0.415	0.420	0.471	0.528	0.524	0.212	0.421	0.484	0.376
PFBS	0.594	NA	0.540	NA	NA	0.703	0.261	0.471	0.540	0.400
PFHS	0.197	NA	0.185	NA	NA	0.193	0.0485	0.110	0.104	0.0777
PFOS	0.396	0.401	0.380	0.415	0.465	0.458	0.124	0.437	0.503	0.337

NA = Not Applicable; analyte not requested for the sampling event.

All sample bottles include the addition of internal standards and surrogates.

Do not pre-rinse the bottles.

Do not pour out the contents of the bottle.

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-18

Requester: Kotsmith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 10/24/2014
Project Description: Cottage Grove 4th Quarter 2014 Sampling
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-18-001	CGMN-GW-MW07-0- 141028	10/28/14 1040	GW	
ISO11-01-03-18-002	CGMN-GW-MW07-DB- 141028	10/28/14 1040		
ISO11-01-03-18-003	CGMN-GW-MW07-FMS- 141028	10/28/14 1040		
ISO11-01-03-18-004	CGMN-GW-MW12-0- 141030	10/30/14 1055		
ISO11-01-03-18-005	CGMN-GW-MW12-DB- 141030	10/30/14 1055		
ISO11-01-03-18-006	CGMN-GW-MW12-FMS- 141030	10/30/14 1055		
ISO11-01-03-18-007	CGMN-GW-MW13-0- 141028	10/28/14 1210		
ISO11-01-03-18-008	CGMN-GW-MW13-DB- 141028	10/28/14 1210		Slight overfill
ISO11-01-03-18-009	CGMN-GW-MW13-FMS- 141028	10/28/14 1210		
ISO11-01-03-18-010	CGMN-GW-MW14R-0- 141029	10/29/14 1115		
ISO11-01-03-18-011	CGMN-GW-MW14R-DB- 141029	10/29/14 1115		
ISO11-01-03-18-012	CGMN-GW-MW14R-FMS- 141029	10/29/14 1115		
ISO11-01-03-18-013	CGMN-GW-MW16-0- 141028	10/28/14 1350		
ISO11-01-03-18-014	CGMN-GW-MW16-DB- 141028	10/28/14 1350	GW	

Sample Condition Upon Receipt: Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): John Hunter Collector's signature: *John Hunter*

Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
<i>Alex West</i>						

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-18 (cont.)

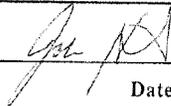
Requester: Kotsmith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 10/24/2014
Project Description: Cottage Grove 4th Quarter 2014 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO11-01-03-18-015	CGMN-GW-MW16-FMS- 141028	10/28/14 1350	GW	
ISO11-01-03-18-016	CGMN-GW-MW101-0- 141029	10/29/14 1520		
ISO11-01-03-18-017	CGMN-GW-MW101-DB- 141029	10/29/14 1520		
ISO11-01-03-18-018	CGMN-GW-MW101-FMS- 141029	10/29/14 1520		
ISO11-01-03-18-019	CGMN-GW-MW104-0- 141029	10/29/14 1355		
ISO11-01-03-18-020	CGMN-GW-MW104-DB- 141029	10/29/14 1355		
ISO11-01-03-18-021	CGMN-GW-MW104-FMS- 141029	10/29/14 1355		
ISO11-01-03-18-022	CGMN-GW-MW105-0- 141029	10/29/14 1305		
ISO11-01-03-18-023	CGMN-GW-MW105-DB- 141029	10/29/14 1305		
ISO11-01-03-18-024	CGMN-GW-MW105-FMS- 141029	10/29/14 1305		
ISO11-01-03-18-025	CGMN-GW-MW108-0- 141029	10/29/14 1210		
ISO11-01-03-18-026	CGMN-GW-MW108-DB- 141029	10/29/14 1210		
ISO11-01-03-18-027	CGMN-GW-MW108-FMS- 141029	10/29/14 1210		
ISO11-01-03-18-028	CGMN-GW-MW110-0- 141028	10/28/14 1435		
ISO11-01-03-18-029	CGMN-GW-MW110-DB- 141028	10/28/14 1435		
ISO11-01-03-18-030	CGMN-GW-MW110-FMS- 141028	10/28/14 1435		
ISO11-01-03-18-031	CGMN-GW-PW09-0- 141030	10/30/14 1025	GW	

Sample Condition Upon Receipt: Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): John Hunter

Collector's signature: 

Relinquished by: _____ Date _____ Time _____ Shipped Via _____

Received by: _____ Date _____ Time _____

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

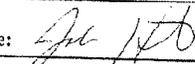
Project: ISO11-01-03-18 (cont.)

Requester: Kotsmith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 10/24/2014
Project Description: Cottage Grove 4th Quarter 2014 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO11-01-03-18-032	CGMN-GW-PW09-DB- 141030	10/30/14 1025	6W	
ISO11-01-03-18-033	CGMN-GW-PW09-FMS- 141030	10/30/14 1025		
ISO11-01-03-18-034	CGMN-GW-PW10-0- 141030	10/30/14 1010		
ISO11-01-03-18-035	CGMN-GW-PW10-DB- 141030	10/30/14 1010		
ISO11-01-03-18-036	CGMN-GW-PW10-FMS- 141030	10/30/14 1010		
ISO11-01-03-18-037	CGMN-GW-RB M61163 -0- 141029	10/29/14 1105		
ISO11-01-03-18-038	CGMN-GW-TRIP-0- 141028	10/28/14 0930		
ISO11-01-03-18-039	CGMN-GW-TRIP-LS- 141028	10/28/14 0930	↓	
ISO11-01-03-18-040	CGMN-GW-TRIP-HS- 141028	10/28/14 0930	6W	

Sample Condition Upon Receipt: Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

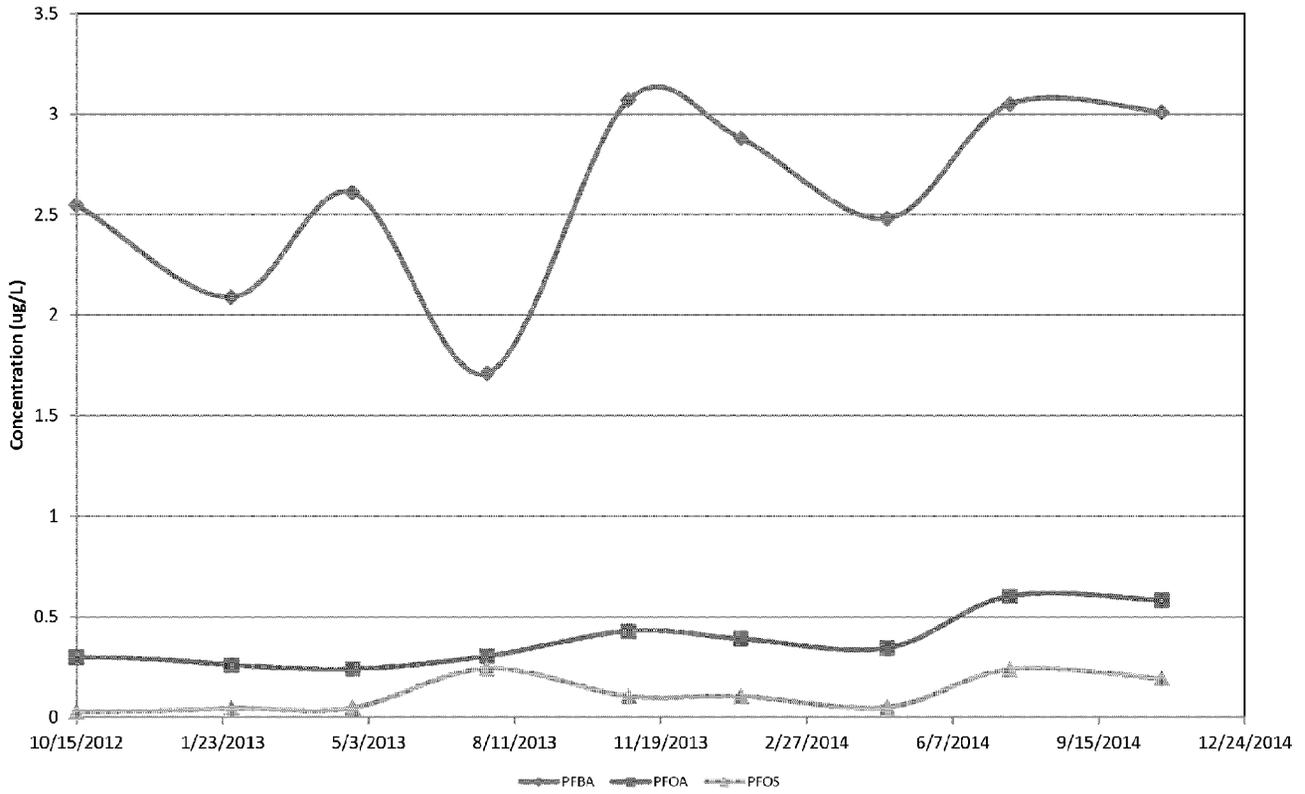
Collected by (print): <u>John Hunter</u>	Collector's signature: 
Relinquished by:	Received by:
Date	Date
Time	Time
Shipped Via	
<u>4</u>	



ATTACHMENT E
PFBA, PFOA AND PFOS TREND GRAPHS

BACKGROUND LOCATION

Monitoring Well MW-07
(Background)
10/2012 - 10/2014
Cottage Grove, MN Site



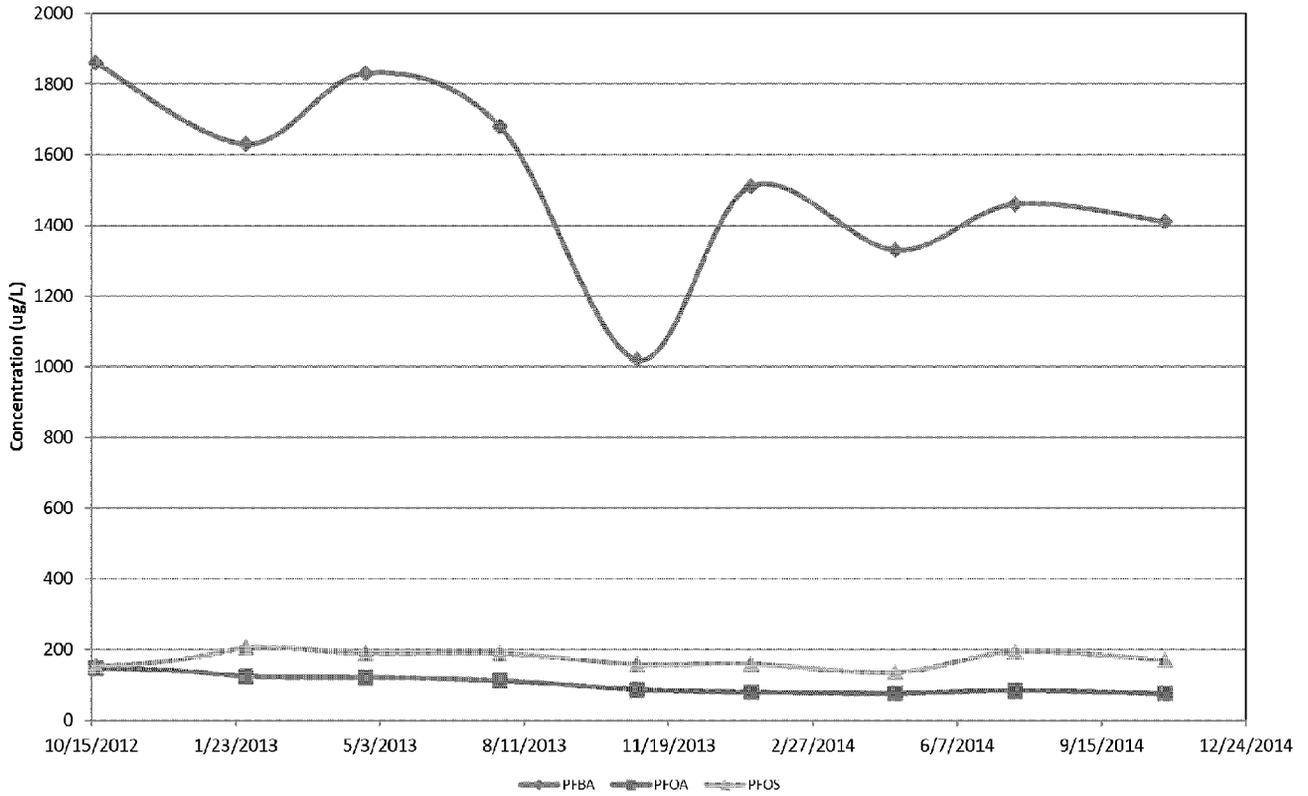
CGMN All FC data Crosstab-thru-2014-07-17(ISO-14).xlsx; MW07

3M_MN01596217

D1/D2 AREA



Monitoring Well MW-101
(D1/D2 Area)
10/2012 - 10/2014
Cottage Grove, MN Site

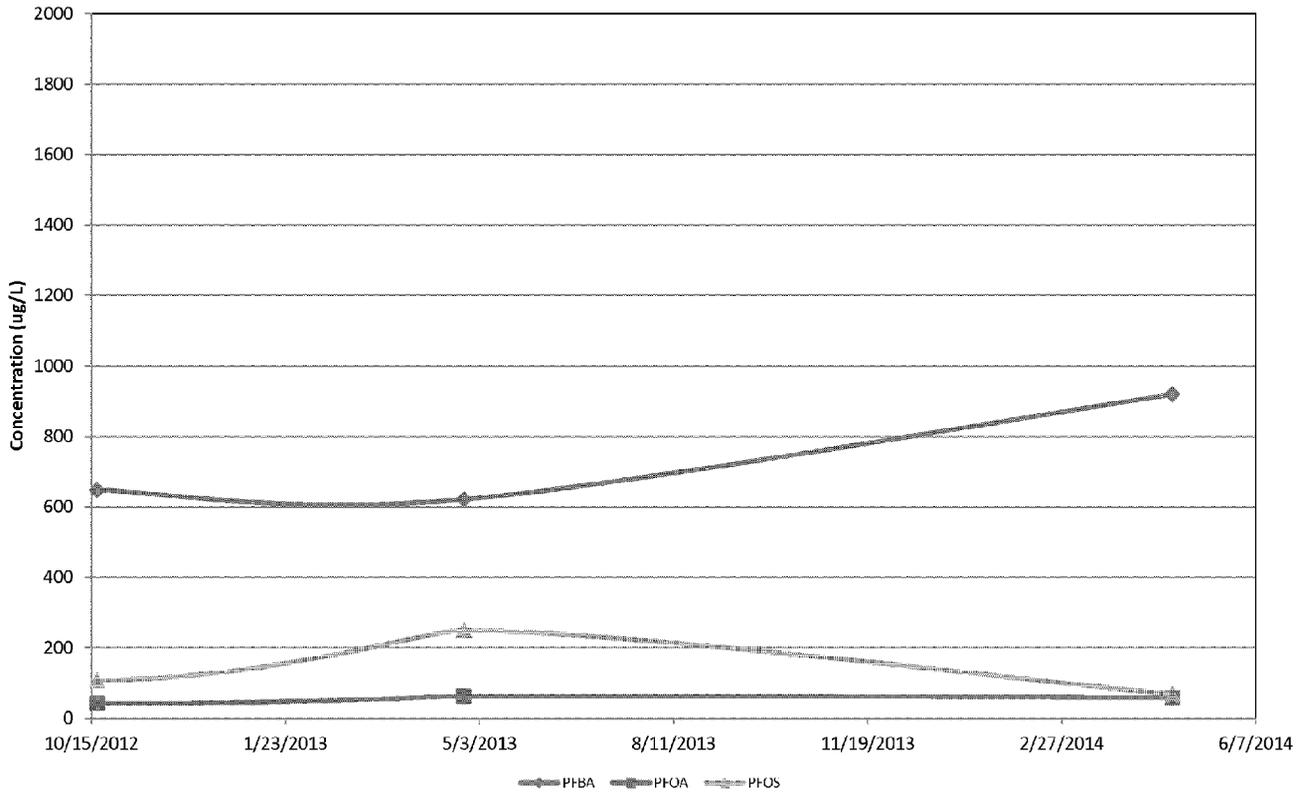


CGMN All FC data Crosstab thru 2014-07-17(ISO-14)(2).xlsx; MW101

3M_MN01596219



Monitoring Well MW-102
(D1/D2 Area)
10/2012 - 4/2014
Cottage Grove, MN Site

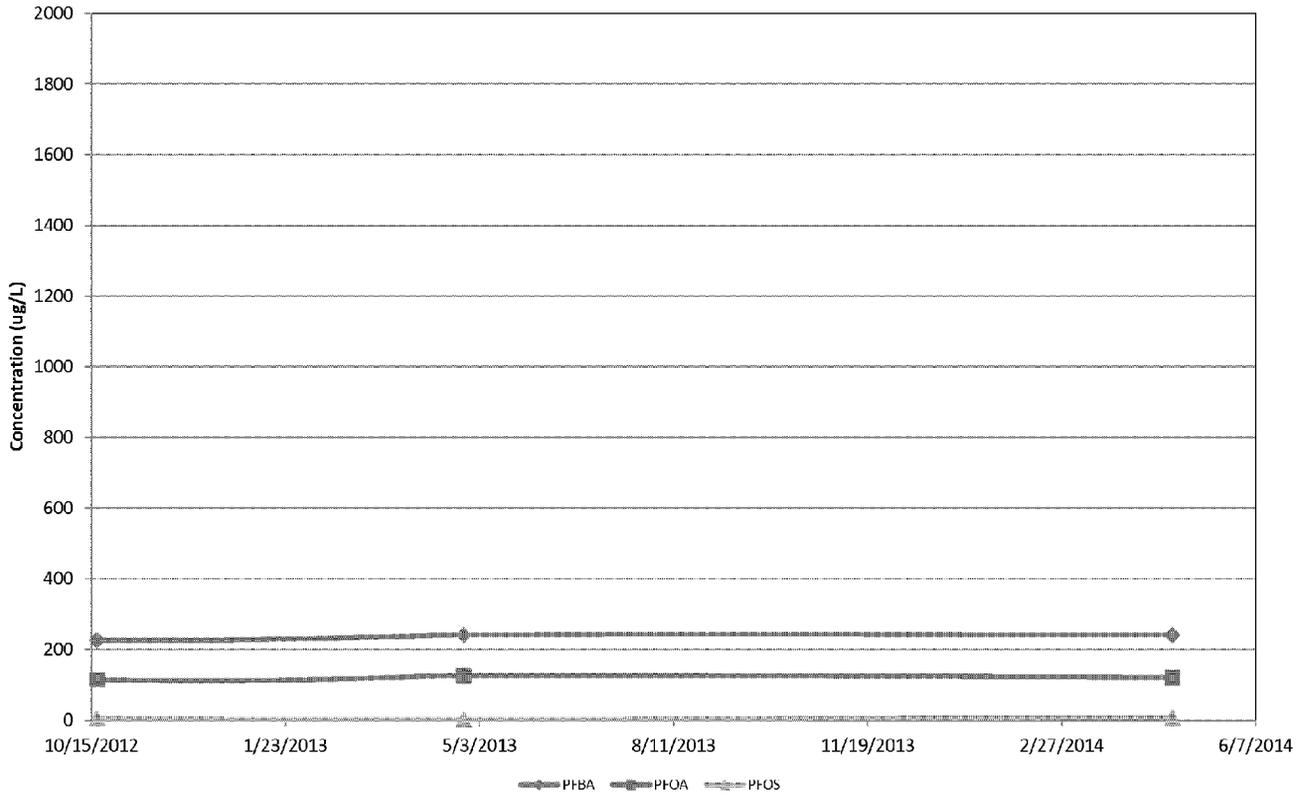


CGMN All FC data Crosstab thru 2014-07-17(ISO-14)(2).xlsx; MW102

3M_MN01596220



Monitoring Well MW-103
(D1/D2 Area)
10/2012 - 4/2014
Cottage Grove, MN Site

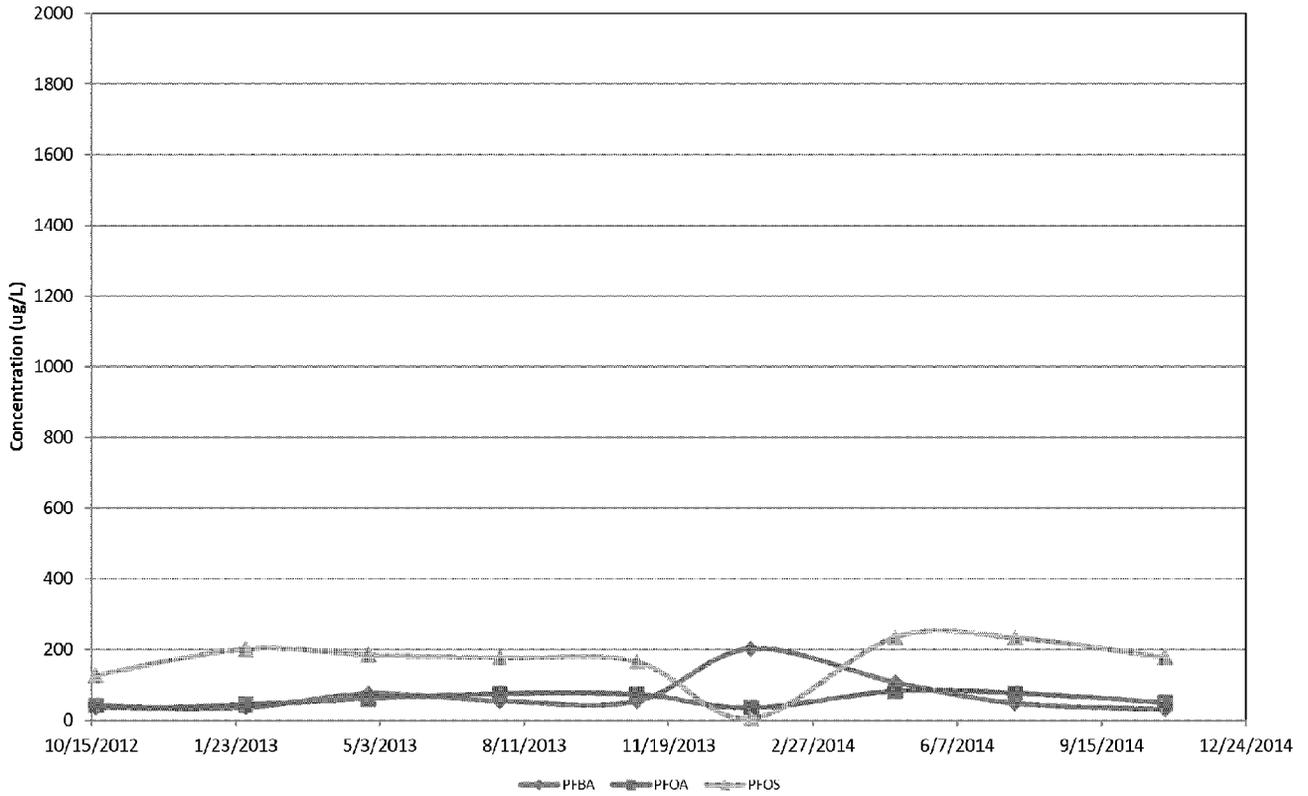


CGMN All FC data Crosstab thru 2014-07-17(ISO-14)(2).xlsx; MW103

3M_MN01596221



Monitoring Well MW-104
(D1/D2 Area)
10/2012 - 10/2014
Cottage Grove, MN Site



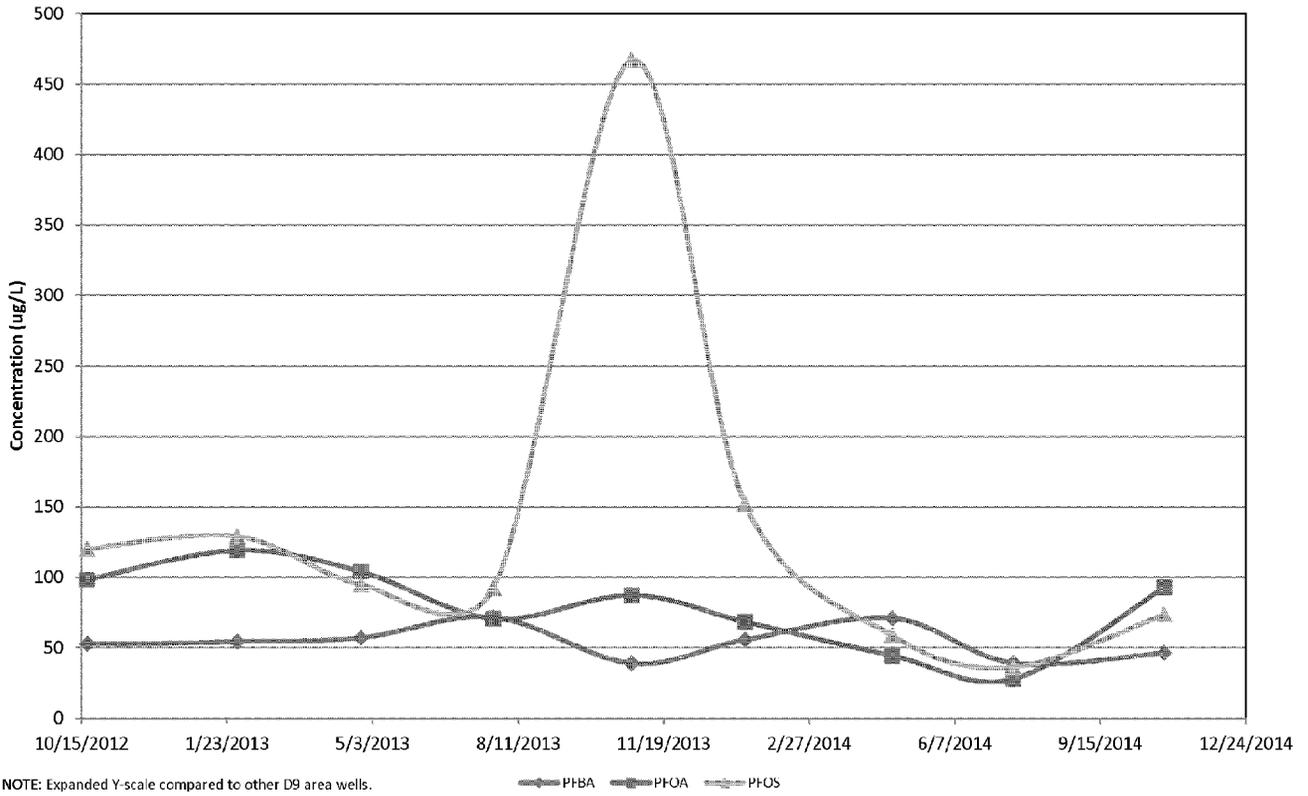
CGMN All FC data Crosstab thru 2014-07-17(ISO-14)(2).xlsx; MW104

3M_MN01596222

D9 AREA



Monitoring Well MW-105
(D9 Area)
10/2012 - 10/2014
Cottage Grove, MN Site



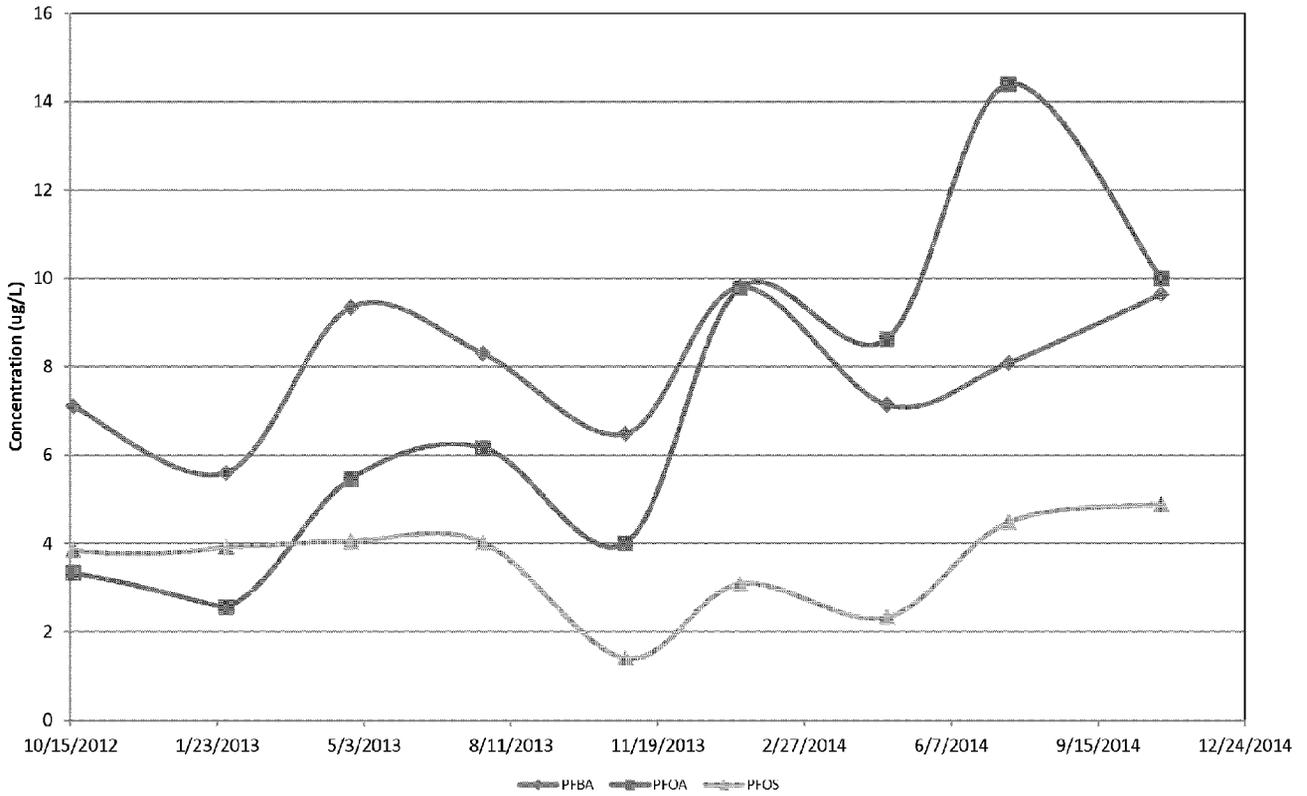
NOTE: Expanded Y-scale compared to other D9 area wells.

CGMN All FC data Crosstab thru 2014-07-17(ISO-14)(2).xlsx; MW105

3M_MN01596224



Monitoring Well MW-13
(D9 Area)
10/2012 - 10/2014
Cottage Grove, MN Site

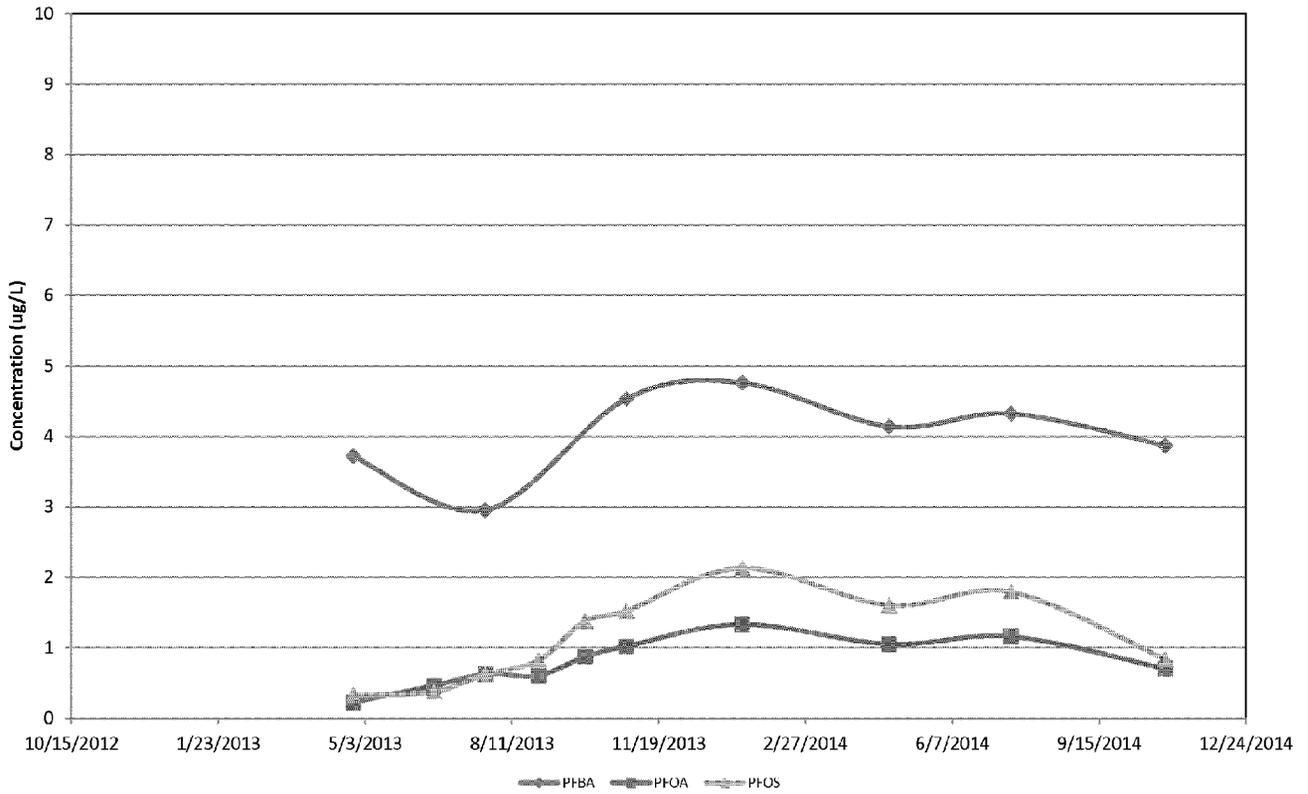


CGMN All FC data Crosstab thru -2014-07-17[ISO-14] (2).xlsx, MW13

3M_MN01596225



Production Well PW-09
(D9 Area)
04/2013 - 10/2014
Cottage Grove, MN Site



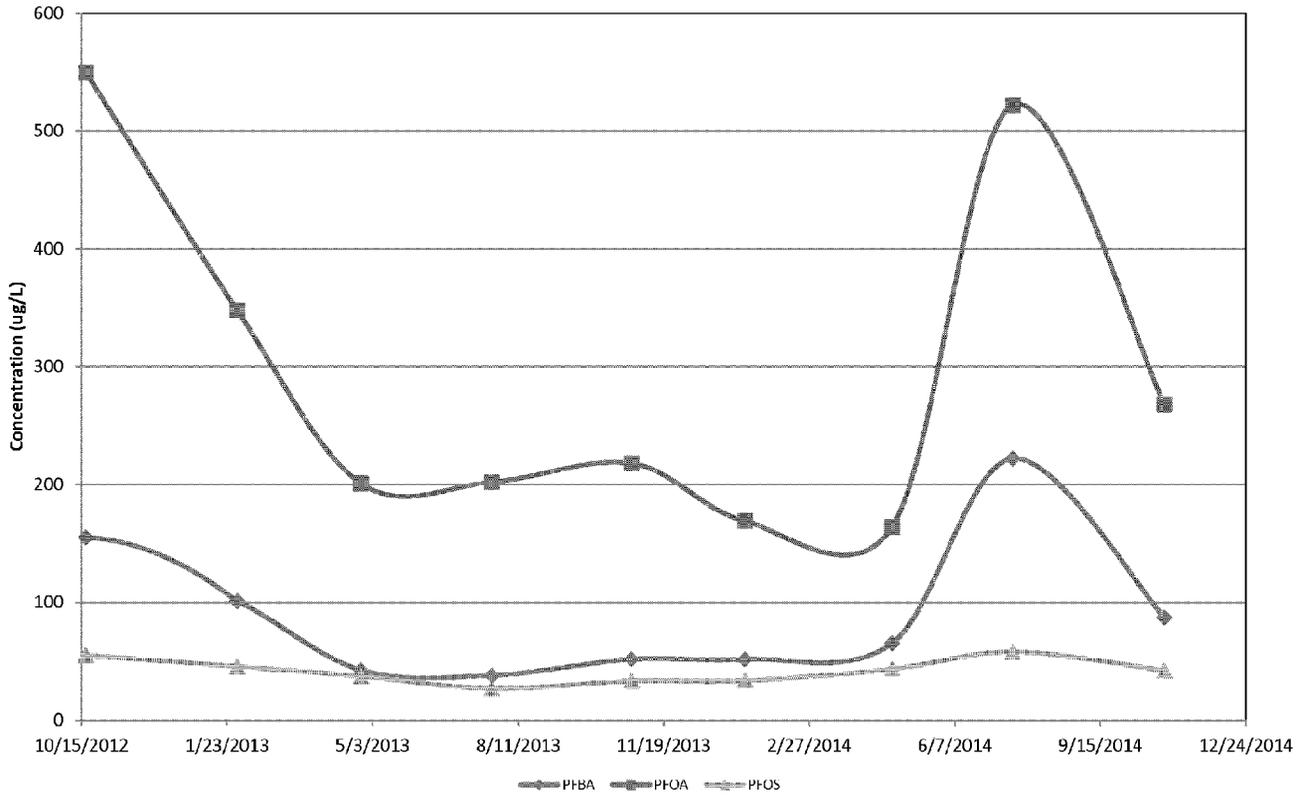
CGMN All FC data Crosstab thru 2014-07-17(ISO-14) (2).xlsx; PW09

3M_MN01596226

WWTP AREA



Monitoring Well MW-108
(WWTP Area)
10/2012 - 10/2014
Cottage Grove, MN Site



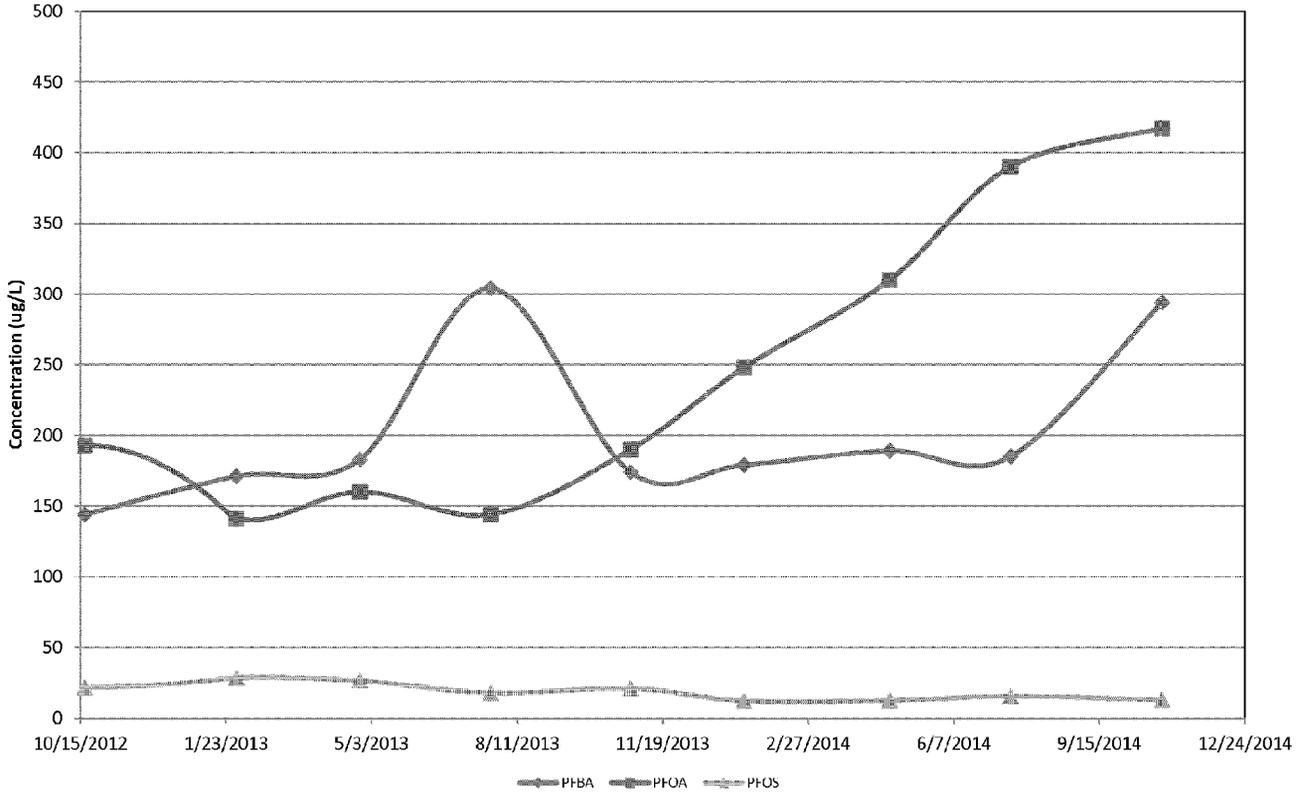
CGMN All FC data Crosstab thru 2014-07-17(ISO-14)(2).xlsx; MW108

3M_MN01596228

D5 AREA



Monitoring Well MW-110
(D5 Area)
10/2012 - 10/2014
Cottage Grove, MN Site

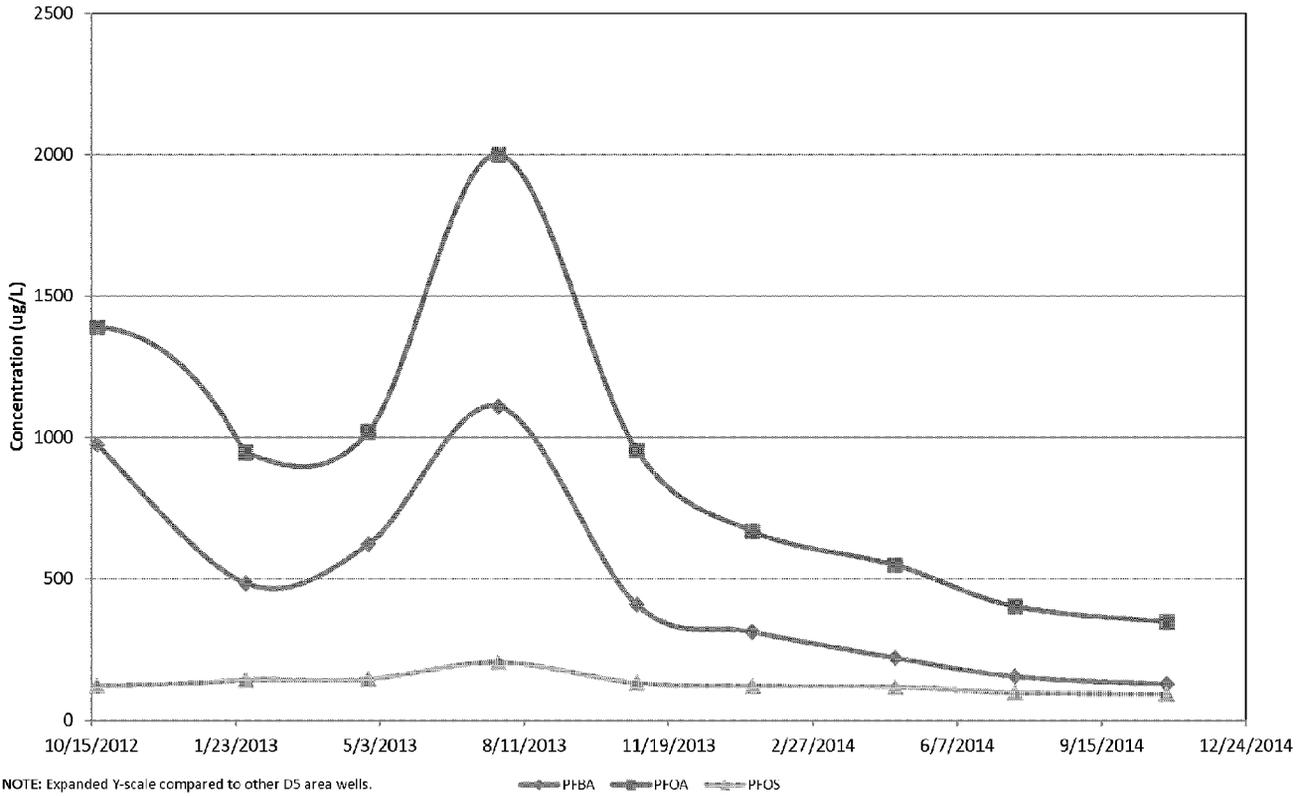


CGMN All FC data Crosstab thru 2014-07-17(ISO-14)(2).xlsx; MW110

3M_MN01596230



Monitoring Well MW-12
(D5 Area)
10/2012 - 10/2014
Cottage Grove, MN Site



NOTE: Expanded Y-scale compared to other D5 area wells.

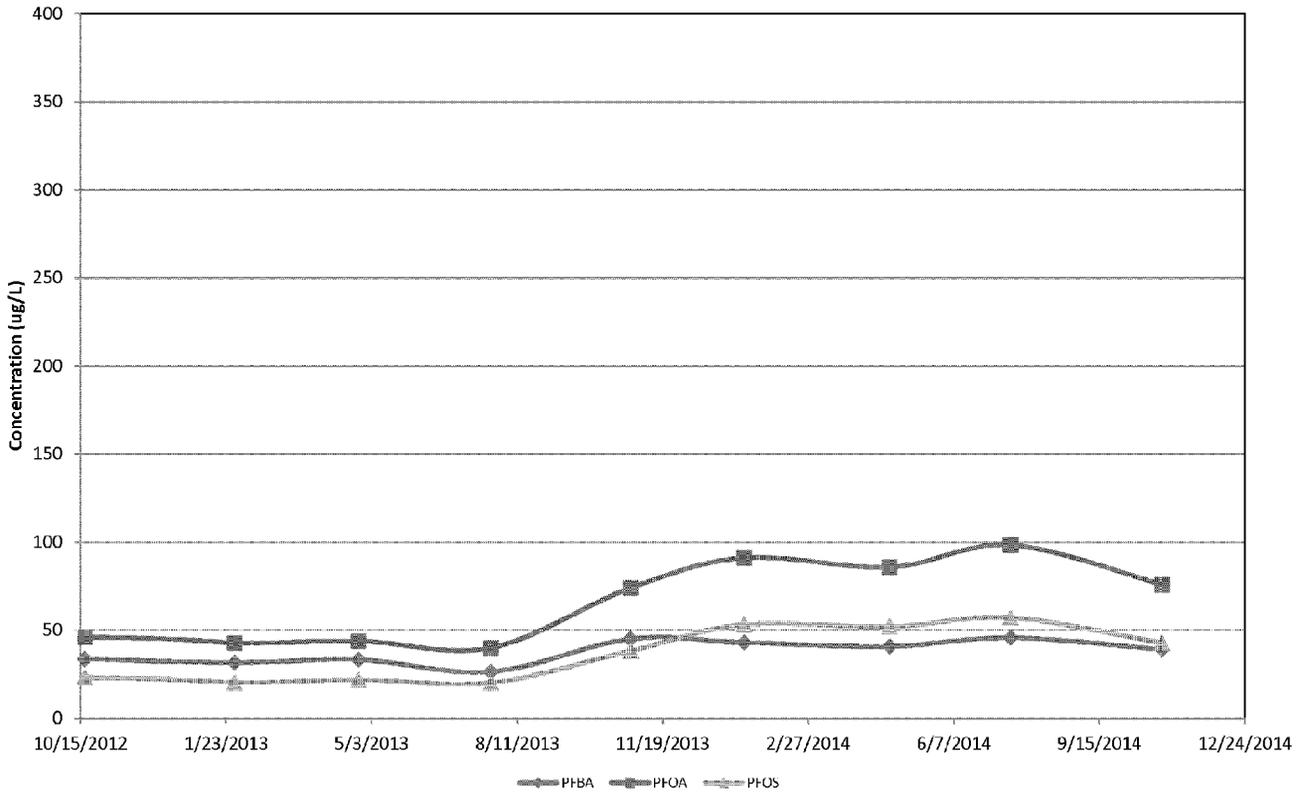
◆ PFBA ■ PFOA ▲ PFOS

CGMN All FC data Crosstab-thru-2014-07-17[ISO-14] (2).xlsx, MW12

3M_MN01596231



Monitoring Well MW-16
(D5 Area)
10/2012 - 10/2014
Cottage Grove, MN Site



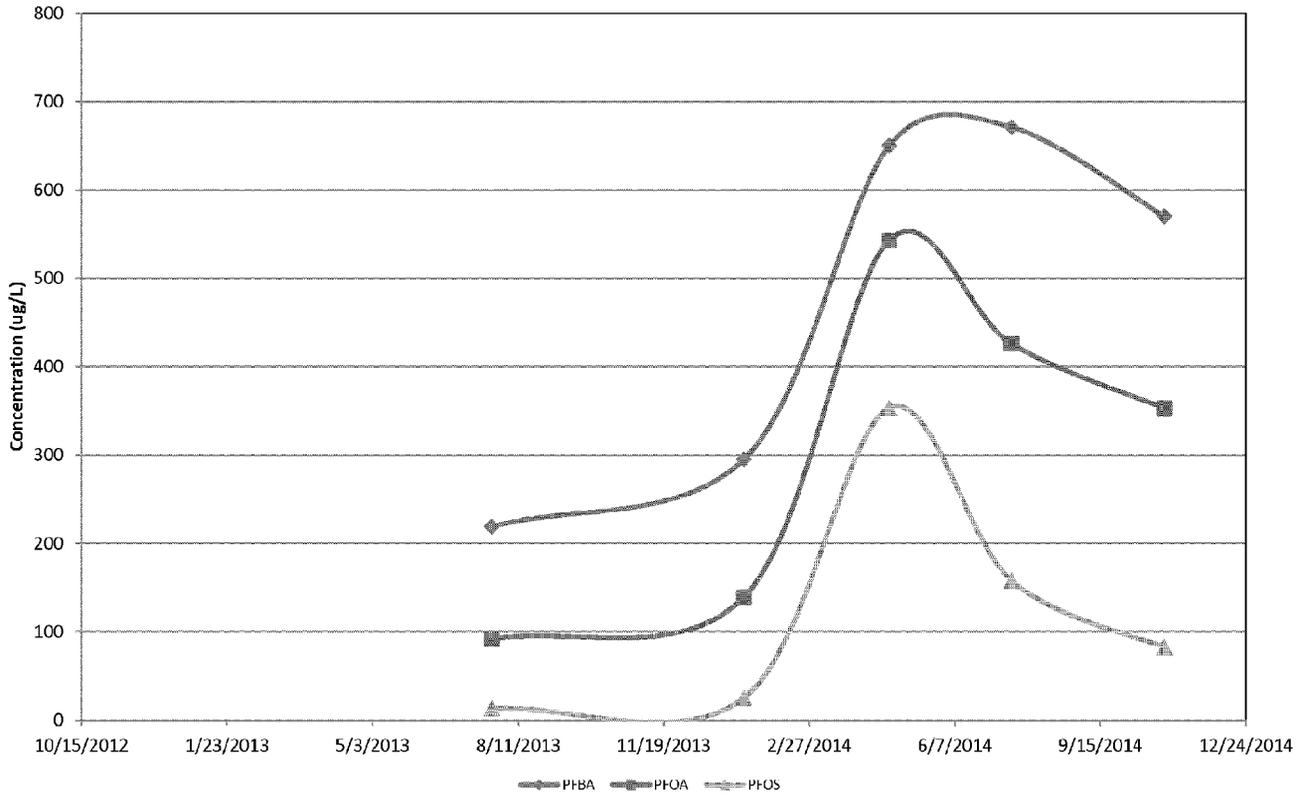
CGMN All FC data Crosstab thru -2014-07-17[ISO-14] (2).xlsx, MW16

3M_MN01596232

D8 AREA



Monitoring Well MW-14R
(D8 Area)
7/2013 - 10/2014
Cottage Grove, MN Site



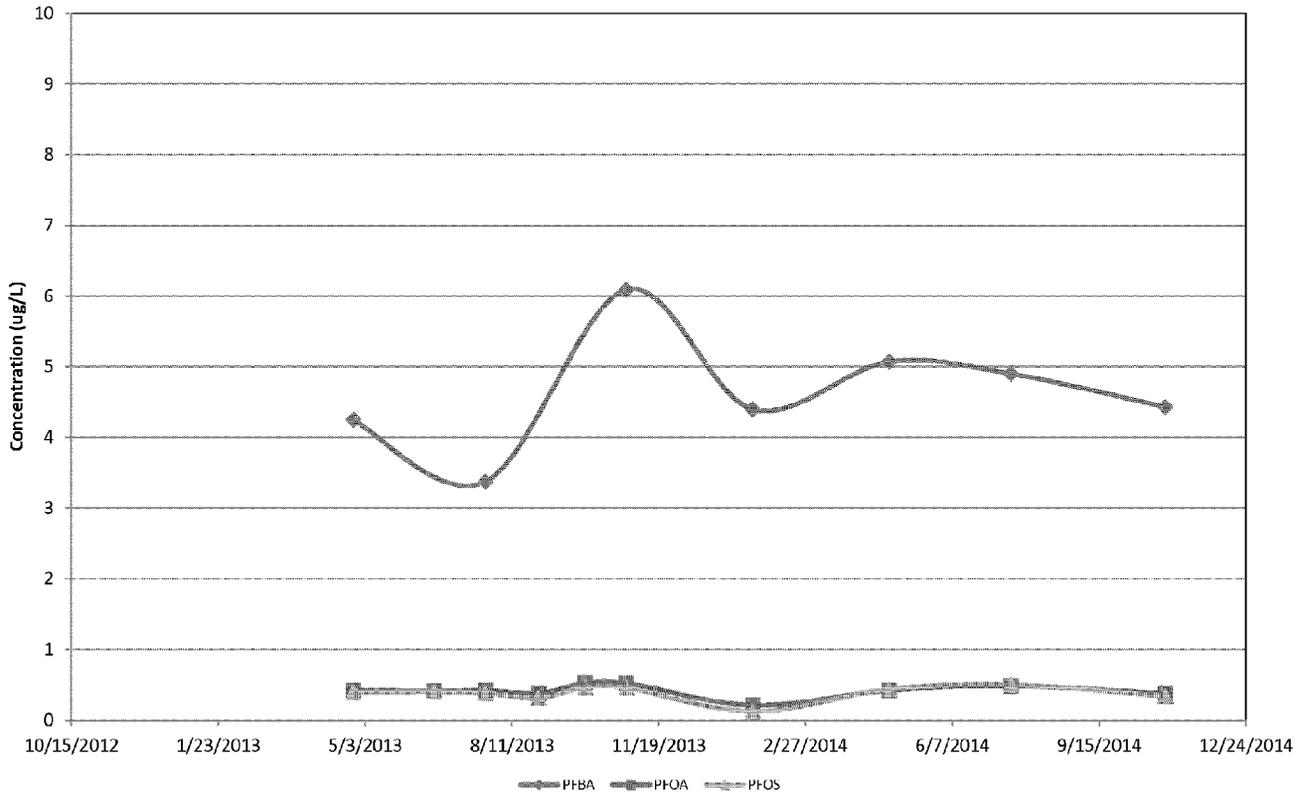
CGMN All FC data Crosstab thru 2014-07-17(ISO-4) [2].xlsx; MW14R

3M_MN01596234

EAST COVE AREA



Production Well PW-10
(East Cove Area)
04/2013 - 7/2014
Cottage Grove, MN Site



CGMN All FC data Crosstab thru 2014-07-17(ISO-14) (2).xlsx; PW10

3M_MN01596236



**ATTACHMENT F
LABORATORY ANALYTICAL PACKAGES FOR PORE WATER
AND SURFACE WATER SAMPLING – SEPTEMBER/OCTOBER 2014**

3M_MN01596237

3630.0288

Final Report

Analysis of Mississippi River Pore Water and Surface Water Samples Near the 3M Cottage Grove Facility – September 2014 Sampling

Laboratory Request Number: ISO11-01-03-17

Method Requirement: 3M Method ETS-8-044.1

Report Date: Date of Last Signature

Testing Laboratory

3M Environmental Health and Safety Operations
Environmental Laboratory
3M Center, Bldg 260-05-N-17
St. Paul, MN 55144

Requester

Jim Kotsmith
3M EHS Operations
3M Building 224-5W-17
Saint Paul, MN 55144-1000
Phone: (651) 737-3635



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with the A2LA Testing Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO11-01-03-17

Analysis of Mississippi River Pore Water and Surface Water Samples Near
the 3M Cottage Grove Facility – September 2014 Sampling

Report Date: Date of Last Signature

1 Summary/Introduction

The 3M Environmental Laboratory prepared and analyzed samples collected by Weston personnel from the Mississippi River near the 3M Cottage Grove facility. Samples were collected September 29 – October 2, 2014. Samples were returned to the 3M Environmental Laboratory on October 3, 2014 and analyzed for perfluorobutanoic acid (PFBA), perfluorooctanoic acid (PFOA), and perfluorooctane sulfonate (PFOS), under 3M Environmental Laboratory project number ISO11-01-03-17.

The 3M Environmental Laboratory prepared sample containers for thirty-two sampling locations; sixteen pore water (interstitial water; IW) locations and sixteen corresponding surface water (SW) locations. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 100 mL. Sample bottle sets for eight of the thirty-two sampling locations consisted of a field sample, field sample duplicate, and a target analyte field matrix spike. All other locations included a field sample and a field sample duplicate only. All samples bottles included the addition of internal standards and surrogate recovery standards (SRSs) [¹³C₃]-PFBA, [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added to the sample containers prior to being sent to the field for sample collection. Sample bottles reserved for target analyte field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection.

Samples were prepared and analyzed according to 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Following sample analysis by internal standard calibration, it was suspected that the internal standards and surrogates were not spiked as intended, since the internal standard and surrogate recovery response was approximately one half of the expected response. This was confirmed with the trip blank matrix spike sample which had recoveries of approximately 200%. Surrogate recovery standards added to the sample containers prior to sample collection were not used to assess sample recovery and the samples were quantitated by external standard calibration. Target analyte laboratory matrix spikes were prepared for those sampling locations that did not include a target analyte field matrix spike.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.

Table 1. Sample Results Summary. ⁽¹⁾

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFOS Concentration (ng/mL)
Surface Water Locations				
ISO11-01-03-17-001	CGMN-SW-MRIW09b-0-141001	<0.0500	0.0332	<0.0232
ISO11-01-03-17-002	CGMN-SW-MRIW09b-DB-141001	<0.0500	0.0274	<0.0232
Average		<0.0500	0.0303	<0.0232
%RPD Sample/Sample Dup		NA	19	NA
ISO11-01-03-17-003	CGMN-SW-MRIW09-0-141001	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-004	CGMN-SW-MRIW09-DB-141001	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA
ISO11-01-03-17-005	CGMN-SW-MRIW09d-0-141001	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-006	CGMN-SW-MRIW09d-DB-141001	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA
ISO11-01-03-17-008	CGMN-SW-MRIW09f-0-141001	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-009	CGMN-SW-MRIW09f-DB-141001	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA
ISO11-01-03-17-010	CGMN-SW-MRIW14b-0-140930	<0.0500	0.0789	<0.0232
ISO11-01-03-17-011	CGMN-SW-MRIW14b-DB-140930	<0.0500	0.0711	<0.0232
Average		<0.0500	0.0750	<0.0232
%RPD Sample/Sample Dup		NA	10	NA
ISO11-01-03-17-012	CGMN-SW-MRIW14-0-140930	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-013	CGMN-SW-MRIW14-DB-140930	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA
ISO11-01-03-17-015	CGMN-SW-MRIW14d-0-130813	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-016	CGMN-SW-MRIW14d-DB-130813	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA
ISO11-01-03-17-017	CGMN-SW-MRIW14f-0-140930	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-018	CGMN-SW-MRIW14f-DB-140930	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA

NA = Not Applicable

- (1) Samples were analyzed by solvent dilution with external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFBA ± 25%, PFOA ± 13%, and PFOS ± 17%.
- (2) The RPD value did not meet method acceptance criteria of ≤20%.
- (3) The field matrix spike sample for location CGMN-IW-MRIW14d did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded to ± 48% for PFBA.

Table 1 continued. Sample Results Summary. ⁽¹⁾

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFOS Concentration (ng/mL)
Surface Water Locations				
ISO11-01-03-17-019	CGMN-SW-MRIW19b-0-140930	0.0808	0.200	0.139
ISO11-01-03-17-020	CGMN-SW-MRIW19b-DB-140930	0.0549	0.205	0.135
Average		0.0679	0.203	0.137
%RPD Sample/Sample Dup		38⁽²⁾	2.5	2.9
ISO11-01-03-17-022	CGMN-SW-MRIW19-0-140930	<0.0500	0.0922	<0.0232
ISO11-01-03-17-023	CGMN-SW-MRIW19-DB-140930	<0.0500	0.0898	<0.0232
Average		<0.0500	0.0910	<0.0232
%RPD Sample/Sample Dup		NA	2.6	NA
ISO11-01-03-17-024	CGMN-SW-MRIW19d-0-140930	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-025	CGMN-SW-MRIW19d-DB-140930	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA
ISO11-01-03-17-026	CGMN-SW-MRIW19f-0-140930	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-027	CGMN-SW-MRIW19f-DB-140930	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA
ISO11-01-03-17-028	CGMN-SW-MRIW25b-0-140930	0.575	0.171	0.150
ISO11-01-03-17-029	CGMN-SW-MRIW25b-DB-140930	0.530	0.174	0.145
Average		0.553	0.173	0.148
%RPD Sample/Sample Dup		8.1	1.7	3.4
ISO11-01-03-17-030	CGMN-SW-MRIW25-0-140930	0.659	0.181	0.128
ISO11-01-03-17-031	CGMN-SW-MRIW25-DB-140930	0.687	0.181	0.126
Average		0.673	0.181	0.127
%RPD Sample/Sample Dup		4.2	0.0	1.6
ISO11-01-03-17-032	CGMN-SW-MRIW25d-0-140930	<0.0500	0.0396	<0.0232
ISO11-01-03-17-033	CGMN-SW-MRIW25d-DB-140930	<0.0500	0.0509	<0.0232
Average		<0.0500	0.0453	<0.0232
%RPD Sample/Sample Dup		NA	25⁽²⁾	NA
ISO11-01-03-17-034	CGMN-SW-MRIW25f-0-140929	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-035	CGMN-SW-MRIW25f-DB-140929	<0.0500	<0.0240	<0.0232
Average		<0.0500	<0.0240	<0.0232
%RPD Sample/Sample Dup		NA	NA	NA

NA = Not Applicable

- (1) Samples were analyzed by solvent dilution with external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFBA \pm 25%, PFOA \pm 13%, and PFOS \pm 17%.
- (2) The RPD value did not meet method acceptance criteria of \leq 20%.
- (3) The field matrix spike sample for location CGMN-IW-MRIW14d did not meet acceptance criteria of $100 \pm 30\%$. The method uncertainty has been expanded to $\pm 48\%$ for PFBA.

Table 1 continued. Sample Results Summary. ⁽¹⁾

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFOS Concentration (ng/mL)
Interstitial Water (Pore Water)				
ISO11-01-03-17-037	CGMN-IW-MRIW09b-0-141002	47.6	8.31	3.00
ISO11-01-03-17-038	CGMN-IW-MRIW09b-DB-141002	44.3	7.88	2.90
Average		46.0	8.10	2.95
%RPD Sample/Sample Dup		7.2	5.3	3.4
ISO11-01-03-17-039	CGMN-IW-MRIW09-0-141002	6.77	1.39	0.392
ISO11-01-03-17-040	CGMN-IW-MRIW09-DB-141002	6.65	1.39	0.374
Average		6.71	1.39	0.383
%RPD Sample/Sample Dup		1.8	0.0	4.7
ISO11-01-03-17-042	CGMN-IW-MRIW09d-0-141001	2.93	0.781	<0.0232
ISO11-01-03-17-043	CGMN-IW-MRIW09d-DB-141001	2.94	0.797	0.0269
Average		2.94	0.789	0.0269
%RPD Sample/Sample Dup		0.34	2.0	NA
ISO11-01-03-17-044	CGMN-IW-MRIW09f-0-141002	15.6	0.523	0.0236
ISO11-01-03-17-045	CGMN-IW-MRIW09f-DB-141002	15.1	0.524	<0.0232
Average		15.4	0.524	0.0236
%RPD Sample/Sample Dup		3.3	0.19	NA
ISO11-01-03-17-046	CGMN-IW-MRIW14b-0-141002	24.4	101	37.1
ISO11-01-03-17-047	CGMN-IW-MRIW14b-DB-141002	24.4	102	37.0
Average		24.4	102	37.1
%RPD Sample/Sample Dup		0.0	0.99	0.27
ISO11-01-03-17-048	CGMN-IW-MRIW14-0-141001	2.29	4.49	0.284
ISO11-01-03-17-049	CGMN-IW-MRIW14-DB-141001	2.03	3.99	0.218
Average		2.16	4.24	0.251
%RPD Sample/Sample Dup		12	12	26⁽²⁾
ISO11-01-03-17-050	CGMN-IW-MRIW14d-0-141001	0.307	0.0780	<0.0232
ISO11-01-03-17-051	CGMN-IW-MRIW14d-DB-141001	1.40	0.354	0.0952
Average		0.854⁽³⁾	0.216	0.0952
%RPD Sample/Sample Dup		128⁽²⁾	128⁽²⁾	NA
ISO11-01-03-17-053	CGMN-IW-MRIW14f-0-141002	72.0	3.96	0.0477
ISO11-01-03-17-054	CGMN-IW-MRIW14f-DB-141002	80.2	4.42	<0.0232
Average		76.1	4.19	0.0477
%RPD Sample/Sample Dup		11	11	NA

NA = Not Applicable

- (1) Samples were analyzed by solvent dilution with external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFBA ± 25%, PFOA ± 13%, and PFOS ± 17%.
- (2) The RPD value did not meet method acceptance criteria of ≤20%.
- (3) The field matrix spike sample for location CGMN-IW-MRIW14d did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded to ± 48% for PFBA.

Table 1 continued. Sample Results Summary. ⁽¹⁾

3M LIMS ID	Sample Description	PFBA Concentration (ng/mL)	PFOA Concentration (ng/mL)	PFOS Concentration (ng/mL)
Interstitial Water (Pore Water)				
ISO11-01-03-17-055	CGMN-IW-MRIW19b-0-141001	69.0	149	33.1
ISO11-01-03-17-056	CGMN-IW-MRIW19b-DB-141001	69.4	144	29.2
Average		69.2	147	31.2
%RPD Sample/Sample Dup		0.58	3.4	13
ISO11-01-03-17-057	CGMN-IW-MRIW19-0-141001	62.6	9.20	91.9
ISO11-01-03-17-058	CGMN-IW-MRIW19-DB-141001	62.5	9.10	92.7
Average		62.6	9.15	92.3
%RPD Sample/Sample Dup		0.16	1.1	0.87
ISO11-01-03-17-059	CGMN-IW-MRIW19d-0-141001	13.3	0.112	<0.0232
ISO11-01-03-17-060	CGMN-IW-MRIW19d-DB-141001	13.5	0.116	<0.0232
Average		13.4	0.114	<0.0232
%RPD Sample/Sample Dup		1.5	3.5	NA
ISO11-01-03-17-062	CGMN-IW-MRIW19f-0-141002	50.0	223	0.896
ISO11-01-03-17-063	CGMN-IW-MRIW19f-DB-141002	53.0	239	0.930
Average		51.5	231	0.913
%RPD Sample/Sample Dup		5.8	6.9	3.7
ISO11-01-03-17-064	CGMN-IW-MRIW25b-0-140930	54.6	380	1300
ISO11-01-03-17-065	CGMN-IW-MRIW25b-DB-140930	53.2	368	1290
Average		53.9	374	1300
%RPD Sample/Sample Dup		2.6	3.2	0.77
ISO11-01-03-17-066	CGMN-IW-MRIW25-0-140930	59.1	23.8	16.3
ISO11-01-03-17-067	CGMN-IW-MRIW25-DB-140930	58.6	23.3	16.1
Average		58.9	23.6	16.2
%RPD Sample/Sample Dup		0.85	2.1	1.2
ISO11-01-03-17-068	CGMN-IW-MRIW25d-0-141001	11.0	37.4	0.0962
ISO11-01-03-17-069	CGMN-IW-MRIW25d-DB-141001	10.5	35.9	0.0920
Average		10.8	36.7	0.0941
%RPD Sample/Sample Dup		4.7	4.1	4.5
ISO11-01-03-17-070	CGMN-IW-MRIW25f-0-141001	16.5	10.2	7.21
ISO11-01-03-17-071	CGMN-IW-MRIW25f-DB-141001	15.9	9.83	6.09
Average		16.2	10.0	6.65
%RPD Sample/Sample Dup		3.7	3.7	17
ISO11-01-03-17-073	CGMN-SW-MRIW09-RB01-141001	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-074	CGMN-IW-MRIW09-RB02-141001	<0.0500	<0.0240	<0.0232
ISO11-01-03-17-075	CGMN-IW-TRIP-0-140929	<0.0500	<0.0240	<0.0232

NA = Not Applicable

- (1) Samples were analyzed by solvent dilution with external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFBA ± 25%, PFOA ± 13%, and PFOS ± 17%.
- (2) The RPD value did not meet method acceptance criteria of ≤20%.
- (3) The field matrix spike sample for location CGMN-IW-MRIW14d did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded to ± 48% for PFBA.

2 Method Summary

2.1 Methods

Analysis for PFBA, PFOA, and PFOS was completed following 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS".

Table 2. Target Analytes.

Target Analytes	Acronym	Reference Material Structure
Perfluorobutanoate (C4 Acid)	PFBA	Linear
Perfluorooctanoate (C8 Acid)	PFOA	Linear + Branched
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched

2.2 Sample Collection

Pore water (interstitial water) and surface water samples were collected in 125 mL Nalgene™ (high-density polyethylene) bottles prepared at the 3M Environmental Laboratory. A set of laboratory prepared Trip Blank and Trip Blank field matrix spikes were sent with the set of collection bottles. Sample bottles were received by the laboratory on October 3, 2014. Samples were stored refrigerated at the laboratory after receipt.

2.3 Sample Preparation

Samples with a dilution factor of 2 were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol and analyzed by external standard calibration. Samples that required further dilution were initially prepared using a dilution factor of 10 by removing a 1 mL aliquot of the well mixed sample and diluting it with 9 mL of laboratory reagent water. Then a 0.4 mL aliquot of the 1:10 diluted sample was diluted again with 0.4 mL of methanol. In addition, target analyte lab matrix spike samples were prepared for samples that did not contain field matrix spikes. The laboratory matrix spike levels are discussed in section 3.9 of the report.

Sampling locations CGMN-IW-MRIW19f and CGMN-IW-MRI25b required further dilution. These samples were prepared by removing a 0.1 mL aliquot of the well mixed sample and diluting it with 9.8 mL of methanol. To each of the diluted samples, a 0.1 mL aliquot of a solution containing SRSs was added at a nominal concentration of 1 ng/mL (dilution factor of 100). The samples were analyzed by external standard calibration.

During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples.

2.4 Analysis

All samples and quality control samples were analyzed for PFBA, PFOA, and PFOS using high performance liquid chromatography/ tandem mass spectrometry (HPLC/MS/MS). Detailed instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the raw data hard copies placed in the final data packet, and are briefly described below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk
Analysis Dates	10/7/14, 10/13/14, 10/15/14, 10/20/14, 10/23/14, 10/24/14
Analytical Method	ETS-8-044.1
Liquid Chromatograph	Agilent 1260
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 μ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 μ
Injection Volume	2, 5, or 10 μ L
Mass Spectrometer	Applied Biosystems API 5500
Ion Source	Turbo Spray
Electrode	Turbo ion electrode
Polarity	Negative
Software	Analyst 1.6.2

Table 4. Liquid Chromatography Conditions.

Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
ETS-8-044.1				
0	0.00	750	90.0	10.0
1	0.50	750	90.0	10.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	90.0	10.0
9	19.0	750	90.0	10.0

Table 5. Mass Transitions.

Analyte	Mass Transition Q1/Q3	Internal Standard ⁽¹⁾	Mass Transition Q1/Q3
PFBA	213/169	[¹³ C ₄]-PFBA	217/172
PFOA	413/369	[¹³ C ₆]-PFOA	421/376
	413/219		
	413/169		
	499/99		
PFOS	499/80	[¹³ C ₆]-PFOS	507/80
	499/130		
	216/172		
[¹³ C ₃]-PFBA	216/172	[¹³ C ₄]-PFBA	217/172
[¹³ C ₄]-PFOA	417/372	[¹³ C ₆]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₆]-PFOS	507/80

Dwell time was 20 or 50 msec for each transition. The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standard was included in the acquisition method on 10/7/14 and 10/13/14; however, internal standard calibration was not used for the quantitation of the analyzed samples.

3 Analytical Results

3.1 Calibration

10/7/13, 10/13/14, 10/20/14, 10/23/14, & 10/24/14 Analysis - Samples were quantitated for all analytes against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 methanol: laboratory reagent water. Between nine and fourteen standards ranging from 0.02 ng/mL to 5 ng/mL, 10 ng/mL, or 100 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

10/15/14 Analysis - Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution into 50 mL of 90:10 methanol: laboratory Milli-Q™ water. Nine standards ranging from 0.02 ng/mL to 10 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

For both analyses, each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes in each analysis.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria of less than or equal to 5% relative standard deviation (RSD) for peak area/ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ for each analysis is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts/ratio are at least twice those of the appropriate blanks. The LOQ for all analytes can be found in Table 6.

Table 6. Limit of Quantitation (LOQ).

Analyte	10/7/14 Analysis LOQ, ng/mL ⁽¹⁾	10/13/14 Analysis LOQ, ng/mL ⁽¹⁾	10/15/14 Analysis LOQ, ng/mL ⁽²⁾	10/20/14 Analysis LOQ, ng/mL ⁽¹⁾	10/23/14 Analysis LOQ, ng/mL ⁽¹⁾	10/24/14 Analysis LOQ, ng/mL ⁽¹⁾
PFBA	0.0500	0.0250	NA	0.0500	NA	NA
PFOA	0.0240	0.0240	0.0192	0.0480	NA	0.0240
PFOS	0.0232	0.0232	0.0185	0.0232	0.0232	NA

NA = Not Applicable

(1) A dilution factor of 2 was applied to the LOQ.

(2) A dilution factor was not applied to the LOQ.

3.4 Continuing Calibration

During the course of each analytical sequence, continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported sample results were bracketed by CCVs that met method criteria of 100% ± 25%.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: solvent blanks (procedural blanks), field/trip blanks, and equipment rinseate blanks for the water samples. Each blank result was reviewed and used to evaluate method performance. Procedural blank results were reviewed according to the method and used to evaluate method performance to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared and analyzed in triplicate. The LCS samples were prepared by spiking known amounts of the analyte into 10 mL of laboratory reagent water or 1 mL of laboratory Milli-Q™ water to produce the desired concentration. The LCSs were diluted with methanol in the same manner as the samples. All LCS results were used to determine overall method uncertainty in Section 3.7.

The method acceptance criteria states that the average recovery of LCS be 100% ± 20% with a RSD ≤20%, when evaluated independently at each concentration level. All LCSs met acceptance criteria with the following exceptions:

10/7/14 Analysis: The low set of LCSs for PFBA had a RSD value of 41%. A method deviation is included with the raw data.

The following calculations were used to generate data in Table 7 for laboratory control spikes.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%$$

$$\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%$$

Table 7. Laboratory Control Spike Recovery.

ETS-8-044.1 External Calibration Analyzed 10/7/14	PFBA			PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141007-1	0.198	0.178	89.8	0.190	0.184	96.8
LCS-141007-2	0.198	0.331	167	0.190	0.202	106
LCS-141007-3	0.198	0.166	83.6	0.190	0.193	101
Average ± %RSD	113% ± 41%⁽¹⁾			101% ± 4.5%		
LCS-141007-4	19.8	19.4	98.1	19.0	19.2	101
LCS-141007-5	19.8	19.4	98.2	19.0	19.4	102
LCS-141007-6	19.8	20.0	101	19.0	19.8	104
Average ± %RSD	99.1% ± 1.7%			102% ± 1.5%		
LCS-141007-7	157	173	110	150	158	105
LCS-141007-8	157	170	108	150	152	101
LCS-141007-9	157	164	105	150	150	100
Average ± %RSD	108% ± 2.3%			102% ± 2.6%		

ETS-8-044.1 External Calibration Analyzed 10/7/14	PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141007-1	0.184	0.153	82.9
LCS-141007-2	0.184	0.158	86.1
LCS-141007-3	0.184	0.158	85.9
Average ± %RSD	85.0% ± 2.1%		
LCS-141007-4	18.4	16.8	91.4
LCS-141007-5	18.4	17.2	93.2
LCS-141007-6	18.4	17.8	96.6
Average ± %RSD	93.7% ± 2.8%		
LCS-141007-7	145	164	113
LCS-141007-8	145	152	105
LCS-141007-9	145	150	103
Average ± %RSD	107% ± 4.9%		

(1) RSD value did not meet acceptance criteria of ≤20%.

Table 7 continued. Laboratory Control Spike Recovery.

ETS-8-044.1 External Calibration Analyzed 10/13/14	PFBA			PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141013-1	0.198	0.176	89.0	0.190	0.182	95.6
LCS-141013-2	0.198	0.170	85.7	0.190	0.178	93.9
LCS-141013-3	0.198	0.177	89.2	0.190	0.190	100
Average ± %RSD	88.0% ± 2.2%			96.5% ± 3.3%		
LCS-141013-4	1.98	1.91	96.6	1.90	1.81	95.4
LCS-141013-5	1.98	2.06	104	1.90	1.92	101
LCS-141013-6	1.98	2.01	101	1.90	1.88	98.8
Average ± %RSD	101% ± 3.7%			98.4% ± 2.9%		
LCS-141013-7	15.9	15.8	99.3	15.2	16.5	108
LCS-141013-8	15.9	17.2	108	15.2	17.1	113
LCS-141013-9	15.9	17.4	110	15.2	16.9	111
Average ± %RSD	106% ± 5.4%			111% ± 2.3%		

ETS-8-044.1 External Calibration Analyzed 10/13/14	PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141013-1	0.184	0.150	81.6
LCS-141013-2	0.184	0.146	79.1
LCS-141013-3	0.184	0.150	81.4
Average ± %RSD	80.7% ± 1.7%		
LCS-141013-4	1.84	1.53	83.2
LCS-141013-5	1.84	1.58	85.9
LCS-141013-6	1.84	1.56	84.9
Average ± %RSD	84.7% ± 1.6%		
LCS-141013-7	14.7	14.0	95.0
LCS-141013-8	14.7	14.3	97.3
LCS-141013-9	14.7	14.3	97.2
Average ± %RSD	96.5% ± 1.3%		

(1) RSD value did not meet acceptance criteria of ≤20%.

Table 7 continued. Laboratory Control Spike Recovery.

ETS-8-044.1 External Calibration Analyzed 10/15/14						
PFOA (Linear + Branched)			PFOS (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141015-1	0.191	0.175	91.6	0.185	0.179	97.0
LCS-141015-2	0.191	0.175	91.5	0.185	0.183	98.8
LCS-141015-3	0.191	0.181	94.6	0.185	0.181	98.0
Average ± %RSD	92.6% ± 1.9%			97.9% ± 0.92%		
LCS-141015-4	1.91	1.98	104	1.85	1.90	103
LCS-141015-5	1.91	1.91	99.9	1.85	1.88	102
LCS-141015-6	1.91	1.92	101	1.85	1.87	101
Average ± %RSD	102% ± 2.1%			102% ± 0.98%		
LCS-141015-7	7.60	7.89	104	7.36	7.50	102
LCS-141015-8	7.60	7.92	104	7.36	7.47	101
LCS-141015-9	7.60	8.05	106	7.36	7.65	104
Average ± %RSD	105% ± 1.1%			102% ± 1.5%		

ETS-8-044.1 External Calibration Analyzed 10/15/14						
¹³ C ₄ -PFOA			¹³ C ₃ -PFOS			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141015-1	0.199	0.201	101	0.190	0.192	101
LCS-141015-2	0.199	0.201	101	0.190	0.192	101
LCS-141015-3	0.199	0.198	99.5	0.190	0.191	100
Average ± %RSD	101% ± 0.86%			101% ± 0.57%		
LCS-141015-4	1.99	2.10	106	1.90	1.99	105
LCS-141015-5	1.99	2.03	102	1.90	1.94	102
LCS-141015-6	1.99	2.01	101	1.90	1.94	102
Average ± %RSD	103% ± 2.6%			103% ± 1.7%		

(1) RSD value did not meet acceptance criteria of ≤20%.

Table 7 continued. Laboratory Control Spike Recovery.

ETS-8-044.1 External Calibration Analyzed 10/20/14	PFBA			PFOA (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141020-1	0.199	0.164	82.3	0.191	0.189	98.7
LCS-141020-2	0.199	0.199	100	0.191	0.191	100
LCS-141020-3	0.199	0.169	85.1	0.191	0.195	102
Average ± %RSD	89.1% ± 11%			100% ± 1.7%		
LCS-141020-4	19.9	22.0	110	19.1	20.9	109
LCS-141020-5	19.9	21.6	109	19.1	20.3	106
LCS-141020-6	19.9	21.3	107	19.1	20.2	106
Average ± %RSD	109% ± 1.4%			107% ± 1.6%		
LCS-141020-7	157	177	113	151	165	110
LCS-141020-8	157	173	110	151	162	107
LCS-141020-9	157	172	110	151	160	106
Average ± %RSD	111% ± 1.6%			108% ± 1.9%		

ETS-8-044.1 External Calibration Analyzed 10/20/14	PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141020-1	0.185	0.157	84.7
LCS-141020-2	0.185	0.165	89.2
LCS-141020-3	0.185	0.162	87.6
Average ± %RSD	87.2% ± 2.6%		
LCS-141020-4	18.5	18.5	100
LCS-141020-5	18.5	17.9	96.7
LCS-141020-6	18.5	18.1	97.9
Average ± %RSD	98.2% ± 1.7%		
LCS-141020-7	146	159	109
LCS-141020-8	146	152	104
LCS-141020-9	146	150	103
Average ± %RSD	105% ± 3.1%		

(1) RSD value did not meet acceptance criteria of ≤20%.

Table 7 continued. Laboratory Control Spike Recovery.

ETS-8-044.1 External Calibration Analyzed 10/23/14			
PFOS (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141023-1	0.185	0.143	77.5
LCS-141023-2	0.185	0.155	83.9
LCS-141023-3	0.185	0.155	83.6
Average ± %RSD	81.7% ± 4.4%		
LCS-141023-4	1.85	1.74	94.3
LCS-141023-5	1.85	1.74	94.0
LCS-141023-6	1.85	1.70	91.8
Average ± %RSD	93.4% ± 1.5%		
LCS-141023-7	7.36	6.97	94.7
LCS-141023-8	7.36	6.97	94.7
LCS-141023-9	7.36	6.86	93.2
Average ± %RSD	94.2% ± 0.92%		

ETS-8-044.1 External Calibration Analyzed 10/24/14			
PFOA (Linear + Branched)			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-141020-1	0.191	0.213	112
LCS-141020-2	0.191	0.216	113
LCS-141020-3	0.191	0.236	124
Average ± %RSD	116% ± 5.7%		
LCS-141020-4	19.1	21.0	110
LCS-141020-5	19.1	20.7	109
LCS-141020-6	19.1	20.6	108
Average ± %RSD	109% ± 0.92%		
LCS-141020-7	151	169	112
LCS-141020-8	151	164	109
LCS-141020-9	151	161	106
Average ± %RSD	109% ± 2.8%		

(1) RSD value did not meet acceptance criteria of ≤20%.

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.1, the most recent fifty QC samples were used. The analytical method uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

Table 8. Analytical Method Uncertainty

Analyte	Standard Deviation (%)	Method Uncertainty (%)
PFBA	12.5	± 25
PFOA	6.56	± 13
PFOS	8.55	± 17

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spikes (FMS) were prepared for select locations. For this sampling event, FMS samples were collected at eight sampling locations to verify that the analytical method is applicable to the collected matrix. FMSs were generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. FMS recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the extraction and analysis of the analytes of interest. FMS concentrations must be at least 50% of the sample concentration to be considered an appropriate spike level. The reference standards for PFOA and PFOS in the field matrix spiking solution consisted of linear and branched isomers. Table 9 lists the locations and spiking levels for which a target analyte FMS was prepared.

In addition, field matrix spikes for this project consisted of stable isotope surrogate recovery standard spikes (SRSs) of [¹³C₃]-PFBA, [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to all sample bottles prior to sample collection. The [¹³C₃]-labeled PFBA was selected to represent PFBA the [¹³C₄]-labeled PFOA was selected to represent PFOA, and the [¹³C₄]-labeled PFOS was selected to represent PFOS. Following sample analysis, it was suspected that the surrogate recovery standards were not spiked as intended; therefore, no surrogate recovery standard results were used to assess sample recovery.

The following calculation was used to generate the field matrix spike recovery in Section 4 of the report:

$$\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%$$

Table 9. Field Matrix Spike Levels.

Sampling location	PFBA (ng/mL)	PFOA (ng/mL)	PFOS (ng/mL)
Surface Water (SW) locations: MRIW09d, MRIW14, MRIW19b, MRI25f	1.01	0.996	0.996
Interstitial Water (IW) locations: MRIW14d and Trip Blank Low	1.01	0.996	0.996
Interstitial Water (IW) locations: MRIW09, MRIW19d, MRIW25f, and Trip Blank High	5.05	4.98	4.98

3.9 Lab Matrix Spikes (LMS)

Due to noncompliant surrogate recovery standards using external standard calibration, laboratory matrix spike (LMS) samples were prepared for all sample locations that did not contain a field matrix spike sample. The LMSs were used to verify that the analytical method is applicable to the collected matrix. LMSs were generated by adding a measured volume of standard solution to an aliquot of the primary sample. The spike concentrations are presented in Table 10.

LMS recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the extraction and analysis of the analytes of interest. LMS concentrations must be 50% of the sample concentration to be considered an appropriate spike level. LMSs are presented in section 4 of this report.

The following calculation was used to calculate the lab matrix spike recovery in Section 4 of the report:

$$\text{LMS Recovery} = \frac{(\text{Sample Concentration of LMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%$$

Table 10. Lab Matrix Spike Levels.

Sampling Locations	Final Concentration (ng/mL)		
	PFBA	PFOA	PFOS
Surface Water (SW) locations: MRIW09b, MRIW09, MRIW09f, MRIW14b, MRIW14d, MRIW14f, MRIW19, MRIW19d, MRIW19f, MRIW25b, MRIW25, and MRIW25d Interstitial Water (IW) locations: MRIW09f and MRIW25d	0.998	0.957	0.926
Interstitial Water (IW) locations: MRIW09d, MRIW14, MRIW14f, and MRIW19f	4.97	4.77	4.61
Interstitial Water (IW) location: MRIW09b	9.98	9.57	9.26
Interstitial Water (IW) locations: MRIW19, MRIW25b, and MRIW25	49.7	47.7	46.1
Interstitial Water (IW) locations: MRIW14b and MRIW19b	98.9	94.9	91.8

4 Data Summary and Discussion

The tables below summarize the sample results and target analyte field matrix spike or laboratory matrix spike recoveries for the sampling locations as well as the Trip Blank. Results and values are average rounded to three significant figures according to EPA rounding rules. Because of rounding, values may vary slightly from those listed in the raw data. Field matrix spike and laboratory matrix spike recoveries meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix and their respective quantitative ranges.

Following the initial analysis using internal standard calibration, almost all of the FMS sample recoveries were greater than 150%, including the trip blank samples. The FMS recoveries appear biased high due to the internal standard response being approximately one half of the expected response. Therefore, the samples were quantitated using external standard calibration, which resulted in surrogate recovery standards with approximately 50% recovery with the exception of two sample locations. Based on the internal standard response and the field matrix spike recoveries, it was concluded that the surrogates and internal standards were spiked incorrectly at the time the bottles were prepared for sampling. Therefore, all samples were quantitated and reported using external standard calibration. No surrogate recovery standard results are reported, with the exception of the PFOA surrogate results for sample location IW-MRIW19f, in which the surrogate recovery standards were added at the time of sample preparation and dilution.

Using external standard calibration, all field matrix spikes and laboratory matrix spikes met acceptance criteria with the following exception:

IW-MRIW14d: The sample/sample duplicate RPD for PFBA was 128%. The FMS recovery was 52.1%. Since the sample location had very high RPDs for all analytes, the sample set was re-prepared and re-analyzed. The re-analysis confirmed the initial results and the sample set has been reported using the initial analysis. The method uncertainty has been expanded to $\pm 48\%$ for PFBA.

Table 11. CGMN SW MRIW09b 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-001	CGMN-SW-MRIW09b-0-141001	<0.0500	NA	0.0332	NA	<0.0232	NA
ISO11-01-03-17-002	CGMN-SW-MRIW09b-DB-141001	<0.0500	NA	0.0274	NA	<0.0232	NA
ISO11-01-03-17-001: LMS	CGMN-SW-MRIW09b-LMS (1ppb)	1.15	115	1.09	111	0.904	97.6
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		0.0303 ng/mL ± 19%		<0.0232 ng/mL	

NA = Not Applicable

Table 12. CGMN SW MRIW09 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-003	CGMN-SW-MRIW09-0-141001	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-004	CGMN-SW-MRIW09-DB-141001	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-003: LMS	CGMN-SW-MRIW09-LMS (1ppb)	1.10	110	1.04	109	0.888	95.9
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 13. CGMN SW MRIW09d 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-005	CGMN-SW-MRIW09d-0-141001	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-006	CGMN-SW-MRIW09d-DB-141001	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-007	CGMN-SW-MRIW09d-FMS-141001	0.994	98.4	0.916	92.0	0.840	84.3
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 14. CGMN SW MRIW09f 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-008	CGMN-SW-MRIW09f-0-141001	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-009	CGMN-SW-MRIW09f-DB-141001	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-008. LMS	CGMN-SW-MRIW09f-LMS (1ppb)	1.12	112	1.06	111	0.906	97.8
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 15. CGMN SW MRIW14b 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-0'0	CGMN-SW-MRIW14b-0'-140930	<0.0500	NA	0.0789	NA	<0.0232	NA
ISO11-01-03-17-0'1	CGMN-SW-MRIW14b-DB-140930	<0.0500	NA	0.0711	NA	<0.0232	NA
ISO11-01-03-17-0'0; LMS	CGMN-SW-MRIW14b-LMS (1ppb)	1.13	113	1.03	105	0.662	95.2
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		0.0750 ng/mL ± 10%		<0.0232 ng/mL	

NA = Not Applicable

Table 16. CGMN SW MRIW14 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-0'2	CGMN-SW-MRIW14-0-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-0'3	CGMN-SW-MRIW14-DB-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-0'4	CGMN-SW-MRIW14-FMS-140930	0.991	98.1	0.922	92.6	0.627	83.0
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 17. CGMN SW MRIW14d 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-0'5	CGMN-SW-MRIW14d-0-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-0'6	CGMN-SW-MRIW14d-DB-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-0'5; LMS	CGMN-SW-MRIW14d-LMS (1ppb)	1.11	111	1.11	116	0.911	98.4
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 18. CGMN SW MRIW14f 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-0'7	CGMN-SW-MRIW14f-0-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-0'8	CGMN-SW-MRIW14f-DB-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-0'7; LMS	CGMN-SW-MRIW14f-LMS (1ppb)	1.08	108	1.07	112	0.892	96.3
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 19. CGMN SW MRIW19b 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-019	CGMN-SW-MRIW19b-0-140930	0.0808	NA	0.200	NA	0.139	NA
ISO11-01-03-17-020	CGMN-SW-MRIW19b-DB-140930	0.0549	NA	0.205	NA	0.135	NA
ISO11-01-03-17-021	CGMN-SW-MRIW19b-FMS-140930	1.08	100	1.14	94.1	1.01	87.7
Average Concentration (ng/mL) ± %RPD		0.0679 ng/mL ± 38% ⁽¹⁾		0.203 ng/mL ± 2.5%		0.137 ng/mL ± 2.9%	

NA = Not Applicable

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 20. CGMN SW MRIW19 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-022	CGMN-SW-MRIW19-0-140930	<0.0500	NA	0.0922	NA	<0.0232	NA
ISO11-01-03-17-023	CGMN-SW-MRIW19-DB-140930	<0.0500	NA	0.0898	NA	<0.0232	NA
ISO11-01-03-17-022: LMS	CGMN-SW-MRIW19-LMS (1ppb)	1.12	112	1.09	104	0.929	100
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		0.0910 ng/mL ± 2.6%		<0.0232 ng/mL	

NA = Not Applicable

Table 21. CGMN SW MRIW19d 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-024	CGMN-SW-MRIW19d-0-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-025	CGMN-SW-MRIW19d-DB-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-024: LMS	CGMN-SW-MRIW19d-LMS (1ppb)	1.15	115	1.12	117	0.966	104
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 22. CGMN SW MRIW19f 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-026	CGMN-SW-MRIW19f-0-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-027	CGMN-SW-MRIW19f-DB-140930	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-026: LMS	CGMN-SW-MRIW19f-LMS (1ppb)	1.10	110	1.03	108	0.884	95.5
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 23. CGMN SW MRIW25b 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-028	CGMN-SW-MRIW25b-0-140930	0.575	NA	0.171	NA	0.150	NA
ISO11-01-03-17-029	CGMN-SW-MRIW25b-DB-140930	0.530	NA	0.174	NA	0.145	NA
ISO11-01-03-17-028; LMS	CGMN-SW-MRIW25b-LMS (1ppb)	1.68	113	1.16	103	0.984	90.3
Average Concentration (ng/mL) ± %RPD		0.553 ng/mL ± 8.1%		0.173 ng/mL ± 1.7%		0.148 ng/mL ± 3.4%	

NA = Not Applicable

Table 24. CGMN SW MRIW25 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-030	CGMN-SW-MRIW25-0-140930	0.659	NA	0.181	NA	0.128	NA
ISO11-01-03-17-031	CGMN-SW-MRIW25-DB-140930	0.687	NA	0.181	NA	0.126	NA
ISO11-01-03-17-030; LMS	CGMN-SW-MRIW25-LMS (1ppb)	1.80	113	1.16	102	1.09	104
Average Concentration (ng/mL) ± %RPD		0.673 ng/mL ± 4.2%		0.181 ng/mL ± 0.0%		0.127 ng/mL ± 1.6%	

NA = Not Applicable

Table 25. CGMN SW MRIW25d 140930

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-032	CGMN-SW-MRIW25d-0-140930	<0.0500	NA	0.0396	NA	<0.0232	NA
ISO11-01-03-17-033	CGMN-SW-MRIW25d-DB-140930	<0.0500	NA	0.0509	NA	<0.0232	NA
ISO11-01-03-17-032; LMS	CGMN-SW-MRIW25d-LMS (1ppb)	1.08	108	1.03	108	0.869	93.8
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		0.0453 ng/mL ± 25% ⁽¹⁾		<0.0232 ng/mL	

NA = Not Applicable

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 26. CGMN SW MRIW25f 140929

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-034	CGMN-SW-MRIW25f-0-140929	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-035	CGMN-SW-MRIW25f-DB-140929	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-036	CGMN-SW-MRIW25f-FMS-140929	0.976	96.6	0.977	98.1	0.869	87.2
Average Concentration (ng/mL) ± %RPD		<0.0500 ng/mL		<0.0240 ng/mL		<0.0232 ng/mL	

NA = Not Applicable

Table 27. CGMN IW MRIW09b 141002

3M LIMS ID	Description	PFBA		PFQA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-037	CGMN-IW-MRIW09b-0-141002	47.6	NA	8.31	NA	3.00	NA
ISO11-01-03-17-038	CGMN-IW-MRIW09b-DB-141002	44.3	NA	7.88	NA	2.90	NA
ISO11-01-03-17-037; LMS	CGMN-IW-MRIW09b-LMS (10ppb)	60.2	NC	19.6	120	13.7	116
Average Concentration (ng/mL) ± %RPD		46.0 ng/mL ± 7.2%		8.10 ng/mL ± 5.3%		2.95 ng/mL ± 3.4%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 28. CGMN IW MRIW09 141002

3M LIMS ID	Description	PFBA		PFQA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-039	CGMN-IW-MRIW09-0-141002	6.77	NA	1.39	NA	0.392	NA
ISO11-01-03-17-040	CGMN-IW-MRIW09-DB-141002	6.65	NA	1.39	NA	0.374	NA
ISO11-01-03-17-041	CGMN-IW-MRIW09-FMS-141002	11.7	98.8	6.65	106	5.12	95.1
Average Concentration (ng/mL) ± %RPD		6.71 ng/mL ± 1.8%		1.39 ng/mL ± 0.0%		0.383 ng/mL ± 4.7%	

NA = Not Applicable

Table 29. CGMN IW MRIW09d 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-042	CGMN-IW-MRIW09d-0-141001	2.93	NA	0.781	NA	<0.0232	NA
ISO11-01-03-17-043	CGMN-IW-MRIW09d-DB-141001	2.94	NA	0.797	NA	0.0269	NA
ISO11-01-03-17-042; LMS	CGMN-IW-MRIW09d-LMS (5ppb)	8.43	111	5.70	103	4.42	95.3
Average Concentration (ng/mL) ± %RPD		2.94 ng/mL ± 0.34%		0.789 ng/mL ± 2.0%		0.0269 ng/mL	

NA = Not Applicable

Table 30. CGMN IW MRIW09f 141002

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-044	CGMN-IW-MRIW09f-0-141002	15.6	NA	0.523	NA	0.0236	NA
ISO11-01-03-17-045	CGMN-IW-MRIW09f-DB-141002	15.1	NA	0.524	NA	<0.0232	NA
ISO11-01-03-17-044; LMS	CGMN-IW-MRIW09f-LMS (1ppb)	16.4	NC	1.50	102	0.959	101
Average Concentration (ng/mL) ± %RPD		15.4 ng/mL ± 3.3%		0.524 ng/mL ± 0.19%		0.0236 ng/mL	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 31. CGMN IW MRIW14b 141002

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-046	CGMN-IW-MRIW14b-0-141002	24.4	NA	101	NA	37.1	NA
ISO11-01-03-17-047	CGMN-IW-MRIW14b-DB-141002	24.4	NA	102	NA	37.0	NA
ISO11-01-03-17-046; LMS	CGMN-IW-MRIW14b-LMS (100ppb)	131	108	207	111	122	92.5
Average Concentration (ng/mL) ± %RPD		24.4 ng/mL ± 0.0%		102 ng/mL ± 0.99%		37.1 ng/mL ± 0.27%	

NA = Not Applicable

Table 32. CGMN IW MRIW14 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-048	CGMN-IW-MRIW14-0-141001	2.29	NA	4.49	NA	0.284	NA
ISO11-01-03-17-049	CGMN-IW-MRIW14-DB-141001	2.03	NA	3.99	NA	0.218	NA
ISO11-01-03-17-048; LMS	CGMN-IW-MRIW14-LMS (5ppb)	7.39	105	9.06	101	4.39	89.8
Average Concentration (ng/mL) ± %RPD		2.16 ng/mL ± 12%		4.24 ng/mL ± 12%		0.251 ng/mL ± 26%⁽¹⁾	

NA = Not Applicable

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 33. CGMN IW MRIW14d 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-050	CGMN-IW-MRIW14d-0-141001	0.307	NA	0.0780	NA	<0.0232	NA
ISO11-01-03-17-051	CGMN-IW-MRIW14d-DB-141001	1.40	NA	0.354	NA	0.0952	NA
ISO11-01-03-17-052	CGMN-IW-MRIW14d-FMS-141001	1.38	52.1 ⁽¹⁾	1.07	85.7	0.926	83.4
Average Concentration (ng/mL) ± %RPD		0.854 ng/mL ± 128% ⁽²⁾		0.216 ng/mL ± 128% ⁽²⁾		0.0952 ng/mL	

NA = Not Applicable

(1) FMS recovery did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded to ±48% for PFBA.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 34. CGMN IW MRIW14f 141002

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-053	CGMN-IW-MRIW14f-0-141002	72.0	NA	3.96	NA	0.0477	NA
ISO11-01-03-17-054	CGMN-IW-MRIW14f-DB-141002	80.2	NA	4.42	NA	<0.0232	NA
ISO11-01-03-17-053; LMS	CGMN-IW-MRIW14f-LMS (5ppb)	77.6	NC	8.70	94.5	4.14	88.8
Average Concentration (ng/mL) ± %RPD		76.1 ng/mL ± 11%		4.19 ng/mL ± 11%		0.0477 ng/mL	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 35. CGMN IW MRIW19b 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-055	CGMN-IW-MRIW19b-0-141001	69.0	NA	149	NA	33.1	NA
ISO11-01-03-17-056	CGMN-IW-MRIW19b-DB-141001	69.4	NA	144	NA	29.2	NA
ISO11-01-03-17-055; LMS	CGMN-IW-MRIW19b-LMS (100ppb)	171	103	240	98.5	109	84.8
Average Concentration (ng/mL) ± %RPD		69.2 ng/mL ± 0.58%		147 ng/mL ± 3.4%		31.2 ng/mL ± 13%	

NA = Not Applicable

Table 36. CGMN IW MRIW19 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-057	CGMN-IW-MRIW19-0-141001	62.6	NA	9.20	NA	91.9	NA
ISO11-01-03-17-058	CGMN-IW-MRIW19-DB-141001	62.5	NA	9.10	NA	92.7	NA
ISO11-01-03-17-057; LMS	CGMN-IW-MRIW19-LMS (50ppb)	116	108	56.8	99.9	125	70.9
Average Concentration (ng/mL) ± %RPD		62.6 ng/mL ± 0.16%		9.15 ng/mL ± 1.1%		92.3 ng/mL ± 0.87%	

NA = Not Applicable

Table 37. CGMN IW MRIW19d 141001

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-059	CGMN-IW-MRIW19d-0-141001	13.3	NA	0.112	NA	<0.0232	NA
ISO11-01-03-17-060	CGMN-IW-MRIW19d-DB-141001	13.5	NA	0.116	NA	<0.0232	NA
ISO11-01-03-17-061	CGMN-IW-MRIW19d-FMS-141001	17.8	NC	4.94	96.9	4.29	86.1
Average Concentration (ng/mL) ± %RPD		13.4 ng/mL ± 1.5%		0.114 ng/mL ± 3.5%		<0.0232 ng/mL	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 38. CGMN IW MRIW19f 141002

3M LIMS ID	Description	PFBA		PFOA		PFOS		¹³ C ₁ -PFOA ⁽²⁾
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	%Recovery
ISO11-01-03-17-062	CGMN-IW-MRIW19f-0-141002	50.0	NA	223	NA	0.896	NA	96.1
ISO11-01-03-17-063	CGMN-IW-MRIW19f-DB-141002	53.0	NA	239	NA	0.930	NA	95.0
ISO11-01-03-17-062; LMS	CGMN-IW-MRIW19f-LMS (5ppb)	54.8	NC	NA ⁽¹⁾	NA ⁽¹⁾	4.96	87.8	NA ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		51.5 ng/mL ± 5.8%		231 ng/mL ± 6.9%		0.913 ng/mL ± 3.7%		95.5% ± 1.2%

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) The LMS sample was not included in the re-analysis of the sample set for PFOA.

(2) The ¹³C₁-PFOA surrogate recovery standard was added to the sample dilutions during sample preparation.

Table 39. CGMN IW MRIW25b 140930

3M LIMS ID	Description	PFBA		PFQA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-064	CGMN-IW-MRIW25b-0-140930	54.6	NA	380	NA	1300	NA
ISO11-01-03-17-065	CGMN-IW-MRIW25b-DB-140930	53.2	NA	368	NA	1290	NA
ISO11-01-03-17-064: LMS	CGMN-IW-MRIW25b-LMS (50ppb)	108	109	410	NC	1220	NC
Average Concentration (ng/mL) ± %RPD		53.9 ng/mL ± 2.6%		374 ng/mL ± 3.2%		1300 ng/mL ± 0.77%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 40. CGMN IW MRIW25 140930

3M LIMS ID	Description	PFBA		PFQA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-066	CGMN-IW-MRIW25-0-140930	59.1	NA	23.8	NA	16.3	NA
ISO11-01-03-17-067	CGMN-IW-MRIW25-DB-140930	58.6	NA	23.3	NA	16.1	NA
ISO11-01-03-17-066: LMS	CGMN-IW-MRIW25-LMS (50ppb)	111	105	70.9	99.3	52.0	77.7
Average Concentration (ng/mL) ± %RPD		58.9 ng/mL ± 0.85%		23.6 ng/mL ± 2.1%		16.2 ng/mL ± 1.2%	

NA = Not Applicable

Table 41. CGMN IW MRIW25d 141001

3M LIMS ID	Description	PFBA		PFQA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-068	CGMN-IW-MRIW25d-0-141001	11.0	NA	37.4	NA	0.0962	NA
ISO11-01-03-17-069	CGMN-IW-MRIW25d-DB-141001	10.5	NA	35.9	NA	0.0920	NA
ISO11-01-03-17-068; LMS	CGMN-IW-MRIW25d-LMS (1ppb)	11.9	NC	36.5	NC	0.939	91.2
Average Concentration (ng/mL) ± %RPD		10.8 ng/mL ± 4.7%		36.7 ng/mL ± 4.1%		0.0941 ng/mL ± 4.5%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 42. CGMN IW MRIW25f 141001

3M LIMS ID	Description	PFBA		PFQA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-070	CGMN-IW-MRIW25f-0-141001	16.5	NA	10.2	NA	7.21	NA
ISO11-01-03-17-071	CGMN-IW-MRIW25f-DB-141001	15.9	NA	9.83	NA	6.09	NA
ISO11-01-03-17-072	CGMN-IW-MRIW25f-FMS-141001	21.5	NC	15.1	NC	11.7	101
Average Concentration (ng/mL) ± %RPD		16.2 ng/mL ± 3.7%		10.0 ng/mL ± 3.7%		6.65 ng/mL ± 17%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 43. CGMN IW TRIP 140929

3M LIMS ID	Description	PFBA		PFOA		PFOS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO11-01-03-17-075	CGMN-IW-TRIP-0-140929	<0.0500	NA	<0.0240	NA	<0.0232	NA
ISO11-01-03-17-076	CGMN-IW-TRIP-LS-140929	0.958	94.9	0.952	95.6	0.937	94.1
ISO11-01-03-17-077	CGMN-IW-TRIP-HS-140929	4.77	94.5	4.73	95.0	4.74	95.2

NA = Not Applicable

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike and lab matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 11- 43 of this report.

6 Data/Sample Retention

All remaining samples and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Signatures



Digitally signed by Susan T. Wolf
DN: c=US, st=MN, l=St. Paul, ou=3M
Environmental Laboratory - authenticated by
LRA, email=stwolf@mmm.com, o=3M, cn=Susan
T. Wolf
Reason: I have reviewed this document
Date: 2014.11.04 15:49:52 -06'00'

Susan Wolf, 3M Principal Analytical Investigator



Digitally signed by William K. Reagen
DN: c=US, st=MN, l=St. Paul, ou=Laboratory
Director, ou=3M Environmental Laboratory -
authenticated by LRA, email=wkreagen@mmm.com,
o=3M, cn=William K. Reagen
Reason: I am approving this document
Date: 2014.11.05 3:16:15 -06'00'

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.



Digitally signed by Casey Chowell
DN: c=US, st=MN, l=St. Paul, ou=Quality Assurance
Unit, ou=3M Environmental Laboratory -
authenticated by LRA, email=chowell@mmm.com,
o=3M, cn=Casey Chowell
Reason: I agree to the terms defined by the
placement of my signature on this document.
Date: 2014.11.04 16:16:59 -06'00'

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M Environmental Laboratory.

All sample bottles include the addition of internal standards and surrogates.

Do not pre-rinse the bottles.

Do not pour out the contents of the bottle.

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-17

Requester: Kotsmith, James Ronald (MAPLEWO)

Department: 452090 Site Source: 01J9C020

Project Number: 0073138015

Date Created: 9/9/2014

Project Description: Mississippi River Surface and Pore Water Sampling - September 2014

Comments:

Completion Date:

Project Lead: Susan T. Wolf

Phone Number: 651-733-9851

Email Address: stwolf@mmm.com

3M Sample Number Sample Description Date/Time Sampled Matrix Comment

ISO11-01-03-17-001	CGMN-SW-MRIW09b-0- 141001	10-1-14/14:50	SW	Slight overfill
ISO11-01-03-17-002	CGMN-SW-MRIW09b-DB- 141001	10-1-14/14:50		Slight overfill
ISO11-01-03-17-003	CGMN-SW-MRIW09-0- 141001	10-1-14/14:10		Slight overfill
ISO11-01-03-17-004	CGMN-SW-MRIW09-DB- 141001	10-1-14/14:10		Slight overfill
ISO11-01-03-17-005	CGMN-SW-MRIW09d-0- 141001	10-1-14/13:15		
ISO11-01-03-17-006	CGMN-SW-MRIW09d-DB- 141001	10-1-14/13:25		
ISO11-01-03-17-007	CGMN-SW-MRIW09d-FMS- 141001	10-1-14/13:15		Slight overfill
ISO11-01-03-17-008	CGMN-SW-MRIW09f-0- 141001 ^a	10-1-14/12:55		10-1-14/12:55
ISO11-01-03-17-009	CGMN-SW-MRIW09f-DB- 141001 ^a	10-1-14/12:55		10-1-14/12:55
ISO11-01-03-17-010	CGMN-SW-MRIW14b-0- 140930	9-30-14/16:35		
ISO11-01-03-17-011	CGMN-SW-MRIW14b-DB- 140930	9-30-14/16:35		
ISO11-01-03-17-012	CGMN-SW-MRIW14-0- 140930	9-30-14/16:10		
ISO11-01-03-17-013	CGMN-SW-MRIW14-DB- 140930	9-30-14/16:10		
ISO11-01-03-17-014	CGMN-SW-MRIW14-FMS- 140930	9-30-14/16:10		

Sample Condition Upon Receipt Acceptable All items accounted for

Temperature: _____ Deg C Received on Ice Other:

Collected by (print): Dave Carns/Alex West

Collector's signature: *[Signature]*

Relinquished by: _____ Date _____ Time _____ Shipped Via _____ Received by: _____ Date _____ Time _____

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-17 (cont.)

Requester: Kotsmith, James Ronald (MAPLEWC)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 9/9/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Project Description: Mississippi River Surface and Pore Water Sampling - September 2014

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO11-01-03-17-015	CGMN-SW-MRIW14d-0- 140930	9-30-14/15:35	SW	
ISO11-01-03-17-016	CGMN-SW-MRIW14d-DB- 140930	9-30-14/15:35		
ISO11-01-03-17-017	CGMN-SW-MRIW14f-0- 140930	9-30-14/14:45		
ISO11-01-03-17-018	CGMN-SW-MRIW14f-DB- 140930	9-30-14/14:45		
ISO11-01-03-17-019	CGMN-SW-MRIW19b-0- 140930	9-30-14/13:40		
ISO11-01-03-17-020	CGMN-SW-MRIW19b-DB- 140930	9-30-14/13:40		
ISO11-01-03-17-021	CGMN-SW-MRIW19b-FMS- 140930	9-30-14/13:40		
ISO11-01-03-17-022	CGMN-SW-MRIW19-0- 140930	9-30-14/12:55		
ISO11-01-03-17-023	CGMN-SW-MRIW19-DB- 140930	9-30-14/12:55		
ISO11-01-03-17-024	CGMN-SW-MRIW19d-0- 140930	9-30-14/12:05		
ISO11-01-03-17-025	CGMN-SW-MRIW19d-DB- 140930	9-30-14/12:05		
ISO11-01-03-17-026	CGMN-SW-MRIW19f-0- 140930	9-30-14/11:15		
ISO11-01-03-17-027	CGMN-SW-MRIW19f-DB- 140930	9-30-14/11:15		
ISO11-01-03-17-028	CGMN-SW-MRIW25b-0- 140930	9-30-14/10:25		
ISO11-01-03-17-029	CGMN-SW-MRIW25b-DB- 140930	9-30-14/10:25		
ISO11-01-03-17-030	CGMN-SW-MRIW25-0- 140930	9-30-14/09:30		
ISO11-01-03-17-031	CGMN-SW-MRIW25-DB- 140930	9-30-14/09:30		

Sample Condition Upon Receipt Acceptable All items accounted for

Temperature: _____ Deg C Received on Ice Other:

Collected by (print):

Dave Carow/Map West

Collector's signature:

[Signature]

Relinquished by:

Date

Time

Shipped Via

Received by:

Date

Time

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-17 (cont.)

Requester: Kotsmith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 9/9/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Project Description: Mississippi River Surface and Pore Water Sampling -
September 2014

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-17-032	CGMN-SW-MRIW25d-0- 140930	9-30-14/08:25	SW	
ISO11-01-03-17-033	CGMN-SW-MRIW25d-DB- 140930	9-30-14/08:25		
ISO11-01-03-17-034	CGMN-SW-MRIW25F-0- 140929	9-29-14/16:40		
ISO11-01-03-17-035	CGMN-SW-MRIW25F-DB- 140929	9-29-14/16:40		
ISO11-01-03-17-036	CGMN-SW-MRIW25F-FMS- 140929	9-29-14/16:40		
ISO11-01-03-17-037	CGMN-IW-MRIW09b-0- 141002	10-2-14/09:25	PW	
ISO11-01-03-17-038	CGMN-IW-MRIW09b-DB- 141002	10-2-14/09:25		
ISO11-01-03-17-039	CGMN-IW-MRIW09-0- 141002	10-2-14/09:05		
ISO11-01-03-17-040	CGMN-IW-MRIW09-DB- 141002	10-2-14/09:05		
ISO11-01-03-17-041	CGMN-IW-MRIW09-FMS- 141002	10-2-14/09:05		
ISO11-01-03-17-042	CGMN-IW-MRIW09d-0- 141001	10-1-14/13:55		Slight over fill
ISO11-01-03-17-043	CGMN-IW-MRIW09d-DB- 141001	10-1-14/13:55		
ISO11-01-03-17-044	CGMN-IW-MRIW09F-0- 141002	10-2-14/08:55		
ISO11-01-03-17-045	CGMN-IW-MRIW09F-DB- 141002	10-2-14/08:55		
ISO11-01-03-17-046	CGMN-IW-MRIW14b-0- 141002	10-2-14/08:40		
ISO11-01-03-17-047	CGMN-IW-MRIW14b-DB- 141002	10-2-14/08:40		
ISO11-01-03-17-048	CGMN-IW-MRIW14-0- 141001	10-1-14/12:20		

Sample Condition Upon Receipt Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): *Dave Cairn / Alex West*

Collector's signature: *[Signature]*

Relinquished by: _____ Date _____ Time _____ Shipped Via _____ Received by: _____ Date _____ Time _____

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-17 (cont.)

Requester: Kotsmith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 9/9/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Project Description: Mississippi River Surface and Pore Water Sampling -
September 2014

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-17-049	CGMN-IW-MRIW14-DB- 141001	10-1-14 / 12:20	PW	
ISO11-01-03-17-050	CGMN-IW-MRIW14d-0 - 141001	10-1-14 / 10:00		
ISO11-01-03-17-051	CGMN-IW-MRIW14d-DB- 141001	10-1-14 / 10:00		Slight overfill
ISO11-01-03-17-052	CGMN-IW-MRIW14d-FMS- 141001	10-1-14 / 10:00		
ISO11-01-03-17-053	CGMN-IW-MRIW14f-0- 141002	10-2-14 / 08:25		
ISO11-01-03-17-054	CGMN-IW-MRIW14f-DB- 141002	10-2-14 / 08:25		
ISO11-01-03-17-055	CGMN-IW-MRIW19b-0- 141001	10-1-14 / 09:30		
ISO11-01-03-17-056	CGMN-IW-MRIW19b-DB- 141001	10-1-14 / 09:30		
ISO11-01-03-17-057	CGMN-IW-MRIW19-0- 141001	10-1-14 / 09:15		Slight overfill
ISO11-01-03-17-058	CGMN-IW-MRIW19-DB- 141001	10-1-14 / 09:15		Slight overfill
ISO11-01-03-17-059	CGMN-IW-MRIW19d-0- 141001	10-1-14 / 09:05		
ISO11-01-03-17-060	CGMN-IW-MRIW19d-DB- 141001	10-1-14 / 09:05		
ISO11-01-03-17-061	CGMN-IW-MRIW19d-FMS- 141001	10-1-14 / 09:05		Slight overfill
ISO11-01-03-17-062	CGMN-IW-MRIW19f-0- 141002	10-2-14 / 08:05		
ISO11-01-03-17-063	CGMN-IW-MRIW19f-DB- 141002	↓ ↓		
ISO11-01-03-17-064	CGMN-IW-MRIW25b-0- 140930	9-30-14 / 10:45	-	Slight overfill

Sample Condition Upon Receipt Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): *Dave Cairns / Alex West*

Collector's signature: *[Signature]*

Relinquished by: _____ Date _____ Time _____ Shipped Via _____ Received by: _____ Date _____ Time _____

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) 733-9873
Alt. Phone: (651) 736-6559
Fax: (651) 733-4687

Project: ISO11-01-03-17 (cont.)

Requester: Kotsmith, James Ronald (MAPLEWO)
Department: 452090 Site Source: 01J9C020
Project Number: 0073138015
Date Created: 9/9/2014

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: 651-733-9851
Email Address: stwolf@mmm.com

Project Description: Mississippi River Surface and Pore Water Sampling -
September 2014

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO11-01-03-17-065	CGMN-IW-MRIW25b-DB- 140930	9-30-14/10:45	PW	SLIGHT OVER FILL
ISO11-01-03-17-066	CGMN-IW-MRIW25f-0- 140930	9-30-14/10:10		
ISO11-01-03-17-067	CGMN-IW-MRIW25-DB- 140930	9-30-14/10:10		
ISO11-01-03-17-068	CGMN-IW-MRIW25d-0- 141001	10-1-14/08:10		
ISO11-01-03-17-069	CGMN-IW-MRIW25d-DB- 141001	10-1-14/08:10		
ISO11-01-03-17-070	CGMN-IW-MRIW25f-0- 141001	10-1-14/08:25		
ISO11-01-03-17-071	CGMN-IW-MRIW25f-DB- 141001	10-1-14/08:25		
ISO11-01-03-17-072	CGMN-IW-MRIW25f-FMS- 141001	10-1-14/08:25		
ISO11-01-03-17-073	CGMN-SW-MRIW09-RB01- 141001	10-1-14/14:00		CGMN-SW-MRIW09-RB01-141001
ISO11-01-03-17-074	CGMN-IW-MRIW09-RB02- 141001	10-1-14/14:00	W	
ISO11-01-03-17-075	CGMN-IW-TRIP-0- 140929	9-29-14/08:00		
ISO11-01-03-17-076	CGMN-IW-TRIP-LS- 140929	9-29-14/08:00		
ISO11-01-03-17-077	CGMN-IW-TRIP-HS- 140929	9-29-14/08:00		

Sample Condition Upon Receipt: Acceptable All items accounted for
Temperature: _____ Deg C Received on Ice Other:

Collected by (print): Dave Cucco / Max West

Collector's signature: 

Relinquished by: _____ Date _____ Time _____ Shipped Via _____ Received by: _____ Date _____ Time _____
