Jean B. Sweeney
Vice President

3M Environmental, Health and Safety Operations

3M Center, Building 0224-05-W-03
St. Paul, MN 55144-1000
651 737 3569

p.1

3M

Certified Mail

8EHQ-0598-00373
AR226-3937

March 10, 2010

NO CBI

Document Processing Center
EPA East – Room 6428 Attn: Section 8(e)
Office of Pollution Prevention and Toxics, US EPA
1200 Pennsylvania Avenue NW
Washington DC 20460-0001

Re: TSCA 8(e) Substantial Risk Notice: Supplemental to Docket No. 8EHQ-0598-373; Sulfonate-based and Carboxylate-based Fluorochemicals – Cordova, IL well water sampling

To whom it may concern,

3M is submitting this notice to supplement its previous submissions on sulfonyl-based and carboxylate-based fluorochemicals and more specifically, our September 5, 2006 submission on water samples taken from groundwater wells and from the water distribution system at the 3M plant site in Cordova, Illinois.

The data contained in the 2006 submission and in this submission have been generated as part of an on-going site-related environmental assessment of fluorochemicals that 3M is performing for its 3M Cordova, Illinois manufacturing facility. As part of this effort, which is being done with the involvement and approval of the Illinois Environmental Protection Agency, 3M has recently received the enclosed well water data taken from various sites on and in the vicinity of the 3M plant. Off site data are limited to five commercial wells, referred to as Riverstone (C-1), Westway (C-2), CFI (C-3), CFI (C-4), and CFI (C-5). 3M's understanding is that none of this water is used for drinking water purposes.

Enclosed please find a map depicting the sampling locations, a data summary table, and a final analytical report (Interim Report #2-Analysis of 3M Cordova Groundwater Samples: December 2009; Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility).

While 3M does not believe that any of these data taken alone or cumulatively meet the "substantial risk" reporting threshold as defined by the EPA, we nevertheless recognize the on-going work of the U.S. EPA to assess fluorochemical exposure pathways. Therefore, we are placing these results in the 8(e) docket as a supplement to previous submissions.

325470

CONTAINS NO CBI

If you have any questions or would like any additional information, please contact Deanna Luebker at (651) 737-1374 or djluebker@mmm.com.

Sincerely,

Jean B. Sweeney (COL)

Jean B. Sweeney
Staff Vice President, 3M Environmental, Health and Safety Operations

Enclosures (3)

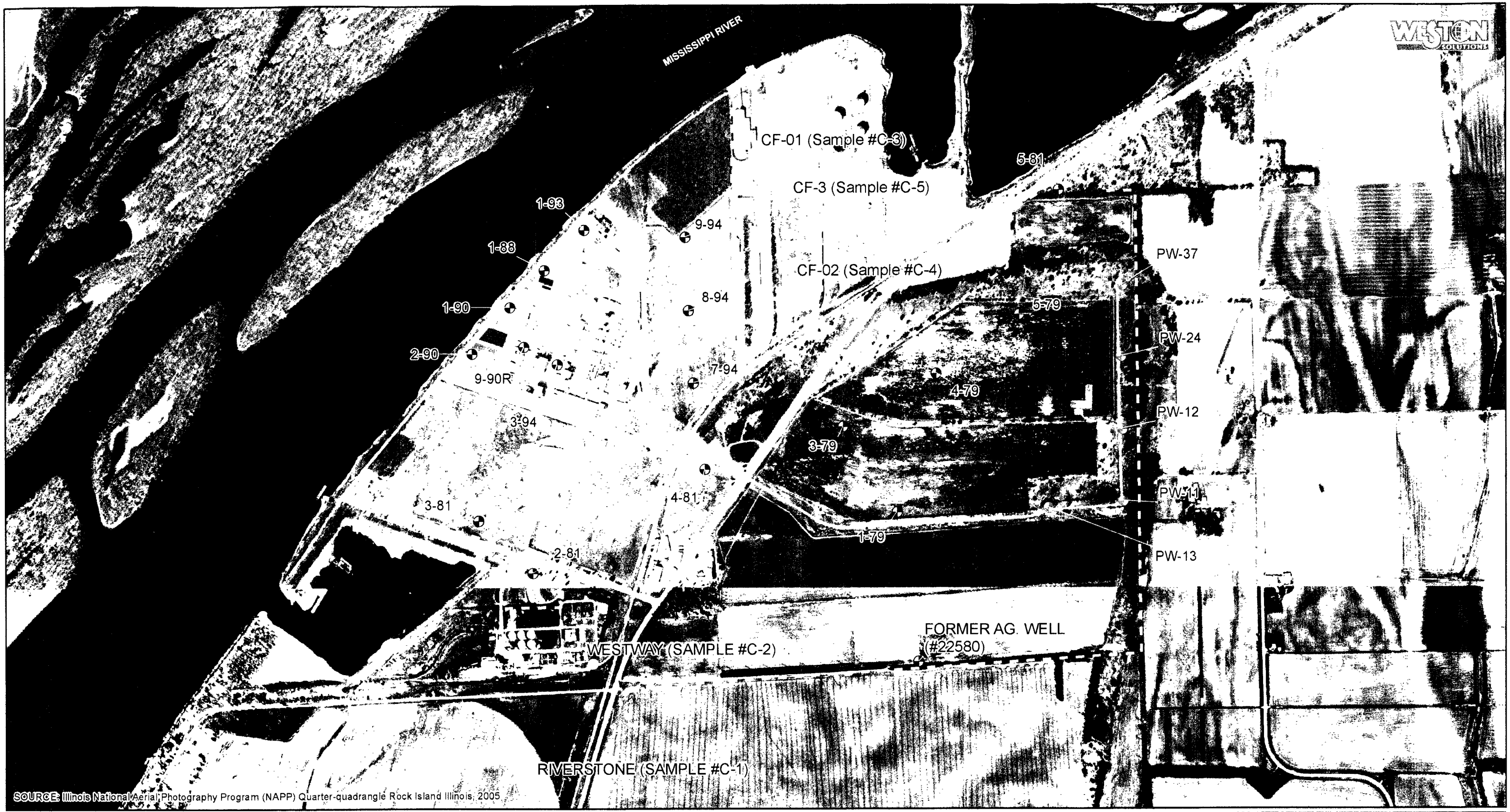
Table 1
Groundwater Sample Results:
March 2008 and December 2009
3M-Cordova, IL

WELL ID	Average Sample Concentration (ng/ml, ppb)					
	Date Sampled	PFBA	PFOA	PFBS	PFHS	PFOS
PLANT AREA WELLS						
MW-1-88	Mar 2008	17.5	0.519	NR	0.63	0.142
	Dec 2009	158	5.85	124	6.04	41.8
MW-1-90	Mar 2008	110	0.369	5.35	0.628	1.25
	Dec 2009	426	5.38	95.8	6.79	37
MW-2-90	Mar 2008	53.1	2.36	0.937	2.29	4.46
	Dec 2009	56.9	4.5	7.54	6.5	5.91
MW-9-90R	Mar 2008	103	1.7	2.87	2.47	27.2
	Dec 2009	372	6.09	76.1	4.82	107
MW-1-93	Mar 2008	22	0.709	0.47	3.78	4.52
	Dec 2009	10.7	0.497	0.217	1.6	5.75
MW-3-94	Mar 2008	734	7.61	120	11.4	51.8
	Dec 2009	290	6.66	238	8.97	76.2
FIELD AREA WELLS (SLUDGE INCORPORATION AREA)						
MW-1-79	Mar 2008	135	6.6	2.5	9.72	22.2
	Dec 2009	240	21	2.57	14.3	24.4
MW-3-79	Mar 2008	32	2.17	0.756	3.95	3.22
	Dec 2009	81.1	4.1	19.8	7.55	16.9
MW-4-79	Mar 2008	1.22	0.032	0.025	0.0339	0.0786
	Dec 2009	8.35	0.123	0.0533	0.046	0.174
MW-5-79	Mar 2008	0.4	< 0.0298	<0.0249	<0.0244	< 0.0250
	Dec 2009	<2.5	0.0865	0.0267	<0.0250	0.0548
MW-2-81	Mar 2008	2.34	< 0.0298	0.26	<0.0293	< 0.0250
	Dec 2009	6.04	0.0344	0.87	<0.0250	0.0538
MW-3-81	Mar 2008	41.3	3.56	0.189	4.32	5.94
	Dec 2009	48.7	5.19	0.357	5.4	7.54
MW-4-81	Mar 2008	145	12.1	0.829	11.6	13.7
	Dec 2009	155	6.26	7.73	6.99	21.5
MW-5-81	Mar 2008	0.135	< 0.0298	<0.0249	<0.0244	< 0.0250
	Dec 2009	0.0891	<0.0303	<0.0255	<0.0250	<0.0253
MW-7-94	Mar 2008	53.7	2.45	0.821	4.98	11
	Dec 2009	63.4	7.23	1.9	7.68	20.8
MW-8-94	Mar 2008	98.4	4	1.05	NR	1.34
	Dec 2009	67.6	12.2	1.04	15.3	1.27
MW-9-94	Mar 2008	14.8	0.0986	0.199	0.763	0.0878
	Dec 2009	63.5	4.8	0.894	6.91	1.93
Former Ag. Well (#22580)	Dec 2009	5.11	<0.0303	0.0604	<0.0250	<0.0253
PRODUCTION WELLS						
PW-11	Mar 2008	2.22	0.0656	<0.0249	0.094	0.266
	Dec 2009	2.09	0.118	<0.0255	0.111	0.278
PW-12	Mar 2008	3.89	0.5	<0.0249	0.53	2.91
	Dec 2009	4.41	1.38	0.0387	1.23	11.3
PW-13	Mar 2008	60.9	4.16	1.82	4.84	11.4
	Dec 2009	57.4	7.93	1.63	6.46	14.8

Table 1
Groundwater Sample Results:
March 2008 and December 2009
3M-Cordova, IL

WELL ID	Average Sample Concentration (ng/ml, ppb)					
	Date Sampled	PFBA	PFOA	PFBS	PFHS	PFOS
PW-24	Mar 2008	2.61	0.455	<0.0249	0.445	3.49
	Dec 2009	1.45	0.347	<0.0255	0.408	0.727
PW-37	Mar 2008	1.39	0.449	<0.0249	0.283	0.383
	Dec 2009	1.39	0.342	<0.0255	0.401	0.698
COMMERCIAL WELLS						
Riverstone (C-1)	Dec 2009	0.788	<0.0303	<0.0255	<0.0250	<0.0253
Westway (C-2)	Dec 2009	4.51	<0.0303	0.145	<0.0250	<0.0253
CFI (C-3)	Dec 2009	0.0767	<0.0303	<0.0255	<0.0250	<0.0253
CFI (C-4)	Dec 2009	0.211	0.0937	<0.0255	<0.0250	0.0413
CFI (C-5)	Dec 2009	0.541	0.0357	<0.0255	<0.0250	<0.0253

NR - Not reported due to quality control failure.



Legend

- Monitor Wells
- Production Wells
- Off-Site Wells
- Site Boundary

N
W E
S

0 1,000 2,000
Feet

Figure 5
Groundwater Sampling Locations
3M Cordova Facility
Cordova, IL

CONFIDENTIAL

3M

Interim Report #2-Analysis of 3M Cordova Groundwater Samples: December 2009

Study Title

Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility

Data Requirement

EPA TSCA Good Laboratory Practice Standards 40 CFR Part 792

Study Director

Jaisimha Kesari P.E., DEE
Weston Solutions, Inc.
1400 Weston Way
West Chester, PA 19380
Phone: 610-701-3761

Author

Susan Wolf
3M Environmental Laboratory

Report Completion Date

Date of signing

Performing Laboratory

3M Environmental Health and Safety Operations
Environmental Laboratory
3M Center, Bldg 260-05-N-17
Maplewood, MN 55144

Project Identification

E07-0149

Total Number of Pages

145

This page has been reserved for specific country requirements.

CONFIDENTIAL

GLP COMPLIANCE STATEMENT

Report Title: Interim Report #2 Analysis of 3M Cordova Groundwater Samples: December 2009
Study Title: Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility".

This analytical phase was conducted in compliance with Toxic Substances Control Act (TSCA) Good Laboratory Practice (GLP) Standards, 40 CFR 792.

Gary A. Hohenstein — 2/5/10

Gary A. Hohenstein, Sponsor Representative — Date

Jaisimha Kesari — 2/12/10

Jaisimha Kesari, P.E., DEE, Study Director — Date

QUALITY ASSURANCE STATEMENT

Report Title: Interim Report #2 Analysis of 3M Cordova Groundwater Samples: December 2009
Study Title: Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility".

This analytical phase was audited by the 3M Environmental Laboratory Quality Assurance Unit (QAU), as indicated in the following table. The findings were reported to the principal investigator (P.I.), laboratory management and study director.

Inspection Dates	Phase	Date Reported to		
		Principal Investigator	Testing Facility Management	Study Director
1/22/2010, 1/25/2010, and 1/26/2010	Data and Report	1/28/2010	2/01/2010	2/01/2010

Casey Howell 2-3-2010

QAU Representative Date

TABLE OF CONTENTS

LIST OF TABLES

1 Study Information

Sponsor

3M Company

Sponsor Representative

Gary A. Hohenstein
Environmental Manager,
Special Projects, EHS Operations
Bldg 224-5W-03
St. Paul, MN 55144-1000
Phone: (651) 737-3570
gahohenstein@mmm.com

Study Director

Jaisimha Kesari, P.E., DEE
Weston Solutions, Inc.
West Chester, PA 19380
Phone: (610) 701-3761
Fax: (610) 701-7401
j.kesari@westonsolutions.com

Study Location

Testing Facility

3M EHS Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55106

Study Personnel

William K. Reagen, Ph.D., Laboratory Manager
Susan Wolf, Principal Investigator. (stwolf@mmm.com); phone (651)-733-9851
Cliff Jacoby, Ph.D Technical Review
Jonathan Steege; analyst

Study Dates

Study Initiation: 11 April 2007
Interim Analytical Initiation: 08 December 2009
Interim Analytical Completion: 14 January 2010
Interim Report Completion: Date of Interim Report Signing

Location of Archives

All original raw data and the analytical report have been archived at the 3M Environmental Laboratory according to 40 CFR Part 792. The test substance and analytical reference standard reserve samples are archived at the 3M Environmental Laboratory according to 40 CFR Part 792

2 Summary

The 3M Environmental Laboratory received one hundred forty-seven samples including one set of trip blanks from the 3M Cordova facility from Weston personnel on December 1-3, 2009 along with their chains of custody (3M C.O.C. #15119 - 15131). Samples were prepared and analyzed for Perfluorobutanoate (PFBA), Perfluorooctanoate (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS) and Perfluorooctane sulfonate (PFOS) under 3M Environmental Laboratory project number E07-0149.

3 Introduction

The objective of this study was to analyze collected groundwater samples from monitoring and production wells located at the 3M Cordova facility for the selected perfluorocarboxylate and perfluorosulfonate analytes.

The 3M Environmental Laboratory prepared sample containers (250 mL polyethylene bottles) which were shipped to the 3M Cordova facility prior to field sampling. Sample containers for each sampling location included a field sample, field sample duplicate, and three field matrix spike samples. Each empty container was marked with a "fill to here" line to produce a final sample volume of 200 mL. Containers designated for field matrix spike samples were fortified with an appropriate matrix spike solution containing the analytes of interest, prior to being sent to the field for sample collection. See section 8.9 of the report for field matrix spike levels.

Samples were prepared and analyzed according to the procedure defined in 3M Environmental Laboratory methods ETS-8-154.3, "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry", ETS-8-110.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water, Soil and Sediment by LC/MS/MS", and ETS-8-044.0 "Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 7 summarizes the sample results using the analytical methods identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.

4 Test & Control Substances

There was no test substance or control substances for this analytical phase in the classic sense. The study was purely analytical in nature. All materials used for this study are listed below and were reference materials as described herein.

5 Reference Substances

Reference Substance	PFBA	PFOA	PFBS
Chemical Name	Perfluorobutanoate	Perfluorooctanoate	Perfluorobutanesulfonate
Chemical Formula	$C_3F_7COO^-$	$C_7F_{15}COO^-$	$C_4F_9SO_3^-$
Identifier	Acid, CAS # 375-22-4	Ammonium Salt, CAS # 335-67-1	Potassium Salt, CAS # 29420-49-3
Source	Sigma	Oakwood	3M
Expiration Date	04/15/2017	09/29/2012	01/10/2017
Storage Conditions	Frozen	Frozen	Frozen
Chemical Lot Number	11921CH	3440	101
TCR Number	TCR07-0011	TCR07-0041	TCR-121
Physical Description	Liquid	White powder	White powder
Purity	98.1%	99.5%	96.7%

Reference Substance	PFHS	PFOS	ECF-PFOA	ECF-PFOS
Chemical Name	Perfluorohexanesulfonate	Perfluorooctane sulfonate	Perfluorooctanoate	Perfluorooctane sulfonate
Chemical Formula	$C_6F_{13}SO_3^-$	$C_8F_{17}SO_3^-$	$C_7F_{15}COO^-$	$C_8F_{17}SO_3^-$
Identifier	Sodium Salt, CAS # 3871-99-6	Potassium Salt, CAS # 2795-39-3	Ammonium Salt, CAS # 335-67-1	Potassium Salt, CAS # 2795-39-3
Source	Wellington	Wellington	3M	3M
Expiration Date	04/02/2013	10/18/2013	02/27/2017	12/14/2016
Storage Conditions	Frozen	Frozen	Frozen	Frozen
Chemical Lot Number	LPFHxSAM06	LPFOSKBM06	332	171
TCR Number	TCR08-0018	TCR08-0001	TCR-123	TCR-696
Physical Description	Crystalline	Crystalline	White powder	White powder
	98%	98%	95%	86.4

6 Test System

The test system for this study is water samples from the 3M Cordova facility collected December 1-3, 2009 by Weston Solutions, Inc. personnel. Samples for this study are "real world" samples, not dosed with a specific lot of test substance.

Sample Description Key Code.

String Number	String Descriptor	Example
1	General Sampling Location	COIL= Cordova, Illinois
2	Sample Type	GW= Ground Water
3	Well Number	Example: MW (monitoring well)
4	Sample Type	0=primary sample
		DB=duplicate sample
		LS=low spike
		EB=equipment rinse blank
5	Sampling Date	091201 = December 1, 2009

7 Method Summary

7.1 Methods

All samples were prepared and analyzed initially by the procedure defined in method ETS-8-110.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water, Soil and Sediment by LC/MS/MS". Samples that could not be reported by ETS-8-110.1 were prepared and analyzed using ETS-8-154.3 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates in Water by Solid Phase Extractions and High Performance Liquid Chromatography/Mass Spectrometry" or ETS-8-044.0 "Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

7.2 Sample Collection

Samples were collected in 250 mL Nalgene™ (high-density polyethylene) bottles prepared at the 3M Environmental Laboratory. Sample bottles were returned to the laboratory at ambient conditions on December 4, 2009. Samples were stored refrigerated at the laboratory after receipt. A set of laboratory prepared Trip Blank and Trip Blank field matrix spikes were sent with each set of collection bottles

7.3 Sample Preparation

7.3.1 ETS-8-110.1

Samples were prepared by removing a 1 mL aliquot of the well mixed sample and diluting it with 9 mL of methanol (dilution factor of 10).

7.3.2 ETS-8-154.3

Samples with concentrations <1 ppb were prepared by ETS-8-154.3, along with calibration standards, method blanks, and associated quality control samples. Briefly, 40 mL of sample were loaded onto a pre-conditioned Waters tC18 solid-phase extraction (SPE) cartridge (Sep-Pak, 1.0 g, 6 cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol. This extraction procedure concentrates the samples by a factor of eight. (Initial volume = 40 mL, final volume = 5 mL).

7.3.3 ETS-8-044.0

Samples, along with calibration standards, method blanks, and associated quality control samples, were prepared by removing an aliquot of the well mixed sample and placing it in an autovial for analysis.

7.4 Analysis

All samples and quality control samples were analyzed for the target analytes listed previously 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.

Table 1. Instrument Parameters.

Instrument Name	ETS Ginger	ETS Jonas
Analytical Method/Analysis Date(s)	ETS-8-110.1 – December 11, 2009 and January 7, 2010 ETS-8-154.3 – December 22, 2009 and January 5, 2010	ETS-8-044.0 – January 14, 2010
Liquid Chromatograph	Agilent 1100	Agilent 1100
Guard column	Prism RP (50 mm X 2.1 mm), 5 µ	Betasil C18 (100 mm X 4.6 mm), 5 µ
Analytical column	Betasil C18 (100 mm X 2.1 mm), 5 µ	Betasil C18 (100 mm X 4.6 mm), 5 µ
Injection Volume	5 µL	100 µL
Mass Spectrometer	Applied Biosystems API 5000	Applied Biosystems API 5000
Ion Source	Turbo Spray	Turbo Spray
Electrode	Z-spray	Z-spray
Polarity	Negative	Negative
Software	Analyst 1.4.2	Analyst 1.4.2

Table 2. Liquid Chromatography Conditions

Step Number	*Total Time (min)*	*Flow Rate (µL/min)*	*Percent A (2 mM ammonium acetate)*	*Percent B (Methanol)*
ETS-8-154.3 and ETS-8-110.1				
0	0	300	90	10
1	2.0	300	90	10
2	14.5	300	10	90
3	15.5	300	10	90
4	16.5	300	90	10
5	20.0	300	90	10
ETS-8-044.0				
0	0	1000	97	3.0
1	0.5	1000	97	3.0
2	11.0	1000	5.0	95
3	13.5	1000	5.0	95
4	13.6	1000	97	3.0
5	17.0	1000	97	3.0

Table 3. Mass Transitions.

Analyte	Mass Transition Q1/Q3	Reference Material Structure
PFBA	213/169	Linear
PFOA	413/369	Linear
	413/219	
	413/169	
PFBS	299/80	Linear
	299/99	
PFHS	399/99	Linear
	399/80	
PFOS	499/99	Linear
	499/80	
	499/130	

Note: The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation. Dwell time was 75 msec.

8 Analytical Results

8.1 Calibration

8.1.1 ETS-8-110.1

Calibration standards were prepared by spiking known amounts of stock solutions containing the analytes of interest into 25 mL of 90:10 methanol:reverse-osmosis (RO) purified laboratory water. Linear PFOS and PFOA reference standards were used for the stock solutions. A total of fourteen calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard included in the final calibration curve met the method calibration accuracy requirement of ±25% (100±30% for the lowest curve point). Correlation coefficients (r) were greater than 0.995 for all analytes.

8.1.2 ETS-8-154.3

Samples were analyzed against an extracted surrogate-matrix matched external calibration curve prepared in reverse-osmosis (RO) purified laboratory water. Calibration standards were prepared by spiking known amounts of stock solutions into 40 mL RO water and carried through the solid-phase extraction procedure. Each spiked water standard was then extracted in the same manner as the collected samples. A total of twelve spiked standards ranging from 0.025 ng/mL to 25 ng/mL (nominal) were prepared. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. 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. The correlation coefficient (r) was greater than 0.995 for all analytes.

8.1.3 ETS-8-044.0

Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into a laboratory-prepared synthetic groundwater containing calcium and magnesium. A total of twelve spiked standards ranging from 0.025 ng/mL to 25 ng/mL (nominal) were analyzed. The 2.5, 10, and 25 ng/mL curve points were disabled for PFBS 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. 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 included in the calibration curve. The correlation coefficient (r) was greater than 0.995 for PFBS.

8.2 System Suitability

Multiple injections of a calibration standard were analyzed at the beginning of the analytical sequence to demonstrate overall system suitability. All compounds met the acceptance criteria of less than or equal to 5% relative standard deviation (RSD) for peak area and less than or equal to 2% RSD for retention time.

8.3 Limit of Quantitation (LOQ)

The LOQ for this analysis 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 nominal LOQ for analytes by analysis date can be found in Table 4.

Table 4. Limit of Quantitation (LOQ)

			LOQ (ng/mL)				
Analysis Date	*Analysis Method*	*Sample Dilution Factor*	*PFBA*	*PFOA*	*PFBS*	*PFHS*	*PFOS*
12/11/09	ETS-8-110.1	10	2.50	0.505	1.02	0.400	0.404
1/7/10	ETS-8-110.1	10	2.50	NA	1.02	NA	NA
12/22/09	ETS-8-154.3	1	NA	0.0303	0.0255	0.0250	0.0253
1/5/2010	ETS-8-154.3	1	0.0300	NA	NA	NA	NA
1/14/10	ETS-8-044.0	1	NA	NA	0.0255	NA	NA

NA = Not Applicable

8.4 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 CCVs met method criteria of 100% ± 25% with the following exceptions:

12/11/09 analysis – Seventeen CCVs were analyzed during the course of the run. One CCV for **PFBS** bracketed reported sample sets COIL-GW-MW-3-94, COIL-GW-MW-1-79, COIL-GW-MW-3-79 and COIL-GW-MW-4-79, had a recovery of 141%.

12/22/09 analysis – Eleven CCVs were analyzed during the course of the run. One CCV for **PFOA** bracketed reported sample sets COIL-GW-C3 (LS and MS), COIL-GW-C4, COIL-GW-C5, Equipment Blanks, and Trip Blanks, had a recovery of 72.4%.

A method deviation has been included in the report.

8.5 Blanks

8.5.1 ETS-8-110.1

Several solvent blanks of 90:10 methanol:reverse osmosis purified water were analyzed throughout the analytical sequence and were used to evaluate method performance and to determine the LOQ for each analyte. Field/trip blanks were also prepared and analyzed with the samples.

8.5.2 ETS-8-154.3

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

8.5.3 ETS-8-044.0

Two types of blanks were prepared and analyzed with the samples: solvent blanks and field/trip blanks. Solvent blank results were reviewed according to the method and used to evaluate method performance to determine the LOQ for each analyte.

8.6 Lab Control Spikes (LCSs)

8.6.1 ETS-8-110.1

Lab control spikes were prepared in duplicate at two concentration levels (nominal concentrations of 5.0 ng/mL and 50 ng/mL. As the reference materials used for quantitation of PFOA and PFOS are predominantly linear, and the analytes present in the water samples were expected to be comprised of branched isomers, LCS samples of both linear and branched PFOA and PFOS were prepared in triplicate, at two concentrations to evaluate the potential for analytical bias. Method ETS-8-110.1 does not specify the number of required LCS or the acceptance criteria, however, typical acceptance criteria for LCS is an average recovery within 100% ± 20%, with a RSD ≤20%. All LCS results met these criteria.

8.6.2 ETS-8-154.3

Low and high lab control spikes were prepared and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 40 mL of reverse osmosis purified laboratory water to produce the desired concentration. The spiked water samples were then extracted and analyzed in the same manner as the samples. Analysis of triplicate LCSs at the two specified levels cross-validates the analytical method as used here for any modifications/deviations from ETS 8-154.3. The method acceptance criteria states that the average recovery of all LCS be between 80%-120% with a RSD ≤20%. All LCS samples met method acceptance criteria with the exception PFBA which had an average recovery of 126%. All LCS were used in the determination of analytical uncertainty. A method deviation is filed with the raw data.

As the reference materials used for quantitation of PFOA and PFOS are predominantly linear, and the analytes present in the water samples are comprised of branched isomers, LCS samples of branched PFOA and PFOS were prepared in triplicate, at two concentrations to evaluate the potential for analytical bias. The average recovery for the branched PFOS was outside of method acceptance criteria with an average recovery of 126%. A method deviation is filed with the raw data.

8.6.3 ETS-8-044.0

Low and high lab control spikes were prepared and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into laboratory-prepared synthetic groundwater containing calcium and magnesium, to produce the desired concentration. The spiked water samples were then prepared and analyzed in the same manner as the samples. Due to the higher curve being disabled to meet method acceptnace criteria, the high LCS at 5 ng/mL could not be calculated as the area counts for these samples exceeded the calibration range. The method acceptance criteria, average of all LCS should be within 100% ± 20% with an RSD ≤20%. The average recovery for the low set of LCS met method acceptance criteria.

The following calculations were used to generate data in Table 5 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 5. Laboratory Control Spike Recovery

ETS-8-110.1 Analyzed 12/11/09	PFBA			Linear PFOA			PFBS			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-091202-1	5.00	5.04	101	5.05	5.12	101	5.10	5.15	101	5.00	5.09	102
LCS-091202-2	5.00	5.14	103	5.05	5.10	101	5.10	5.15	101	5.00	5.12	102
LCS-091202-3	5.00	5.15	103	5.05	5.14	102	5.10	5.10	99.9	5.00	5.13	103
LCS-091202-4	50.0	58.9	118	50.5	59.1	117	51.0	43.2	84.6	50.0	43.4	86.9
LCS-091202-5	50.0	65.4	131	50.5	67.3	133	51.0	47.6	93.4	50.0	46.5	93.0
LCS-091202-6	50.0	66.5	133	50.5	67.6	134	51.0	48.3	94.7	50.0	47.3	94.6
Average ± %RSD	115% ± 13%			115% ± 14 %			95.8% ± 6.7%			96.9% ± 6.7%		

ETS-8-110.1 Analyzed 12/11/09	Linear PFOS				Branched PFOA			Branched PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-091202-1	5.05	5.09	101	LCS-091202-7	5.00	5.65	113	5.00	5.06	101
LCS-091202-2	5.05	5.15	102	LCS-091202-8	5.00	5.66	113	5.00	5.15	103
LCS-091202-3	5.05	5.11	101	LCS-091202-9	5.00	5.79	116	5.00	5.11	102
LCS-091202-4	50.5	43.0	85.1	LCS-091202-10	50.0	61.7	123	50.0	55.1	110
LCS-091202-5	50.5	47.2	93.5	LCS-091202-11	50.0	60.5	121	50.0	54.6	109
LCS-091202-6	50.5	47.7	94.4	LCS-091202-12	50.0	60.1	120	50.0	54.4	109
Average ± %RSD	98.2% ± 6.8%			Average ± %RSD	118% ± 3.6 %			106% ± 3.9%		

NA = Not Applicable

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

(2) No peak was detected in the sample. Sample was not used in the calculation of average and RSD.

(3) The area count for the sample was greater than the highest curve point. A true concentration could not be calculated.

Table 5 continued. Laboratory Control Spike Recovery

ETS-8-154.3 Analyzed 12/22/09 and 1/5/2010	PFBA			Linear PFOA			PFBS			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-091215-1	0.200	0.223	111	0.202	0.215	107	0.204	0.236	115	0.200	0.236	118
LCS-091215-2	0.200	0.259	129	0.202	0.208	103	0.204	0.231	113	0.200	0.235	117
LCS-091215-3	0.200	0.236	118	0.202	0.213	105	0.204	0.230	113	0.200	0.227	113
LCS-091215-4	5.00	6.39	128	5.05	6.11	121	5.10	5.72	112	5.00	5.62	112
LCS-091215-5	5.00	6.13	123	5.05	6.10	121	5.10	5.73	112	5.00	5.61	112
LCS-091215-6	5.00	7.23	145	5.05	6.14	122	5.10	5.76	113	5.00	5.59	112
Average ± %RSD	126% ± 9.2%(1)			113% ± 8.0 %			113% ± 1.0%			114% ± 2.4%		

ETS-8-154.3 Analyzed 12/22/09	Linear PFOS				Branched PFOA			Branched PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-091215-1	0.202	0.226	112	LCS-091215-7	0.200	0.255	127	0.200	0.251	125
LCS-091215-2	0.202	0.218	108	LCS-091215-8	0.200	0.251	126	0.200	0.243	122
LCS-091215-3	0.202	0.222	110	LCS-091215-9	0.200	0.250	125	0.200	0.249	125
LCS-091215-4	5.05	5.77	114	LCS-091215-10	5.00	5.89	118	5.00	6.51	130
LCS-091215-5	5.05	5.74	114	LCS-091215-11	5.00	5.56	111	5.00	6.27	125
LCS-091215-6	5.05	5.78	114	LCS-091215-12	5.00	5.62	112	5.00	6.35	127
Average ± %RSD	112% ± 2.3%			Average ± %RSD	120% ± 6.0 %			126% ± 2.1%(1)		

NA = Not Applicable

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

(2) No peak was detected in the sample. Sample was not used in the calculation of average and RSD.

(3) The area count for the sample was greater than the highest curve point. A true concentration could not be calculated.

Table 5 continued. Laboratory Control Spike Recovery

ETS-8-110.1 Analyzed 1/7/2010	PFBA			Linear PFOA			PFBS			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-091228-1	5.00	5.32	106	5.05	5.46	108	5.10	5.52	108	5.00	5.33	107
LCS-091228-2	5.00	5.14	103	5.05	5.24	104	5.10	5.33	105	5.00	5.18	104
LCS-091228-3	5.00	5.00	100	5.05	5.39	107	5.10	5.35	105	5.00	5.09	102
LCS-091228-4	50.0	68.8	138	50.5	67.2	133	51.0	50.8	99.6	50.0	48.6	97.2
LCS-091228-5	50.0	70.0	140	50.5	68.9	136	51.0	51.3	101	50.0	48.0	96.0
LCS-091228-6	50.0	71.1	142	50.5	68.9	136	51.0	53.6	105	50.0	49.3	98.7
Average ± %RSD	122% ± 17 %(1)			121% ± 13%(1)			104% ± 3.0%			101% ± 4.2%		

ETS-8-110.1 Analyzed 1/7/2010	Linear PFOS				Branched PFOA			Branched PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-091228-1	5.05	5.45	108	LCS-091228-7	5.00	6.33	127	5.00	5.73	115
LCS-091228-2	5.05	5.36	106	LCS-091228-8	5.00	6.03	121	5.00	5.62	112
LCS-091228-3	5.05	5.31	105	LCS-091228-9	5.00	6.11	122	5.00	5.61	112
LCS-091228-4	50.5	49.5	97.9	LCS-091228-10	50.0	63.5	127	50.0	59.3	119
LCS-091228-5	50.5	50.7	100	LCS-091228-11	50.0	62.4	125	50.0	59.2	118
LCS-091228-6	50.5	51.8	103	LCS-091228-12	50.0	NA(2)	NA(2)	50.0	NA(2)	NA(2)
Average ± %RSD	103% ± 3.7%			Average ± %RSD	124% ± 2.2 %(1)			115% ± 2.8%		

NA = Not Applicable

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

(2) No peak was detected in the sample. Sample was not used in the calculation of average and RSD.

(3) The area count for the sample was greater than the highest curve point. A true concentration could not be calculated.

Table 5 continued. Laboratory Control Spike Recovery

ETS-8-044.0 Analyzed 1/14/2010	PFBS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-100113-1	0.204	0.213	104
LCS-100113-2	0.204	0.209	102
LCS-100113-3	0.204	0.212	104
LCS-100113-4	5.10	NA(3)	NA(3)
LCS-100113-5	5.10	NA(3)	NA(3)
LCS-100113-6	5.10	NA(3)	NA(3)
Average ± %RSD	103% ± 3.7%		

NA = Not Applicable
(1) The average recovery did not meet method acceptance criteria of 100% ± 20%.
(2) No peak was detected in the sample. Sample was not used in the calculation of average and RSD.
(3) The area count for the sample was greater than the highest curve point. A true concentration could not be calculated.

8.7 Analytical Method Uncertainty

The analytical uncertainty was determined based on historical QC data that is used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The analytical uncertainty was determined by the statistical evaluation of the recoveries for the individual analyte as determined for laboratory control samples. The standard deviation was calculated for the set of recovery results (in %). The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which correspond with a confidence level of 95%. A minimum of twenty data points is needed to determine method uncertainty by this method.

Table 6. Analytical Method Uncertainty

Analyte	Analytical Method	Mean Recovery (%)	Standard Deviation (%)	Method Uncertainty (%)
PFBA	ETS-8-110.1	103	± 15.3	± 31
PFOA	ETS-8-110.1	117	± 10.7	± 21
PFBS	ETS-8-110.1	94.2	± 9.70	± 19
PFHS	ETS-8-110.1	95.0	± 10.3	± 21
PFOS	ETS-8-110.1	104	± 10.1	± 20
PFBA	ETS-8-154.3	106	± 15.1	± 30
PFOA	ETS-8-154.3	118	± 12.7	± 25
PFBS	ETS-8-154.3	110	± 7.66	± 15
PFHS	ETS-8-154.3	109	± 7.12	± 14
PFOS	ETS-8-154.3	117	± 10.2	± 20
PFBS	ETS-8-044.0	102	± 11.4	± 23

8.8 Sample Results

Table 7 summarizes the sample results using the analytical methods identified above. All results for quality control samples prepared and analyzed with the samples are reported in section 9 of the report.

Table 7. Sample Concentrations

		Sample Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFBA	PFOA	PFBS	PFHS	PFOS
Production Wells						
E07-0149-118	COIL-GW-MW-1-88-0-091203	158	5.85	124	6.12	41.8
E07-0149-119	COIL-GW-MW-1-88-DB-091203	158	5.84	123	5.95	41.7
	Average	**158[1]**	**5.85[1]**	**124[1]**	**6.04[1]**	**41.8[1]**
	%RPD Sample/Sample Duplicate	**0.0**	**0.17**	**0.81**	**2.8**	**0.24**
E07-0149-123	COIL-GW-MW-1-90-0-091202	420	5.26	92.2	6.56	35.6
E07-0149-124	COIL-GW-MW-1-90-DB-091202	431	5.50	99.4	7.01	38.3
	Average	**426[1]**	**5.38[1]**	**95.8[1]**	**6.79[1]**	**37.0[1]**
	%RPD Sample/Sample Duplicate	**2.6**	**4.5**	**7.5**	**6.6**	**7.3**
E07-0149-128	COIL-GW-MW-2-90-0-091202	56.5	4.51	7.81	6.47	5.80
E07-0149-129	COIL-GW-MW-2-90-DB-091202	57.2	4.49	7.27	6.53	6.01
	Average	**56.9[1]**	**4.50[1]**	**7.54[1]**	**6.50[1]**	**5.91[1]**
	%RPD Sample/Sample Duplicate	**1.2**	**0.44**	**7.2**	**0.92**	**3.6**
E07-0149-133	COIL-GW-MW-9-90R-0-091203	385	5.79	77.5	4.41	117
E07-0149-134	COIL-GW-MW-9-90R-DB-091203	359	6.39	74.7	5.22	96.4
	Average	**372[1,3]**	**6.09[1]**	**76.1[1]**	**4.82[1]**	**107[1]**
	%RPD Sample/Sample Duplicate	**7.0**	**9.9**	**3.7**	**17**	**19**
E07-0149-138	COIL-GW-MW-1-93-0-091202	11.4	0.503	0.211	1.66	5.95
E07-0149-139	COIL-GW-MW-1-93-DB-091202	10.0	0.490	0.222	1.53	5.55
	Average	**10.7[1]**	**0.497[2]**	**0.217[2]**	**1.60[1]**	**5.75[1]**
	%RPD Sample/Sample Duplicate	**13**	**2.6**	**5.1**	**8.2**	**7.0**

NA = Not Applicable

NC = Not Calculated; The sample and sample duplicate produced one result above the LOQ and one result below the LOQ. The reported average concentration represents only the result above the LOQ. A true average was not calculated.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were extracted by solid-phase extraction using method ETS-8-154.3. ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) Some sample results may have adjusted analytical uncertainties due to field matrix spike recoveries. See tables in section 9 of the report for additional sample comments.

(4) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

(5) Reported samples were from method ETS-8-044.0, direct injection analysis. ETS-8-044 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 7 continued. Sample Concentrations

		Sample Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFBA	PFOA	PFBS	PFHS	PFOS
Production Wells						
E07-0149-143	COIL-GW-MW-3-94-0-091203	293	6.80	241	9.17	78.0
E07-0149-144	COIL-GW-MW-3-94-DB-091203	286	6.51	234	8.77	74.4
	Average	290[1]	6.66[1]	238[1]	8.97[1]	76.2[1]
	%RPD Sample/Sample Duplicate	2.4	4.4	2.9	4.5	4.7
Field Wells						
E07-0149-148	COIL-GW-MW-1-79-0-091202	240	20.5	1.80	13.7	23.7
E07-0149-149	COIL-GW- MW-1-79-DB-091202	239	21.4	3.34	14.8	25.1
	Average	240[1,3]	21.0[1]	2.57[1]	14.3[1,3]	24.4[1]
	%RPD Sample/Sample Duplicate	0.42	4.3	60[4]	7.7	5.7
E07-0149-153	COIL-GW-MW-3-79-0-091203	81.3	4.05	19.7	7.40	16.9
E07-0149-154	COIL-GW- MW-3-79-DB-091203	80.9	4.15	19.9	7.70	16.9
	Average	81.1[1]	4.10[1]	19.8[1]	7.55[1]	16.9[1,3]
	%RPD Sample/Sample Duplicate	0.49	2.4	1.0	4.0	0.0
E07-0149-158	COIL-GW-MW-4-79-0-091203	6.97	0.123	0.0532	0.0445	0.176
E07-0149-159	COIL-GW- MW-4-79-DB-091203	9.72	0.123	0.0533	0.0474	0.172
	Average	8.35[1]	0.123[2]	0.0533[2]	0.0460[2]	0.174[2]
	%RPD Sample/Sample Duplicate	33[4]	0.0	0.19	6.3	2.3
E07-0149-163	COIL-GW-MW-5-79-0-091202	<2.50	0.0865	0.0273	<0.0250	0.0533
E07-0149-164	COIL-GW- MW-5-79-DB-091202	<2.50	0.0865	0.0261	<0.0250	0.0562
	Average	<2.50[1]	0.0865[2]	0.0267[2]	<0.0250[2]	0.0548[2]
	%RPD Sample/Sample Duplicate	NA	0.0	4.5	NA	5.3

NA = Not Applicable

NC = Not Calculated; The sample and sample duplicate produced one result above the LOQ and one result below the LOQ. The reported average concentration represents only the result above the LOQ. A true average was not calculated.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were extracted by solid-phase extraction using method ETS-8-154.3. ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) Some sample results may have adjusted analytical uncertainties due to field matrix spike recoveries. See tables in section 9 of the report for additional sample comments.

(4) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

(5) Reported samples were from method ETS-8-044.0, direct injection analysis. ETS-8-044 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 7 continued. Sample Concentrations

		Sample Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFBA	PFOA	PFBS	PFHS	PFOS
Field Wells						
E07-0149-168	COIL-GW-MW-2-81-0-091202	6.18	0.0344	0.891	<0.0250	0.0537
E07-0149-169	COIL-GW-MW-2-81-DB-091202	5.89	0.0343	0.849	<0.0250	0.0538
	Average	6.04[1]	0.0344[2]	0.870[2]	<0.0250[2]	0.0538[2]
	%RPD Sample/Sample Duplicate	4.8	0.29	4.8	NA	0.19
E07-0149-173	COIL-GW-MW-3-81-0-091202	49.3	5.63	0.352	5.70	7.95
E07-0149-174	COIL-GW-MW-3-81-DB-091202	48.1	4.74	0.361	5.10	7.12
	Average	48.7[1]	5.19[1]	0.357[5]	5.40[1]	7.54[1]
	%RPD Sample/Sample Duplicate	2.5	17	2.5	11	11
E07-0149-178	COIL-GW-MW-4-81-0-091202	155	6.30	7.78	7.08	22.0
E07-0149-179	COIL-GW-MW-4-81-DB-091202	155	6.21	7.67	6.89	21.0
	Average	155[1]	6.26[1]	7.73[1,3]	6.99[1]	21.5[1]
	%RPD Sample/Sample Duplicate	0.0	1.4	1.4	2.7	4.7
E07-0149-183	COIL-GW-MW-5-81-0-091201	0.0791	<0.0303	<0.0255	<0.0250	<0.0253
E07-0149-184	COIL-GW-MW-5-81-DB-091201	0.0991	<0.0303	<0.0255	<0.0250	<0.0253
	Average	0.0891[2]	<0.0303[2]	<0.0255[2]	<0.0250[2]	<0.0253[2]
	%RPD Sample/Sample Duplicate	22[4]	NA	NA	NA	NA
E07-0149-188	COIL-GW-MW-7-94-0-091202	63.4	7.23	1.54	7.53	21.1
E07-0149-189	COIL-GW-MW-7-94-DB-091202	63.4	7.22	2.26	7.83	20.4
	Average	63.4[1]	7.23[1]	1.90[1]	7.68[1]	20.8[1]
	%RPD Sample/Sample Duplicate	0.0	0.14	38[4]	3.9	3.4

NA = Not Applicable

NC = Not Calculated; The sample and sample duplicate produced one result above the LOQ and one result below the LOQ. The reported average concentration represents only the result above the LOQ. A true average was not calculated.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported samples were extracted by solid-phase extraction using method ETS-8-154.3. ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) Some sample results may have adjusted analytical uncertainties due to field matrix spike recoveries. See tables in section 9 of the report for additional sample comments.

(4) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

(5) Reported samples were from method ETS-8-044.0, direct injection analysis. ETS-8-044 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 7 continued. Sample Concentrations

		Sample Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFBA	PFOA	PFBS	PFHS	PFOS
Field Wells						
E07-0149-193	COIL-GW-MW-8-94-0-091201	67.3	11.9	1.14	14.0	1.35
E07-0149-193	COIL-GW-MW-8-94-DB-091201	67.9	12.5	0.937	16.6	1.19
	Average	**67.6[1]**	**12.2[1]**	**1.04[2]**	**15.3[1]**	**1.27[2]**
	%RPD Sample/Sample Duplicate	**0.89**	**4.9**	**20**	**17**	**13**
E07-0149-198	COIL-GW-MW-9-94-0-091201	64.3	4.98	0.906	6.89	2.12
E07-0149-199	COIL-GW-MW-9-94-DB-091201	62.7	4.61	0.882	6.92	1.74
	Average	**63.5[1]**	**4.80[1]**	**0.894[6]**	**6.91[1]**	**1.93[1]**
	%RPD Sample/Sample Duplicate	**2.5**	**7.7**	**2.7**	**0.43**	**20**
E07-0149-203	COIL-GW-MW-22580-0-091203	5.31	<0.0303	<0.0255	<0.0250	<0.0253
E07-0149-204	COIL-GW-MW-22580-DB-091203	4.91	<0.0303	0.0604	<0.0250	<0.0253
	Average	**5.11[1]**	**<0.0303[2]**	**0.0604[2]**	**<0.0250[2]**	**<0.0253[2]**
	%RPD Sample/Sample Duplicate	**7.8**	**NA**	**NC**	**NA**	**NA**
Production Wells						
E07-0149-208	COIL-GW-PW-11-0-091201	1.97	0.118	<0.0255	0.111	0.288
E07-0149-209	COIL-GW-PW-11-DB-091201	2.20	0.118	<0.0255	0.110	0.268
	Average	**2.09[2]**	**0.118[2]**	**<0.0255[2]**	**0.111[2]**	**0.278[2]**
	%RPD Sample/Sample Duplicate	**11**	**0.0**	**NA**	**0.90**	**7.2**
E07-0149-213	COIL-GW-PW-12-0-091201	4.68	1.42	0.0404	1.23	11.5
E07-0149-214	COIL-GW-PW-12-DB-091201	4.13	1.33	0.0369	1.22	11.1
	Average	**4.41[1]**	**1.38[1]**	**0.0387[2]**	**1.23[1]**	**11.3[1]**
	%RPD Sample/Sample Duplicate	**12**	**6.5**	**9.1**	**0.82**	**3.5**

NA = Not Applicable

NC = Not Calculated; The sample and sample duplicate produced one result above the LOQ and one result below the LOQ. The reported average concentration represents only the result above the LOQ. A true average was not calculated.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were extracted by solid-phase extraction using method ETS-8-154.3. ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) Some sample results may have adjusted analytical uncertainties due to field matrix spike recoveries. See tables in section 9 of the report for additional sample comments.

(4) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

(5) Reported samples were from method ETS-8-044.0, direct injection analysis. ETS-8-044 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 7 continued. Sample Concentrations

		Sample Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFBA	PFOA	PFBS	PFHS	PFOS
Production Wells						
E07-0149-218	COIL-GW-PW-13-0-091201	57.9	7.86	1.67	6.49	14.9
E07-0149-219	COIL-GW-PW-13-DB-091201	56.9	7.99	1.59	6.43	14.7
	Average	57.4[1]	7.93[1]	1.63[1]	6.46[1]	14.8[1]
	%RPD Sample/Sample Duplicate	1.7	1.6	4.9	0.93	1.4
E07-0149-223	COIL-GW-PW-24-0-091201	1.40	0.344	<0.0255	0.403	0.729
E07-0149-224	COIL-GW-PW-24-DB-091201	1.50	0.350	<0.0255	0.412	0.725
	Average	1.46[2]	0.347[2]	<0.0255[2]	0.408[2]	0.727[2]
	%RPD Sample/Sample Duplicate	6.9	1.7	NA	2.2	0.55
E07-0149-228	COIL-GW-PW-37-0-091201	1.41	0.342	<0.0255	0.403	0.709
E07-0149-229	COIL-GW-PW-37-DB-091201	1.36	0.342	<0.0255	0.398	0.686
	Average	1.39[2]	0.342[2]	<0.0255[2]	0.401[2]	0.698[2]
	%RPD Sample/Sample Duplicate	3.6	0.0	NA	1.2	3.3
Commercial Wells						
E07-0149-233	COIL-GW-C1-0-091203	0.756	<0.0303	<0.0255	<0.0250	<0.0253
E07-0149-234	COIL-GW-C1-DB-091203	0.819	<0.0303	<0.0255	<0.0250	<0.0253
	Average	0.788[2]	<0.0303[2]	<0.0255[2]	<0.0250[2]	<0.0253[2,3]
	%RPD Sample/Sample Duplicate	8.0	NA	NA	NA	NA
E07-0149-238	COIL-GW-C2-0-091203	4.32	<0.0303	0.144	<0.0250	<0.0253
E07-0149-239	COIL-GW-C2-DB-091203	4.69	<0.0303	0.145	<0.0250	<0.0253
	Average	4.51[1]	<0.0303[2]	0.145[2]	<0.0250[2]	<0.0253[2]
	%RPD Sample/Sample Duplicate	8.2	NA	0.69	NA	NA

NA = Not Applicable

NC = Not Calculated; The sample and sample duplicate produced one result above the LOQ and one result below the LOQ. The reported average concentration represents only the result above the LOQ. A true average was not calculated.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were extracted by solid-phase extraction using method ETS-8-154.3. ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) Some sample results may have adjusted analytical uncertainties due to field matrix spike recoveries. See tables in section 9 of the report for additional sample comments.

(4) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

(5) Reported samples were from method ETS-8-044.0, direct injection analysis. ETS-8-044 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 7 continued. Sample Concentrations

		Sample Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFBA	PFOA	PFBS	PFHS	PFOS
Commercial Wells						
E07-0149-243	COIL-GW-C3-0-091203	0.0758	<0.0303	<0.0255	<0.0250	<0.0253
E07-0149-244	COIL-GW-C3-DB-091203	0.0775	<0.0303	<0.0255	<0.0250	<0.0253
	Average	0.0767[2,3]	<0.0303[2]	<0.0255[2]	<0.0250[2]	<0.0253[2]
	%RPD Sample/Sample Duplicate	2.2	NA	NA	NA	NA
E07-0149-248	COIL-GW-C4-0-091203	0.213	0.0931	<0.0255	<0.0250	0.0420
E07-0149-249	COIL-GW-C4-DB-091203	0.209	0.0943	<0.0255	<0.0250	0.0406
	Average	0.211[2,3]	0.0937[2]	<0.0255[2]	<0.0250[2]	0.0413[2]
	%RPD Sample/Sample Duplicate	1.9	1.3	NA	NA	3.4
E07-0149-253	COIL-GW-C5-0-091203	0.543	0.0361	<0.0255	<0.0250	<0.0253
E07-0149-254	COIL-GW-C5-DB-091203	0.538	0.0353	<0.0255	<0.0250	<0.0253
	0*Average*	0.541[2]	0.0357[2]	<0.0255[2]	<0.0250[2]	<0.0253[2]
	%RPD Sample/Sample Duplicate	0.93	2.2	NA	NA	NA

NA = Not Applicable

NC = Not Calculated; The sample and sample duplicate produced one result above the LOQ and one result below the LOQ. The reported average concentration represents only the result above the LOQ. A true average was not calculated.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were extracted by solid-phase extraction using method ETS-8-154.3. ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) Some sample results may have adjusted analytical uncertainties due to field matrix spike recoveries. See tables in section 9 of the report for additional sample comments.

(4) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

(5) Reported samples were from method ETS-8-044.0, direct injection analysis. ETS-8-044 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

8.9 Field Matrix Spikes (FMS)

Low and high field matrix spikes were collected at each sampling point (with the exception of the rinse blanks) to verify that the analytical method is applicable to 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 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. Field matrix spikes are presented in the section 9 of this report.

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

Table 8. Field Matrix Spike Concentrations

		Final Concentration (ng/mL)				
Location	**Description**	**PFBA**	**PFOA**	**PFBS**	**PFHS**	**PFOS**
MW-1-88, MW-1-93, and MW-3-79	Low Field Matrix Spike	1.00	0.997	0.999	1.00	0.999
	Mid Field Matrix Spike	10.0	9.97	9.99	10.0	9.99
	High Field Matrix Spike	50.0	49.9	50.0	50.0	50.0
MW-1-90, MW-2-90, MW-9-90R, MW-1-79, MW-3-81, MW-4-81, MW-7-94, MW-8-94, and PW-13	Low Field Matrix Spike	1.00	0.997	0.999	1.00	0.999
	Mid Field Matrix Spike	10.0	9.97	9.99	10.0	9.99
	High Field Matrix Spike	100	101	102	100	101
MW-3-94	Low Field Matrix Spike	10.0	9.97	9.99	10.0	9.99
	Mid Field Matrix Spike	100	101	102	100	101
	High Field Matrix Spike	500	505	510	500	505
MW-4-79, MW-5-79. MW-2-81, MW-5-81, MW-9-94, MW-22580[1], PW-11, PW-12, PW-24, PW-37, and C1[1]-C5	Low Field Matrix Spike	0.250	0.249	0.250	0.250	0.250
	Mid Field Matrix Spike	1.00	0.997	0.999	1.00	0.999
	High Field Matrix Spike	10.0	9.97	9.99	10.0	9.99
Trip Blank	Low Field Matrix Spike	0.250	0.249	0.250	0.250	0.250
	Mid Field Matrix Spike	10.0	9.97	9.99	10.0	9.99
	High Field Matrix Spike	100	101	102	100	101

(1) Sample bottle E07-0149-0206 (COIL-GW-MW-22580-MS) was overfilled by more than 10% (222 mL). Sample bottle E07-0149-0237 (COIL-GW-C1-HS) was overfilled by more than 10% (225mL). The true value of these two spikes has been adjusted accordingly.

8.10 Laboratory Matrix Spike (LMS)

The high field matrix spike concentration was not sufficient for sample location COIL-GW-MW-1-88 for PFBA and PFBS, for MW-1-90 for PFBA, for MW-9-90R for PFBA, for MW-1-79 for PFBA, and for MW-9-94 for PFBA. Laboratory matrix spikes where prepared on the sample prior to analysis by ETS-8-110.1. The preparation of a laboratory matrix spike was inadvertnetly missed for location MW-9-94. A protocol deviation is included in the raw data package. LMS recoveries on this sample are presented on Table 9. Laboratory matrix spikes were prepared on the following samples:

Table 9. Lab Matrix Spike Concentrations.

		Concentration (ng/mL)	Concentration (ng/mL)
Description	Sample ID	PFBA	PFBS
COIL-GW-MW-1-88; 100 ppb Lab Matrix Spike	E07-0149-118	100	102
COIL-GW-MW-1-90; 500 ppb Lab Matrix Spike	E07-0149-123	250	NA
COIL-GW-MW-9-90R; 50 ppb Lab Matrix Spike	E07-0149-133	500	NA
COIL-GW-MW-1-79; 250 ppb Lab Matrix Spike	E07-0149-148	250	NA

NA = Not Applicable

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

9 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for the sampling locations as well as the Trip Blanks. Results and average values are 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 spikes (FMS) recoveries meeting the method acceptance criteria of ± 30%, demonstrate that the method(s) were appropriate for the given matrix and their respective quantitative ranges. In those instances where the most appropriate field matrix spike recoveries did not meet method acceptance criteria, analytical uncertainty has been adjusted accordingly and the data footnoted.

COIL-GW-MW-9-90R: The high field matrix spike was not sufficient for PFBA, therefore, a lab matrix spike (LMS) at 500 ppb was prepared. The recovery of the LMS for PFBA was 148%. Since the LMS was the only appropriate spike level as compared to the sample concentration, the analytical uncertainty for PFBA has been adjusted to 100% ± 48%.

COIL-GW-MW-1-79: The MS sample for PFHS had a recovery of 139%. The recovery of the HS for PFHS was 90.8%. Since the MS was the most appropriate spike level as compared to the sample concentration, the analytical uncertainty for PFHS has been adjusted to 100% ± 39%.

The high field matrix spike was not sufficient for PFBA, therefore, a lab matrix spike (LMS) at 250 ppb was prepared. The recovery of the LMS for PFBA was 145%. Since the LMS was the only appropriate spike level as compared to the sample concentration, the analytical uncertainty for PFBA has been adjusted to 100% ± 45%.

COIL-GW-MW-3-79: The MS sample for PFOS had a recovery of 69.1%. The recovery of the HS for PFOS was 79.7%. Since the MS was the most appropriate spike level as compared to the sample concentration, the analytical uncertainty for PFOS has been adjusted to 100% ± 31%.

COIL-GW-MW-4-81: The MS sample for PFBS had a recovery of 131%. The recovery of the HS for PFBS was 106%. Since the MS was the most appropriate spike level as compared to the sample concentration, the analytical uncertainty for PFBS has been adjusted to 100% ± 31%.

COIL-C1: The LS sample for PFOS had a recovery of 66.9%. The recovery of the MS and HS for PFOS was 80.2% and 85.4%, respectively. Since the LS was the most appropriate spike level as compared to the sample concentration, the analytical uncertainty for PFOS has been adjusted to 100% ± 33%.

Table 10. COIL-GW-MW-1-88 (Plant Area Well)

		PFBA[1]		PFOA[1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-118	COIL-GW-MW-1-88-0-091203	158[2]	NA	5.85	NA
E07-0149-119	COIL-GW-MW-1-88-DB-091203	158[2]	NA	5.84	NA
E07-0149-120	COIL-GW-MW-1-88-LS-091203	NA[2]	NA[2]	6.48	NC
E07-0149-121	COIL-GW-MW-1-88-MS-091203	NA[2]	NA[2]	15.3	94.8
E07-0149-122	COIL-GW-MW-1-88-HS-091203	NA[2]	NA[2]	54.1	96.8
E07-0149-118; 100 ppb LMS	COIL-GW-MW-1-88-0-091203	251[2]	93.0	NA	NA
Average Concentration (ng/mL) ± %RPD		*158 ng/mL ± 0.0%*		*5.85 ng/mL ± 0.17%*	

		PFBS[1]		PFHS[1]		PFOS[1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-118	COIL-GW-MW-1-88-0-091203	124	NA	6.12	NA	41.8	NA
E07-0149-119	COIL-GW-MW-1-88-DB-091203	123	NA	5.95	NA	41.7	NA
E07-0149-120	COIL-GW-MW-1-88-LS-091203	NA[2]	NA[2]	6.77	NC	41.1	NC
E07-0149-121	COIL-GW-MW-1-88-MS-091203	NA[2]	NA[2]	16.1	101	48.6	NC
E07-0149-122	COIL-GW-MW-1-88-HS-091203	NA[2]	NA[2]	57.4	103	87.2	91.0
E07-0149-118; 100 ppb LMS	COIL-GW-MW-1-88-0-091203	228	102	NA	NA	NA	NA
Average Concentration (ng/mL) ± %RPD		*124 ng/mL ± 0.81%*		*6.04 ng/mL ±2.8%*		*41.8 ng/mL ± 0.24%*	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were re-analyzed on 1/7/10 with a LMS sample. The LS, MS, and HS were not analyzed as the spike level was not appropriate as compared to the sample concentration.

Table 11. COIL-GW-MW-1-90 (Plant Area Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-123	COIL-GW-MW-1-90-0-091202	420[2]	NA	5.26	NA
E07-0149-124	COIL-GW-MW-1-90-DB-091202	431[2]	NA	5.50	NA
E07-0149-125	COIL-GW-MW-1-90-LS-091202	NA[2]	NA[2]	6.51	NC
E07-0149-126	COIL-GW-MW-1-90-MS-091202	NA[2]	NA[2]	14.8	94.5
E07-0149-127	COIL-GW-MW-1-90-HS-091202	NA[2]	NA[2]	104	97.6
E07-0149-123; 250 ppb LMS	COIL-GW-MW-1-90-0-091202	722[2]	119	NA	NA
Average Concentration (ng/mL) ± %RPD		426 ng/mL± 2.6%		5.38 ng/mL± 4.5%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-123	COIL-GW-MW-1-90-0-091202	92.2	NA	6.56	NA	35.6	NA
E07-0149-124	COIL-GW-MW-1-90-DB-091202	99.4	NA	7.01	NA	38.3	NA
E07-0149-125	COIL-GW-MW-1-90-LS-091202	98.9	NC	7.66	NC	38.9	NC
E07-0149-126	COIL-GW-MW-1-90-MS-091202	109	NC	16.9	101	48.4	NC
E07-0149-127	COIL-GW-MW-1-90-HS-091202	193	95.3	105	98.2	127	89.2
E07-0149-123; 250 ppb LMS	COIL-GW-MW-1-90-0-091202	NA	NA	NA	NA	NA	NA
Average Concentration (ng/mL) ± %RPD		95.8 ng/mL± 7.5%		6.79 ng/mL± 6.6%		37.0 ng/mL± 7.3%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were re-analyzed on 1/7/10 with a LMS sample. The LS, MS, and HS were not analyzed as the spike level was not appropriate as compared to the sample concentration.

Table 12. COIL-GW-MW-2-90 (Plant Area Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-128	COIL-GW-MW-2-90-0-091202	56.5	NA	4.51	NA
E07-0149-129	COIL-GW-MW-2-90-DB091202	57.2	NA	4.49	NA
E07-0149-130	COIL-GW-MW-2-90-LS091202	56.1	NC	5.62	NC
E07-0149-131	COIL-GW-MW-2-90-MS091202	65.0	NC	14.4	99.3
E07-0149-132	COIL-GW-MW-2-90-HS091202	151	94.2	107	101
Average Concentration (ng/mL) ± %RPD		56.9 ng/mL ± 1.2%		4.50 ng/mL± 0.44%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-128	COIL-GW-MW-2-90-0-091202	7.81	NA	6.47	NA	5.80	NA
E07-0149-129	COIL-GW-MW-2-90-DB091202	7.27	NA	6.53	NA	6.01	NA
E07-0149-130	COIL-GW-MW-2-90-LS091202	8.83	NC	7.67	NC	7.45	NC
E07-0149-131	COIL-GW-MW-2-90-MS091202	17.9	104	16.1	96.0	14.9	90.0
E07-0149-132	COIL-GW-MW-2-90-HS091202	102	92.6	107	101	91.2	84.5
Average Concentration (ng/mL) ± %RPD		7.54 ng/mL± 7.2%		6.50 ng/mL± 0.92%		5.91 ng/mL± 3.6%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 13. COIL-GW-MW-9-90R (Plant Area Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-133	COIL-GW-MW-9-90R-0-091203	385	NA	5.79	NA
E07-0149-134	COIL-GW-MW-9-90R-DB-091203	359	NA	6.39	NA
E07-0149-135	COIL-GW-MW-9-90R-LS-091203	NA[2]	NA[2]	6.95	NC
E07-0149-136	COIL-GW-MW-9-90R-MS-091203	NA[2]	NA[2]	16.5	104
E07-0149-137	COIL-GW-MW-9-90R-HS-091203	NA[2]	NA[2]	100	93.0
E07-0149-133 500 ppb LMS	COIL-GW-MW-9-90R-0-091203	1110	148[3]	NA	NA
Average Concentration (ng/mL) ± %RPD		372 ng/mL ± 7.0%[4]		6.09 ng/mL ± 9.9%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-133	COIL-GW-MW-9-90R-0-091203	77.5	NA	4.41	NA	117	NA
E07-0149-134	COIL-GW-MW-9-90R-DB-091203	74.7	NA	5.22	NA	96.4	NA
E07-0149-135	COIL-GW-MW-9-90R-LS-091203	74.8	NC	5.99	NC	100	NC
E07-0149-136	COIL-GW-MW-9-90R-MS-091203	86.4	NC	16.3	115	110	NC
E07-0149-137	COIL-GW-MW-9-90R-HS-091203	174	96.0	95.7	90.9	199	91.4
Average Concentration (ng/mL) ± %RPD		76.1 ng/m L ± 3.7%		4.82 ng/m L ± 17%		107 ng/mL ± 19%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were re-analyzed on 1/7/10 with a LMS sample. The LS, MS, and HS were not analyzed as the spike level was not appropriate as compared to the sample concentration.

(3) The lab matrix spike did not meet method acceptance criteria of 100 % ± 30%.

(4) The analytical uncertainty has been adjusted for PFBA to 100 % ± 48%.

Table 14. COIL-GW-MW-1-93 (Plant Area Well)

		PFBA [1]		PFOA	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-138	COIL-GW-MW-1-93-0-091202	11.4	NA	0.503 [2]	NA
E07-0149-139	COIL-GW-MW-1-93-DB-091202	10.0	NA	0.490 [2]	NA
E07-0149-140	COIL-GW-MW-1-93-LS-091202	11.4	NC	1.43 [2]	93.6
E07-0149-141	COIL-GW-MW-1-93-MS-091202	20.4	97.0	9.78 [1]	93.1
E07-0149-142	COIL-GW-MW-1-93-HS-091202	56.7	92.0	45.8 [1]	90.9
Average Concentration (ng/mL) ± %RPD		10.7 ng/mL ± 13%		0.497 ng/mL ± 2.6%	

		PFBS		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-138	COIL-GW-MW-1-93-0-091202	0.211 [2]	NA	1.66	NA	5.95	NA
E07-0149-139	COIL-GW-MW-1-93-DB-091202	0.222 [2]	NA	1.53	NA	5.55	NA
E07-0149-140	COIL-GW-MW-1-93-LS-091202	1.39 [2]	117	2.41	81.5	6.28	NC
E07-0149-141	COIL-GW-MW-1-93-MS-091202	9.70 [1]	94.9	11.1	95.1	14.5	87.6
E07-0149-142	COIL-GW-MW-1-93-HS-091202	45.2 [1]	90.1	49.0	94.8	47.3	83.2
Average Concentration (ng/mL) ± %RPD		0.217 ng/mL ± 5.1%		1.60 ng/mL ± 8.2%		5.75 ng/mL ± 7.0%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 15. COIL-GW-MW-3-94 (Plant Area Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-143	COIL-GW-MW-3-94-0-091203	293	NA	6.80	NA
E07-0149-144	COIL-GW-MW-3-94-DB-091203	286	NA	6.51	NA
E07-0149-145	COIL-GW-MW-3-94-LS-091203	296	NC	16.5	98.7
E07-0149-146	COIL-GW-MW-3-94-MS-091203	378	NC	102	94.4
E07-0149-147	COIL-GW-MW-3-94-HS-091203	725	87.1	448	87.4
Average Concentration (ng/mL) ± %RPD		290 ng/mL ± 2.4%		6.66 ng/mL ± 4.4%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-143	COIL-GW-MW-3-94-0-091203	241	NA	9.17	NA	78.0	NA
E07-0149-144	COIL-GW-MW-3-94-DB-091203	234	NA	8.77	NA	74.4	NA
E07-0149-145	COIL-GW-MW-3-94-LS-091203	240	NC	18.9	99.3	84.0	NC
E07-0149-146	COIL-GW-MW-3-94-MS-091203	314	NC	103	94.0	162	85.0
E07-0149-147	COIL-GW-MW-3-94-HS-091203	671	85.0	441	86.4	493	82.5
Average Concentration (ng/mL) ± %RPD		238 ng/mL ± 2.9%		8.97 ng/mL ± 4.5%		76.2 ng/mL ± 4.7%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 16. COIL-GW-MW-1-79 (Field Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-148	COIL-GW-MW-1-79-0-091202	240[2]	NA	20.5	NA
E07-0149-149	COIL-GW-MW-1-79-DB-091202	239[2]	NA	21.4	NA
E07-0149-150	COIL-GW-MW-1-79-LS-091202	NA[2]	NA[2]	20.3	NC
E07-0149-151	COIL-GW-MW-1-79-MS-091202	NA[2]	NA[2]	28.8	78.7
E07-0149-152	COIL-GW-MW-1-79-HS-091202	NA[2]	NA[2]	117	95.1
E07-0149-148; 250 ppb LMS	COIL-GW-MW-1-79-0-091202	601[2]	145 [3]	NA	NA
Average Concentration (ng/mL) ± %RPD		240 ng/mL ± 0.42% [4]		21.0 ng/mL ± 4.3%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-148	COIL-GW-MW-1-79-0-091202	1.80	NA	13.7	NA	23.7	NA
E07-0149-149	COIL-GW-MW-1-79-DB-091202	3.34	NA	14.8	NA	25.1	NA
E07-0149-150	COIL-GW-MW-1-79-LS-091202	3.17	NC	16.8	NC	26.8	NC
E07-0149-151	COIL-GW-MW-1-79-MS-091202	14.2	116	28.1	139 [3]	36.7	123
E07-0149-152	COIL-GW-MW-1-79-HS-091202	98.5	94.0	105	90.8	109	83.8
E07-0149-148; 250 ppb LMS	COIL-GW-MW-1-79-0-091202	NA	NA	NA	NA	NA	NA
Average Concentration (ng/mL) ± %RPD		2.57 ng/mL ± 60% [5]		14.3 ng/mL ± 7.7% [4]		24.4 ng/mL ± 5.7%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Samples were re-analyzed on 1/7/10 with a LMS sample. The LS, MS, and HS were not analyzed as the spike level was not appropriate as compared to the sample concentration.

(3) The field/lab matrix spike did not meet method acceptance criteria of 100 % ± 30%.

(4) The analytical uncertainty has been adjusted for PFBA to 100 % ± 45% and for PFHS to 100 % ± 39%.

(5) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%

Table 17. COIL-GW-MW-3-79 (Field Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-153	COIL-GW-MW-3-79-0-091203	81.3	NA	4.05	NA
E07-0149-154	COIL-GW-MW-3-79-DB-091203	80.9	NA	4.15	NA
E07-0149-155	COIL-GW-MW-3-79-LS-091203	72.7	NC	4.36	NC
E07-0149-156	COIL-GW-MW-3-79-MS-091203	89.4	NC	13.0	89.3
E07-0149-157	COIL-GW-MW-3-79-HS-091203	123	83.8	52.0	96.1
Average Concentration (ng/mL) ± %RPD		81.1 ng/mL ± 0.49%		4.10 ng/mL ± 2.4%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-153	COIL-GW-MW-3-79-0-091203	19.7	NA	7.40	NA	16.9	NA
E07-0149-154	COIL-GW-MW-3-79-DB-091203	19.9	NA	7.70	NA	16.9	NA
E07-0149-155	COIL-GW-MW-3-79-LS-091203	17.2	NC	7.74	NC	14.7	NC
E07-0149-156	COIL-GW-MW-3-79-MS-091203	28.3	85.1	16.5	89.5	23.8	69.1 [2]
E07-0149-157	COIL-GW-MW-3-79-HS-091203	63.7	87.9	57.2	99.3	56.7	79.7
Average Concentration (ng/mL) ± %RPD		19.8ng/mL ± 1.0%		7.55 ng/mL ± 4.0%		16.9 ng/mL ± 0.0% [3]	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) The field matrix spike did not meet method acceptance criteria of 100 % ± 30%.

(3) The analytical uncertainty has been adjusted for PFOS to 100 % ± 31%.

Table 18. COIL-GW-MW-4-79 (Field Well)

		PFBA [1]		PFOA	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-158	COIL-GW-MW-4-79-0-091203	6.97	NA	0.123 [2]	NA
E07-0149-159	COIL-GW-MW-4-79-DB-091203	9.72	NA	0.123 [2]	NA
E07-0149-160	COIL-GW-MW-4-79-LS-091203	8.67	NC	0.355 [2]	93.1
E07-0149-161	COIL-GW-MW-4-79-MS-091203	9.74	NC	1.06 [2]	94.0
E07-0149-162	COIL-GW-MW-4-79-HS-091203	18.0	96.6	10.5 [1]	104
Average Concentration (ng/mL) ± %RPD		8.35 ng/mL ± 33% [3]		0.123 ng/mL ± 0.0%	

		PFBS		PFHS		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-158	COIL-GW-MW-4-79-0-091203	0.0532 [2]	NA	0.0445 [2]	NA	0.176 [2]	NA
E07-0149-159	COIL-GW-MW-4-79-DB-091203	0.0533 [2]	NA	0.0474 [2]	NA	0.172 [2]	NA
E07-0149-160	COIL-GW-MW-4-79-LS-091203	0.354 [2]	120	0.331 [2]	114	0.421 [2]	98.9
E07-0149-161	COIL-GW-MW-4-79-MS-091203	1.28 [2]	123	1.23 [2]	118	1.26 [2]	109
E07-0149-162	COIL-GW-MW-4-79-HS-091203	10.7 [1]	107	10.5 [1]	105	9.45 [1]	92.9
Average Concentration (ng/mL) ± %RPD		0.0533 ng/mL ± 0.19%		0.0460 ng/mL ± 6.3%		0.174 ng/mL ± 2.3%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

Table 19. COIL-GW-MW-5-79 (Field Well)

		PFBA [1]		PFOA [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-163	COIL-GW-MW-5-79-0-091202	<2.50	NA	0.0865	NA
E07-0149-164	COIL-GW-MW-5-79-DB-091202	<2.50	NA	0.0865	NA
E07-0149-165	COIL-GW-MW-5-79-LS-091202	<2.50	NC	0.314	91.3
E07-0149-166	COIL-GW-MW-5-79-MS-091202	<2.50	NC	1.01	92.6
E07-0149-167	COIL-GW-MW-5-79-HS-091202	10.8	108	9.85	97.9
Average Concentration (ng/mL) ± %RPD		<2.50 ng/mL		0.0865 ng/mL ± 0.0%	

		PFBS [2]		PFHS [2]		PFOS [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-163	COIL-GW-MW-5-79-0-091202	0.0273	NA	<0.0250	NA	0.0533	NA
E07-0149-164	COIL-GW-MW-5-79-DB-091202	0.0261	NA	<0.0250	NA	0.0562	NA
E07-0149-165	COIL-GW-MW-5-79-LS-091202	0.340	125	0.296	118	0.285	92.2
E07-0149-166	COIL-GW-MW-5-79-MS-091202	1.20	117	1.16	116	1.10	105
E07-0149-167	COIL-GW-MW-5-79-HS-091202	9.95	99.3	10.3	103	8.90	88.5
Average Concentration (ng/mL) ± %RPD		0.0267 ng/mL ± 4.5%		<0.0250 ng/mL		0.0548 ng/mL ± 5.3%	

NA = Not Applicable

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 20. COIL-GW-MW-2-81 (Field Well)

		PFBA [1]		PFOA [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-168	COIL-GW-MW-2-81-0-091202	6.18	NA	0.0344	NA
E07-0149-169	COIL-GW-MW-2-81-DB-091202	5.89	NA	0.0343	NA
E07-0149-170	COIL-GW-MW-2-81-LS-091202	5.41	NC	0.263	91.7
E07-0149-171	COIL-GW-MW-2-81-MS-091202	7.98	NC	0.996	96.5
E07-0149-172	COIL-GW-MW-2-81-HS-091202	15.9	98.7	9.92	99.2
Average Concentration (ng/mL) ± %RPD		6.04 ng/mL ± 4.8%		0.0344 ng/mL ± 0.29%	

		PFBS [2]		PFHS [2]		PFOS [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-168	COIL-GW-MW-2-81-0-091202	0.891	NA	<0.0250	NA	0.0537	NA
E07-0149-169	COIL-GW-MW-2-81-DB-091202	0.849	NA	<0.0250	NA	0.0538	NA
E07-0149-170	COIL-GW-MW-2-81-LS-091202	1.15	112	0.299	120	0.302	99.4
E07-0149-171	COIL-GW-MW-2-81-MS-091202	2.09	122	1.23	123	1.14	109
E07-0149-172	COIL-GW-MW-2-81-HS-091202	10.1	92.4	10.0	100	8.81	87.7
Average Concentration (ng/mL) ± %RPD		0.870 ng/mL ± 4.8%		<0.0250 ng/mL		0.0538 ng/mL ± 0.19%	

NA = Not Applicable
NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.
(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.
(2) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 21. COIL-GW-MW-3-81 (Field Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-173	COIL-GW-MW-3-81-0-091202	49.3	NA	5.63	NA
E07-0149-174	COIL-GW-MW-3-81-DB-091202	48.1	NA	4.74	NA
E07-0149-175	COIL-GW-MW-3-81-LS-091202	51.4	NC	5.68	NC
E07-0149-176	COIL-GW-MW-3-81-MS-091202	56.3	NC	14.6	94.4
E07-0149-177	COIL-GW-MW-3-81-HS-091202	144	95.3	106	100
Average Concentration (ng/mL) ± %RPD		48.7ng/mL ± 2.5%		5.19 ng/mL ± 17%	

		PFBS		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-173	COIL-GW-MW-3-81-0-091202	0.352[2]	NA	5.70	NA	7.95	NA
E07-0149-174	COIL-GW-MW-3-81-DB-091202	0.361[2]	NA	5.10	NA	7.12	NA
E07-0149-175	COIL-GW-MW-3-81-LS-091202	1.35[2]	99.4	5.93	NC	7.99	NC
E07-0149-176	COIL-GW-MW-3-81-MS-091202	9.07[1]	87.2	14.6	92.0	15.5	79.7
E07-0149-177	COIL-GW-MW-3-81-HS-091202	95.2[1]	93.0	100	94.6	94.5	86.1
Average Concentration (ng/mL) ± %RPD		0.357 ng/mL ± 2.5%		5.40 ng/mL ± 11%		7.54 ng/mL ± 11%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-044.0, direct injection analysis. Method ETS-8-044 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 22. COIL-GW-MW-4-81 (Field Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-178	COIL-GW-MW-4-81-0-091202	155	NA	6.30	NA
E07-0149-179	COIL-GW-MW-4-81-DB-091202	155	NA	6.21	NA
E07-0149-180	COIL-GW-MW-4-81-LS-091202	155	NC	7.13	NC
E07-0149-181	COIL-GW-MW-4-81-MS-091202	197	NC	17.6	114
E07-0149-182	COIL-GW-MW-4-81-HS-091202	264	109	103	95.8
Average Concentration (ng/mL) ± %RPD		155 ng/mL ± 0.0%		6.26 ng/mL ± 1.4%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-178	COIL-GW-MW-4-81-0-091202	7.78	NA	7.08	NA	22.0	NA
E07-0149-179	COIL-GW-MW-4-81-DB-091202	7.67	NA	6.89	NA	21.0	NA
E07-0149-180	COIL-GW-MW-4-81-LS-091202	8.81	NC	8.29	NC	22.6	NC
E07-0149-181	COIL-GW-MW-4-81-MS-091202	20.8	131 [2]	19.1	121	34.0	NC
E07-0149-182	COIL-GW-MW-4-81-HS-091202	116	106	104	97.0	112	89.6
Average Concentration (ng/mL) ± %RPD		7.73ng/mL ± 1.4% [3]		6.99 ng/mL ± 2.7%		21.5 ng/mL ± 4.7%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) The field matrix spike did not meet method acceptance criteria of 100 % ± 30%.

(3) The analytical uncertainty has been adjusted for PFBS to 100 % ± 31%.

Table 23. COIL-GW-MW-5-81 (Field Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-183	COIL-GW-MW-5-81-0-091201	0.0791	NA	<0.0303	NA
E07-0149-184	COIL-GW-MW-5-81-DB-091201	0.0991	NA	<0.0303	NA
E07-0149-185	COIL-GW-MW-5-81-LS-091201	0.415	130	0.264	106
E07-0149-186	COIL-GW-MW-5-81-MS-091201	1.34	125	0.907	91.0
E07-0149-187	COIL-GW-MW-5-81-HS-091201	9.87[2]	97.8	9.50[2]	95.3
Average Concentration (ng/mL) ± %RPD		0.0891 ng/mL ± 22%[3]		<0.0303 ng/mL	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-183	COIL-GW-MW-5-81-0-091201	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-184	COIL-GW-MW-5-81-DB-091201	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-185	COIL-GW-MW-5-81-LS-091201	0.316	127	0.294	118	0.256	103
E07-0149-186	COIL-GW-MW-5-81-MS-091201	1.21	121	1.13	113	0.951	95.2
E07-0149-187	COIL-GW-MW-5-81-HS-091201	9.74[2]	97.5	9.88[2]	98.8	8.56[2]	85.7
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		<0.0250 ng/mL		<0.0253 ng/mL	

NA = Not Applicable

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

p. 47

Table 24. COIL-GW-MW-7-94 (Field Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-188	COIL-GW-MW-7-94-0-091202	63.4	NA	7.23	NA
E07-0149-189	COIL-GW-MW-7-94-DB-091202	63.4	NA	7.22	NA
E07-0149-190	COIL-GW-MW-7-94-LS-091202	63.0	NC	8.55	NC
E07-0149-191	COIL-GW-MW-7-94-MS-091202	69.5	NC	17.1	99.0
E07-0149-192	COIL-GW-MW-7-94-HS-091202	160	96.6	109	101
Average Concentration (ng/mL) ± %RPD		63.4 ng/mL ± 0.0%		7.23 ng/mL ± 0.14%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-188	COIL-GW-MW-7-94-0-091202	1.54	NA	7.53	NA	21.1	NA
E07-0149-189	COIL-GW-MW-7-94-DB-091202	2.26	NA	7.83	NA	20.4	NA
E07-0149-190	COIL-GW-MW-7-94-LS-091202	2.76	86.1	8.56	NC	20.8	NC
E07-0149-191	COIL-GW-MW-7-94-MS-091202	10.8	89.1	17.4	97.2	29.1	NC
E07-0149-192	COIL-GW-MW-7-94-HS-091202	102	98.1	101	93.3	109	87.4
Average Concentration (ng/mL) ± %RPD		1.90 ng/mL ± 38% [2]		7.68 ng/mL ± 3.9%		20.8 ng/mL ± 3.4%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) The relative percent difference (RPD) did not meet method acceptance criteria of ≤20%.

Table 25. COIL-GW-MW-8-94 (Field Well)

		PFBA (1)		PFOA (1)	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-193	COIL-GW-MW-8-94-0-091201	67.3	NA	11.9	NA
E07-0149-194	COIL-GW-MW-8-94-DB-091201	67.9	NA	12.5	NA
E07-0149-195	COIL-GW-MW-8-94-LS-091201	69.2	NC	13.5	NC
E07-0149-196	COIL-GW-MW-8-94-MS-091201	77.9	NC	22.7	105
E07-0149-197	COIL-GW-MW-8-94-HS-091201	161	93.4	116	103
Average Concentration (ng/mL) ± %RPD		67.6ng/mL ± 0.89%		12.2 ng/mL ± 4.9%	

		PFBS (2)		PFHS (1)		PFOS (2)	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-193	COIL-GW-MW-8-94-0-091201	1.14	NA	14.0	NA	1.35	NA
E07-0149-194	COIL-GW-MW-8-94-DB-091201	0.937	NA	16.6	NA	1.19	NA
E07-0149-195	COIL-GW-MW-8-94-LS-091201	2.25	121	17.1	NC	2.28	101
E07-0149-196	COIL-GW-MW-8-94-MS-091201	10.7	96.7	26.5	112	9.17	79.1
E07-0149-197	COIL-GW-MW-8-94-HS-091201	93.1	90.3	109	93.7	83.8	81.7
Average Concentration (ng/mL) ± %RPD		1.04 ng/mL ± 20%		15.3 ng/mL ± 17%		1.27 ng/mL ± 13%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 26. COIL-GW-MW-9-94 (Field Well)

		PFBA [1,2]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-198	COIL-GW-MW-9-94-0-091201	64.3	NA	4.98	NA
E07-0149-199	COIL-GW-MW-9-94-DB-091201	62.7	NA	4.61	NA
E07-0149-200	COIL-GW-MW-9-94-LS-091201	59.6	NC	4.27	NC
E07-0149-201	COIL-GW-MW-9-94-MS-091201	63.7	NC	5.10	NC
E07-0149-202	COIL-GW-MW-9-94-HS-091201	72.0	NC	15.7	109
Average Concentration (ng/mL) ± %RPD		63.5 ng/mL ± 2.5%		4.80 ng/mL ± 7.7%	

		PFBS		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-198	COIL-GW-MW-9-94-0-091201	0.906 [3]	NA	6.89	NA	2.12	NA
E07-0149-199	COIL-GW-MW-9-94-DB-091201	0.882 [3]	NA	6.92	NA	1.74	NA
E07-0149-200	COIL-GW-MW-9-94-LS-091201	NA [4]	NA [4]	6.08	NC	2.19	NC
E07-0149-201	COIL-GW-MW-9-94-MS-091201	1.88 [3]	98.7	7.14	NC	2.75	82.1
E07-0149-202	COIL-GW-MW-9-94-HS-091201	10.7 [1]	98.2	19.0	121	10.4	84.8
Average Concentration (ng/mL) ± %RPD		0.894 ng/mL ± 2.7%		6.91 ng/mL ± 0.43%		1.93 ng/mL ± 20%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) The high field matrix spike at 10 ng/mL was not appropriate as compared to the sample concentration for PFBA. A laboratory matrix spike was not prepared on this sample. This is a deviation from the protocol.

(3) Reported sample concentrations were from method ETS-8-044.0, direct injection analysis. Method ETS-8-044 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(4) Sample not analyzed by ETS-8-044.0, since the spike level was not appropriate as compared to the expected sample concentration.

Table 27. COIL-GW-MW-22580 (Field Well)

		PFBA [1]		PFOA [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-203	COIL-GW-MW-22580-0-091203	5.31	NA	<0.0303	NA
E07-0149-204	COIL-GW-MW-22580-DB-091203	4.91	NA	<0.0303	NA
E07-0149-205	COIL-GW-MW-22580-LS-091203	4.63	NC	0.238	95.5
E07-0149-206[3]	COIL-GW-MW-22580-MS-091203	5.56	NC	0.841	93.6
E07-0149-207	COIL-GW-MW-22580-HS-091203	14.4	92.9	10.6	106
Average Concentration (ng/mL) ± %RPD		5.11 ng/mL ± 7.8%		<0.0303 ng/mL	

		PFBS [2]		PFHS [2]		PFOS [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-203	COIL-GW-MW-22580-0-091203	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-204	COIL-GW-MW-22580-DB-091203	0.0604	NA	<0.0250	NA	<0.0253	NA
E07-0149-205	COIL-GW-MW-22580-LS-091203	0.299	95.5	0.272	109	0.236	94.5
E07-0149-206[3]	COIL-GW-MW-22580-MS-091203	1.11	117	1.02	113	0.959	107
E07-0149-207	COIL-GW-MW-22580-HS-091203	9.73	96.8	11.0	110	8.75	87.6
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		<0.0250 ng/mL		<0.0253 ng/mL	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) Sample bottle was overfilled by more than 10% (222 mL). The field matrix spike true value was adjusted accordingly.

Table 28. COIL-GW-Well 11 (Production Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-208	COIL-GW-PW-11-0-091201	1.97	NA	0.118	NA
E07-0149-209	COIL-GW-PW-11-DB-091201	2.20	NA	0.118	NA
E07-0149-210	COIL-GW-PW-11-LS-091201	2.49	NC	0.343	90.3
E07-0149-211	COIL-GW-PW-11-MS-091201	3.10	102	0.984	86.9
E07-0149-212	COIL-GW-PW-11-HS-091201	11.6[2]	95.2	9.83[2]	97.4
Average Concentration (ng/mL) ± %RPD		2.09 ng/mL ± 11%		0.118 ng/mL ± 0.0%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-208	COIL-GW-PW-11-0-091201	<0.0255	NA	0.111	NA	0.288	NA
E07-0149-209	COIL-GW-PW-11-DB-091201	<0.0255	NA	0.110	NA	0.268	NA
E07-0149-210	COIL-GW-PW-11-LS-091201	0.293	117	0.386	110	0.491	85.3
E07-0149-211	COIL-GW-PW-11-MS-091201	1.10	110	1.18	107	1.19	91.3
E07-0149-212	COIL-GW-PW-11-HS-091201	9.44[2]	94.5	9.88[2]	97.7	8.34[2]	80.7
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		0.111 ng/mL ± 0.90%		0.278 ng/mL ± 7.2%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 29. COIL-GW-Well 12 (Production Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-213	COIL-GW-PW-12-0-091201	4.68	NA	1.42	NA
E07-0149-214	COIL-GW-PW-12-DB-091201	4.13	NA	1.33	NA
E07-0149-215	COIL-GW-PW-12-LS-091201	5.30	NC	1.71	NC
E07-0149-216	COIL-GW-PW-12-MS-091201	6.43	NC	2.50	113
E07-0149-217	COIL-GW-PW-12-HS-091201	15.5	111	11.6	103
Average Concentration (ng/mL) ± %RPD		4.41 ng/mL± 12%		1.38 ng/mL± 6.5%	

		PFBS [2]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-213	COIL-GW-PW-12-0-091201	0.0404	NA	1.23	NA	11.5	NA
E07-0149-214	COIL-GW-PW-12-DB-091201	0.0369	NA	1.22	NA	11.1	NA
E07-0149-215	COIL-GW-PW-12-LS-091201	0.324	114	1.48	NC	11.7	NC
E07-0149-216	COIL-GW-PW-12-MS-091201	1.11	107	2.35	113	12.4	NC
E07-0149-217	COIL-GW-PW-12-HS-091201	10.1[1]	101	11.7	105	19.9	86.1
Average Concentration (ng/mL) ± %RPD		0.0387 ng/mL± 9.1%		1.23 ng/mL± 0.82%		11.3 ng/mL± 3.5%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 30. COIL-GW-Well 13 (Production Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-218	COIL-GW-PW-13-0-091201	57.9	NA	7.86	NA
E07-0149-219	COIL-GW- PW-13-DB-091201	56.9	NA	7.98	NA
E07-0149-220	COIL-GW- PW-13-LS-091201	57.8	NC	8.33	NC
E07-0149-221	COIL-GW- PW-13-MS-091201	66.1	NC	17.5	96.1
E07-0149-222	COIL-GW- PW-13-HS-091201	151	93.6	107	98.1
Average Concentration (ng/mL) ± %RPD		57.4ng/mL ± 1.7%		7.92 ng/mL ± 1.5%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-218	COIL-GW-PW-13-0-091201	1.67	NA	6.49	NA	14.9	NA
E07-0149-219	COIL-GW- PW-13-DB-091201	1.59	NA	6.43	NA	14.7	NA
E07-0149-220	COIL-GW- PW-13-LS-091201	2.43	80.1	7.27	NC	15.1	NC
E07-0149-221	COIL-GW- PW-13-MS-091201	11.2	95.8	15.9	94.4	22.5	77.1
E07-0149-222	COIL-GW- PW-13-HS-091201	97.8	94.3	101	94.5	94.4	78.8
Average Concentration (ng/mL) ± %RPD		1.63 ng/mL ± 4.9%		6.46 ng/mL ± 0.93%		14.8 ng/mL ± 1.4%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 31. COIL-GW-Well 24 (Production Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-223	COIL-GW-PW-24-0-091201	1.40	NA	0.344	NA
E07-0149-224	COIL-GW-PW-24-DB-091201	1.50	NA	0.350	NA
E07-0149-225	COIL-GW-PW-24-LS-091201	1.64	NC	0.585	95.5
E07-0149-226	COIL-GW-PW-24-MS-091201	2.41	96.0	1.21	86.6
E07-0149-277	COIL-GW-PW-24-HS-091201	11.2[2]	97.5	10.6[2]	103
Average Concentration (ng/mL) ± %RPD		1.45 ng/mL± 6.9%		0.347 ng/mL ± 1.7%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-223	COIL-GW-PW-24-0-091201	<0.0255	NA	0.403	NA	0.729	NA
E07-0149-224	COIL-GW-PW-24-DB-091201	<0.0255	NA	0.412	NA	0.725	NA
E07-0149-225	COIL-GW-PW-24-LS-091201	0.305	122	0.697	116	0.936	NC
E07-0149-226	COIL-GW-PW-24-MS-091201	1.10	110	1.50	109	1.66	93.4
E07-0149-277	COIL-GW-PW-24-HS-091201	10.0[2]	100	10.9[2]	105	9.45[2]	87.3
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		0.408 ng/mL ± 2.2%		0.727 ng/mL ± 0.55%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 32. COIL-GW-Well 37 (Production Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-228	COIL-GW-PW-37-0-091201	1.41	NA	0.342	NA
E07-0149-229	COIL-GW-PW-37-DB-091201	1.36	NA	0.342	NA
E07-0149-230	COIL-GW-PW-37-LS-091201	1.69	NC	0.561	87.9
E07-0149-231	COIL-GW-PW-37-MS-091201	2.48	110	1.22	88.1
E07-0149-232	COIL-GW-PW-37-HS-091201	11.2[2]	98.2	10.6[2]	103
Average Concentration (ng/mL) ± %RPD		1.39 ng/mL ± 3.6%		0.342 ng/mL ± 0.0%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-228	COIL-GW-PW-37-0-091201	<0.0255	NA	0.403	NA	0.709	NA
E07-0149-229	COIL-GW-PW-37-DB-091201	<0.0255	NA	0.398	NA	0.686	NA
E07-0149-230	COIL-GW-PW-37-LS-091201	0.301	121	0.680	112	0.917	NC
E07-0149-231	COIL-GW-PW-37-MS-091201	1.13	113	1.50	110	1.67	97.3
E07-0149-232	COIL-GW-PW-37-HS-091201	9.91[2]	99.2	11.0[2]	106	9.19[2]	85.0
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		0.401 ng/mL ± 1.2%		0.698 ng/mL ± 3.3%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 33. COIL-GW-C1 (Commercial Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-233	COIL-GW-C1-0-091203	0.756	NA	<0.0303	NA
E07-0149-234	COIL-GW-C1-DB-091203	0.819	NA	<0.0303	NA
E07-0149-235	COIL-GW-C1-LS-091203	0.961	NC	0.217	87.1
E07-0149-236	COIL-GW-C1-MS-091203	1.85	106	0.846	84.9
E07-0149-237[5]	COIL-GW-C1-HS-091203	9.54[2]	98.5	8.97[2]	101
Average Concentration (ng/mL) ± %RPD		0.788 ng/mL ± 8.0%		<0.0303 ng/mL	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-233	COIL-GW-C1-0-091203	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-234	COIL-GW-C1-DB-091203	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-235	COIL-GW-C1-LS-091203	0.267	107	0.253	101	0.167	66.9[3]
E07-0149-236	COIL-GW-C1-MS-091203	1.09	109	1.02	102	0.801	80.2
E07-0149-237[5]	COIL-GW-C1-HS-091203	8.79[2]	99.0	9.29[2]	105	7.58[2]	85.4
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		<0.0250 ng/mL		<0.0253 ng/mL[4]	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) The field matrix spike did not meet method acceptance criteria of 100% ± 30%.

(4) The analytical uncertainty for PFOS had been adjusted to 100% ± 33%.

(5) Sample bottle was overfilled by more than 10% (225 mL). The field matrix spike true value was adjusted accordingly.

Table 34. COIL-GW-C2 (Commercial Well)

		PFBA [1]		PFOA [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-238	COIL-GW-C2-0-091203	4.32	NA	<0.0303	NA
E07-0149-239	COIL-GW-C2-DB-091203	4.69	NA	<0.0303	NA
E07-0149-240	COIL-GW-C2-LS-091203	4.82	NC	0.224	88.7
E07-0149-241	COIL-GW-C2-MS-091203	5.76	NC	0.872	87.5
E07-0149-242	COIL-GW-C2-HS-091203	15.3	108	9.83[1]	98.6
Average Concentration (ng/mL) ± %RPD		4.51 ng/mL ± 8.2%		<0.0303 ng/mL	

		PFBS [2]		PFHS [2]		PFOS [2]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-238	COIL-GW-C2-0-091203	0.144	NA	<0.0250	NA	<0.0253	NA
E07-0149-239	COIL-GW-C2-DB-091203	0.145	NA	<0.0250	NA	<0.0253	NA
E07-0149-240	COIL-GW-C2-LS-091203	0.400	100	0.245	98.0	0.205	81.2
E07-0149-241	COIL-GW-C2-MS-091203	1.21	107	1.04	104	0.930	93.1
E07-0149-242	COIL-GW-C2-HS-091203	9.86[1]	97.3	9.99[1]	99.9	8.29[1]	83.0
Average Concentration (ng/mL) ± %RPD		0.145 ng/mL ± 0.69%		<0.0250 ng/mL		<0.0253 ng/mL	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 35. COIL-GW-C3 (Commercial Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-243	COIL-GW-C3-0-091203	0.0758	NA	<0.0303	NA
E07-0149-244	COIL-GW-C3-DB-091203	0.0775	NA	<0.0303	NA
E07-0149-245	COIL-GW-C3-LS-091203	0.427	140[3]	0.208	82.4
E07-0149-246	COIL-GW-C3-MS-091203	1.37	129	0.819	82.1
E07-0149-247	COIL-GW-C3-HS-091203	10.2[2]	101	10.1[2]	101
Average Concentration (ng/mL) ± %RPD		0.0767 ng/mL ± 2.2%[4]		<0.0303 ng/mL	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-243	COIL-GW-C3-0-091203	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-244	COIL-GW-C3-DB-091203	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-245	COIL-GW-C3-LS-091203	0.264	104	0.240	96.0	0.210	83.2
E07-0149-246	COIL-GW-C3-MS-091203	1.04	104	0.987	98.7	0.862	86.3
E07-0149-247	COIL-GW-C3-HS-091203	9.74[2]	97.5	10.2[2]	102	8.30[2]	83.1
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		<0.0250 ng/mL		<0.0253 ng/mL	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) The field matrix spike did not meet method acceptance criteria of 100% ± 30%.

(4) The analytical uncertainty for PFBA had been adjusted to 100% ± 40%.

Table 36. COIL-GW-C4 (Commercial Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-248	COIL-GW-C4-0-091203	0.213	NA	0.0931	NA
E07-0149-249	COIL-GW-C4-DB-091203	0.209	NA	0.0943	NA
E07-0149-250	COIL-GW-C4-LS-091203	0.564	141[3]	0.296	80.1
E07-0149-251	COIL-GW-C4-MS-091203	1.41	120	0.925	83.4
E07-0149-252	COIL-GW-C4-HS-091203	12.1[2]	119	9.71[2]	96.5
Average Concentration (ng/mL) ± %RPD		0.211 ng/mL ± 1.9%[4]		0.0937 ng/mL ± 1.3%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-248	COIL-GW-C4-0-091203	<0.0255	NA	<0.0250	NA	0.0420	NA
E07-0149-249	COIL-GW-C4-DB-091203	<0.0255	NA	<0.0250	NA	0.0406	NA
E07-0149-250	COIL-GW-C4-LS-091203	0.289	113	0.257	103	0.233	75.9
E07-0149-251	COIL-GW-C4-MS-091203	1.09	109	1.04	104	0.924	88.4
E07-0149-252	COIL-GW-C4-HS-091203	10.0[2]	100	10.1[2]	101	8.36[2]	83.3
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		<0.0250 ng/mL		0.0413 ng/mL ± 3.4%	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(3) The field matrix spike did not meet method acceptance criteria of 100% ± 30%.

(4) The analytical uncertainty for PFBA had been adjusted to 100% ± 41%.

p. 60

Table 37. COIL-GW-C5 (Commercial Well)

		PFBA [1]		PFOA [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-253	COIL-GW-C5-0-091203	0.543	NA	0.0361	NA
E07-0149-254	COIL-GW-C5-DB-091203	0.538	NA	0.0353	NA
E07-0149-255	COIL-GW-C5-LS-091203	0.856	NC	0.247	83.7
E07-0149-256	COIL-GW-C5-MS-091203	1.69	115	0.925	89.2
E07-0149-257	COIL-GW-C5-HS-091203	9.59[2]	90.5	9.80[2]	97.9
Average Concentration (ng/mL) ± %RPD		0.541 ng/mL ± 0.93%		0.0357ng/mL ± 2.2%	

		PFBS [1]		PFHS [1]		PFOS [1]	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-253	COIL-GW-C5-0-091203	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-254	COIL-GW-C5-DB-091203	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-255	COIL-GW-C5-LS-091203	0.283	111	0.259	104	0.233	92.3
E07-0149-256	COIL-GW-C5-MS-091203	1.09	109	1.05	105	0.952	95.3
E07-0149-257	COIL-GW-C5-HS-091203	9.29[2]	93.0	10.4[2]	104	8.29[2]	83.0
Average Concentration (ng/mL) ± %RPD		<0.0255 ng/mL		<0.0250 ng/mL		<0.0253 ng/mL	

NA = Not Applicable

NC = Not Calculated; Endogenous sample concentration is greater than 2x spike level.

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

Table 38. COIL-GW-TRIP BLANK

		PFBA		PFOA	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-258[1]	COIL-GW-MW-3-94-EB	<0.0300	NA	<0.0303	NA
E07-0149-263[1]	COIL-GW-MW-1-88-EB	<0.0300	NA	<0.0303	NA
E07-0149-264[1]	COIL-GW-MW-22580-EB	<0.0300	NA	<0.0303	NA
E07-0149-268[1]	COIL-GW-TRIP01-0	<0.0300	NA	<0.0303	NA
E07-0149-269[1]	COIL-GW-TRIP01-LS	0.323	129	0.250	100
E07-0149-270[2]	COIL-GW-TRIP01-MS	8.43	84.3	8.13	81.5
E07-0149-270[2]	COIL-GW-TRIP01-MS	10.2	102	10.3	103
E07-0149-271[2]	COIL-GW-TRIP01-HS	94.6	94.6	102	101

		PFBS		PFHS		PFOS	
3M LIMS ID	Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
E07-0149-258[1]	COIL-GW-MW-3-94-EB	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-263[1]	COIL-GW-MW-1-88-EB	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-264[1]	COIL-GW-MW-22580-EB	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-268[1]	COIL-GW-TRIP01-0	<0.0255	NA	<0.0250	NA	<0.0253	NA
E07-0149-269[1]	COIL-GW-TRIP01-LS	0.280	112	0.280	112	0.265	106
E07-0149-270[1]	COIL-GW-TRIP01-MS	10.8	108	10.9	109	10.8	108
E07-0149-270[2]	COIL-GW-TRIP01-MS	9.94	99.5	10.8	108	9.34	93.5
E07-0149-271[2]	COIL-GW-TRIP01-HS	93.9	92.1	93.7	93.7	86.6	85.7

NA = Not Applicable

(1) Reported sample concentrations were from method ETS-8-154.3, solid-phase extraction. Method ETS-8-154 is included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

(2) Reported sample concentrations were from method ETS-8-110.1, direct on-column injection of a samples diluted 1:10 with methanol. Method ETS-8-110 is not included on the 3M Environmental Laboratory's ISO 17025 scope of accreditation.

10 Conclusion

Laboratory control spikes and field matrix spikes were used to determine the analytical method accuracy and precision for all analytes. Analysis was successfully completed following 3M Environmental Laboratory methods described herein.

11 Data/Sample Retention

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

12 Attachments

Attachment A: Test Protocol
Attachment B: Test Protocol Amendment
Attachment C: Analytical Method
Attachment D: Representative Chromatograms and Calibration Curves
Attachment E: Method/Protocol Deviations

13. Signatures

Report Approval:

Susan Wolf, 3M Principal Analytical Investigator — 2/3/2010 — Date

William K. Reagen, Ph.D., 3M Environmental Laboratory Manager — 3 FEB 2010 — Date

Clifton B. Jacoby, Ph.D., 3M Technical Review — 3 Feb. 2010 — Date

Gary A. Hohenstein, Sponsor Representative — 2/5/10 — Date

Jaisimha Kesari, Study Director — 2/12/10 — Date

ATTACHMENT A: TEST PROTOCOL

ANALYTICAL STUDY PROTOCOL

Study Title

Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility".

Data Requirement

GLP

Date:

April 11, 2007

Performing Laboratory

3M Environmental, Health, and Safety Operations
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Laboratory Project Identification

E07-0149

Study Identification

Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility".

Test Substance	The test materials are perfluorobutanoic acid (PFBA), perfluorooctanoic acid (PFOA), perfluorobutanesulfonate (PFBS), perfluorohexanesulfonate (PFHS), and perfluorooctanesulfonate (PFOS).
Sponsor	3M Corporation St. Paul, MN 55144
Sponsor Representative	Gary Hohenstein 3M Environmental Health and Safety Operations Bldg 42-02-E-27 St. Paul, MN 55144 Phone: (651) 778-5150 Fax (651) 778-7203 gahohenstein@mmm.com
Study Director	Jai Kesari Weston Solutions 1400 Weston Way West Chester, PA 19380 Phone: (610) 701-3761 Fax: (610-701-7401 j.kesari@westonsolutions.com

Principal Analytical Investigator (PAI)

Susan Wolf
3M Environmental Health and Safety Operations
3M Center, Bldg. 260-05-N-17
St. Paul, MN 55144
Phone: (651) 733-9851
Fax (651) 733-4687
stwolf@mmm.com

Study Location(s)

Testing Facility

3M Environmental, Health, and Safety Operations
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Proposed Study Timetable

Experimental Start Date Sample Receipt: Early April, 2007

Experimental Termination Date May 31, 2007

1. STUDY

Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility".

2. PURPOSE

The purpose of this study is to analyze groundwater samples from seventeen monitoring wells and five production wells from the 3M Cordova facility, for perfluorobutanoic acid (PFBA), perfluorooctanoic acid (PFOA), (PFBS), perfluorohexanesulfonate (PFHS), and perfluorooctanesulfonate (PFOS). The samples will be prepared and analyzed using the current version 3M Environmental Laboratory analytical method ETS-8-154: "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates In Water By Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry". The targeted limit of quantitation will be **0.025ng/mL** (ppb) for all compounds of interest.

3. REGULATORY COMPLIANCE

The analytical portion will be conducted in accordance with the United States Environmental Protection Agency Good Laboratory Practice Regulations for Non-clinical Laboratory Studies, 40 CFR 792.

4. QUALITY ASSURANCE

The 3M Environmental Laboratory Quality Assurance Unit will audit the study conduct, raw data, and final report to determine compliance with Good Laboratory Practice Regulations, this protocol, and 3M Environmental Laboratory Standard Operating Procedures.

5. TEST SUBSTANCES

Test Substance	PFBA	PFOA	PFBS
Chemical Name	Perfluorobutanoic Acid	Perfluorooctanoic Acid	Perfluorobutane sulfonate
Chemical Formula	$C_3F_7COO^-$	$C_7F_{15}COO^-$	$C_4F_9SO_3^-$
Identifier	Acid CAS# 375-22-4	Ammonium Salt CAS# 3825-26-1	Potassium Salt CAS# 29420-49-3

Test Substance	PFHS	PFOS
Chemical Name	Perfluorohexane sulfonate	Perfluorooctane sulfonate
Chemical Formula	$C_6F_{13}SO_3^-$	$C_8F_{17}SO_3^-$
Identifier	Potassium Salt CAS# 3871-99-6	Potassium Salt CAS# 2795-39-3

Samples for this study are "real world" samples, not dosed with a specific lot of test substance, at any particular rate. Therefore only the analytical phase of this study will be performed under the GLP standards.

The test substances for this study are not test substances in the classic sense. The known source, expiration date, storage conditions, chemical lot number, TCR number, physical description, and purity of the test substances that are being monitored in the test system are not known as they would be known for the reference substances.

6. REFERENCE SUBSTANCES

Reference Substance	PFBA	PFOA	PFBS	PFHS	PFOS
Chemical Name	Perfluorobutanoic Acid	Perfluorooctanoic Acid	Perfluorobutanesulfonate	Perfluorohexanesulfonate	Perfluorooctanesulfonate
Chemical Formula	$C_3F_7COO^-$	$C_7F_{15}COO^-$	$C_4F_9SO_3^-$	$C_6F_{13}SO_3^-$	$C_8F_{17}SO_3^-$
Identifier	Acid CAS# 375-22-4	Ammonium Salt CAS# 3825-26-1	Potassium Salt CAS# 29420-49-3	Potassium Salt CAS# 3871-99-6	Potassium Salt CAS# 2795-39-3
(1) Source	Aldrich	3M	3M	3M	3M
(2) Expiration Date	02/16/2017	02/27/2017	01/18/2017	02/12/2017	08/31/2016
Storage Conditions	Frozen	Frozen	Frozen	Frozen	Frozen
Chemical Lot Number	09713EC	332	2	NB 120067-69	217
TCR Number	TCR-757	TCR-123	TCR-282	TCR-083	TCR-1166
Physical Description	Liquid, room temp	White powder	Good/white crystals	White powder	White powder
Purity	98.7%	95%	97.3%	98.6%	86.9%
(3) Solubility	No information available	>20%	54,400 ppm	No information available	680 ppm

(1) Documentation regarding synthesis of the test substances is located at the source.
(2) Samples will be analyzed against solutions prepared from these reference substances, and these solutions will be assigned expiration dates according to SOP ETS-4-027.
(3) All test substances are believed to be soluble in water at the levels to be investigated. During the course of the study, if there is any indication that the substances are not soluble at the levels tested (visible precipitates), solubility will be further investigated and discussed in the report.

7. TEST SYSTEM

The test system is groundwater. Additional sources may be added as requested. Sections 40 CFR 792.120 (6), (9), (10) and (11) are not applicable as only the analytical phase of this study is under the Good Laboratory Practices.

The test systems were chosen to access the impact of the reference substances in the 3M Cordova, Illinois facility, and are defined in the work plan. The samples will be collected by Weston Solutions and shipped to the testing facility. The control samples will be prepared by the testing facility.

Prior to collection, each sample bottle sent to the testing location will be identified with a laboratory sample reference number. This reference number will be unique and will distinguish each laboratory sample that is carried through the analytical procedure. Chromatographic data will be identified by the laboratory sample reference number.

8. SPECIMEN RECEIPT

The groundwater samples will be collected from seventeen monitoring wells and five production wells from the 3M Cordova facility. Water samples will be collected by Weston Solutions personnel and shipped to the 3M Environmental Lab on ice. Samples will be kept refrigerated at 2°C- 8°C from the time of sample receipt until analysis.

9. EXPERIMENTAL DESIGN

9.1 Preparatory and Analytical Method

Samples will be prepared and analyzed using ETS-8-154: "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates In Water By Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry". Extracted calibration standards will be prepared in a similar matrix (Milli Q or matrix matched water) to be determined along with the QC samples.

All containers used for water sample collection will be shipped to the sample location. A field duplicate and at least two levels of field spikes of each sample will be collected. Some sites may have additional field spikes to bracket the possible concentration ranges of analytes in a location. The field spikes will contain PFBA, PFOA, PFBS, PFHS and PFOS. The field spikes will be used to determine the precision and accuracy of the method for each site sampled. Laboratory spikes and laboratory duplicates will not be a component of this study but, if necessary, can be done with approval or by request of the study director.

Recoveries of field matrix spikes (FMS) are anticipated to be between 70% and 130% of the fortified levels. If the recoveries of the field spikes fall outside the acceptable range, the sample result will be reported as "NR" (not reported due to QC failures).

For each extraction batch at least three method blanks will be prepared. In addition, triplicate lab control spikes at a minimum of two different concentrations are to be performed with each extraction batch.

9.2 Methods for Control of Bias

Control of bias is accomplished using laboratory control samples (as indicators of method accuracy and precision) and blanks. Laboratory control samples are used to determine the overall data uncertainty for each batch. Milli Q or matrix matched water will be used to prepare the laboratory control samples. Triplicate laboratory control samples at a minimum of two different concentrations are to be performed with each extraction batch. The relative standard deviation of the pooled recoveries must be less than or equal to 20% and the average recovery must be 80-120%. Low lab control spikes will be prepared at concentrations approximately five to ten times higher than the targeted LOQ and high laboratory control spikes will be prepared at concentrations near the mid-point of the calibration curve.

For each analytical batch of samples, solvent and method blanks will be analyzed. Method blanks must be done with each extraction batch. A range of three to seven method blanks must be prepared interspersed throughout the extraction batch and analyzed throughout the analytical sequence. The mean area count for each analyte in the method blanks must be less than 50% of the area count of the LOQ standard. If the mean area counts of the method blanks exceed 50% of the LOQ standard, then the LOQ must be raised to the first standard level in the curve that meets criteria. Alternatively, the method blanks could be evaluated statistically to determine outliers or technical justification to eliminate a blank may be made when determining the LOQ. The criteria are then applied again to evaluate method blank compliance. If any method blanks are removed from the LOQ determination, document in the report or the raw data as appropriate.

10. Data Quality Objectives

Method performance criteria will be demonstrated for each analytical batch in accordance with ETS-8-154. The appropriate laboratory control samples prepared with each set of samples should verify the method's accuracy is 100% ± 20%, and the method's precision is within ±20% relative standard deviation.

11. Statistical Analysis

Averages and standard deviations will be calculated. Additional calculations are provided in ETS-8-43.

12. Report

A report of the results of the study will be prepared by 3M Environmental Laboratory. The report will include, but not be limited to, the following, when applicable:

12.1 Name and address of the facility performing the study,

12.2 Dates upon which the study was initiated and completed.
12.3 A statement of compliance by the Study Director addressing any exceptions to Good Laboratory Practice Standards.
12.4 Objectives and procedures as stated in the approved protocol, including any amendments to the original protocol.
12.5 The test substance identification by name, chemical abstracts number or code number, strength, purity, and composition or other appropriate characteristics, if provided by the Sponsor.
12.6 Stability and the solubility of the test substances under the conditions of administration, if provided by the Sponsor.
12.7 A description of the methods used to conduct the test(s).
12.8 A description of the test system.
12.9 A description of any circumstances that may have affected the quality or the integrity of the data.
12.10 The name of the Study Director and the names of other scientists, professionals, and supervisory personnel involved in the study.
12.11 A description of the transformations, calculations, or operations performed on the data, a summary and analysis of the analytical chemistry data, and a statement of the conclusions drawn from the analyses.
12.12 Statistical methods used to evaluate the data, if applicable.
12.13 The signed and dated reports of each of the individual scientists or other professionals involved in the study, if applicable.
12.14 The location where raw data and the final report are to be stored.
12.15 A statement prepared by the Quality assurance unit listing the dates that study inspections and audits were made and the dates of any findings reported to the Study Director and Management.
12.16 If it is necessary to make corrections or additions to a final report after it has been accepted, the changes will be made in the form of an amendment issued by the Study Director. The amendment will clearly identify the part of the final report that is being amended, the reasons for the amendment, and will be signed by the Study Director.

13. Location of Raw Data, Records, and Final Report

Original data or copies thereof, will be available at 3M Environmental Laboratory to facilitate audits of the study during its progress and before acceptance of the final report. When the final report is completed, all original paper data, including those items listed below will be retained in the permanent record archives of 3M Environmental Laboratory following signing of the report

13.1 The following raw data and records will be retained in the study folder in the archives according to 3M Environmental Laboratory Standard Operating Procedures.
- **13.1.1** Approved protocol and amendments
- **13.1.2** Study correspondence
- **13.1.3** Shipping records
- **13.1.4** Raw data
- **13.1.5** Approved final report (original signed copy)

13.1.6 Electronic copies of data

13.2 The following supporting records will be retained separately from the study folder in the archives according to 3M Environmental Laboratory Standard Operating Procedures:

13.2.1 Training records
13.2.2 Calibration records
13.2.3 Instrument maintenance logs
13.2.4 Standard Operating Procedures, Equipment Procedures, and Methods
13.2.5 Appropriate specimens

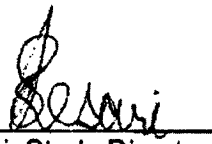
14. DATA / SAMPLE RETENTION

A portion of the test substance used in this study will be retained for a period not less than 10 years following the application of the final test rule. The extracts will only be retained so long as their quality affords evaluation.

15. PROTOCOL AMENDMENTS AND DEVIATIONS

Planned changes to the protocol will be in the form of written amendments signed by the Study Director and the Sponsor's Representative. Amendments will be considered as part of the protocol and will be attached to the final protocol. Any other changes (unplanned) will be in the form of written deviations, signed by the Study Director and filed with the raw data. All changes to the protocol and the reason for the changes will be indicated in the final report.

16. SIGNATURES



4/17/07

Jai Kesari, Study Director Date

Gary A. Hohenstein, Sponsor Representative Date

04/11/07

William K, Reagen, 3M Environmental Laboratory Management Date

13.1.6 Electronic copies of data

13.2 The following supporting records will be retained separately from the study folder in the archives according to 3M Environmental Laboratory Standard Operating Procedures:

13.2.1 Training records
13.2.2 Calibration records
13.2.3 Instrument maintenance logs
13.2.4 Standard Operating Procedures, Equipment Procedures, and Methods
13.2.5 Appropriate specimens

14. DATA / SAMPLE RETENTION

A portion of the test substance used in this study will be retained for a period not less than 10 years following the application of the final test rule. The extracts will only be retained so long as their quality affords evaluation.

15. PROTOCOL AMENDMENTS AND DEVIATIONS

Planned changes to the protocol will be in the form of written amendments signed by the Study Director and the Sponsor's Representative. Amendments will be considered as part of the protocol and will be attached to the final protocol. Any other changes (unplanned) will be in the form of written deviations, signed by the Study Director and filed with the raw data. All changes to the protocol and the reason for the changes will be indicated in the final report.

16. SIGNATURES

Jai Kesari, Study Director Date

Gary A. Hohenstein April 17, 2007

Gary A. Hohenstein, Sponsor Representative Date

William K. Reagen, 3M Environmental Laboratory Management Date

ATTACHMENT B: TEST PROTOCOL AMENDMENT

CONFIDENTIAL

3M Protocol E07-0149
Amendment #2

Study Title

Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility"

PROTOCOL AMENDMENT NO. 2

Amendment Date:
November 19, 2008

Performing Laboratory
3M Environmental, Health, and Safety Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Laboratory Project Identification
E07-0149

CONFIDENTIAL

3M Protocol E07-0149
Amendment #2

This amendment modifies the following portion of protocol:

Analysis of Perfluorobutanoic Acid (PFBA), Perfluorooctanoic Acid (PFOA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) in Groundwater Using LC/MS/MS for the "3M Fluorochemical (FC) Assessment Work Plan for the 3M Cordova, IL Facility"

PROTOCOL READS:

6. REFERENCE SUBSTANCE

Reference Substance	PFBA	PFOA	PFBS
Chemical Name	Perfluorobutanoic Acid	Perfluorooctanoic Acid	Perfluorobutanesulfonate
Chemical Formula	$C_3F_7COO^-$	$C_7F_{15}COO^-$	$C_4F_9SO_3^-$
Identifier	Acid CAS# 375-22-4	Ammonium Salt CAS# 3825-26-1	Potassium Salt CAS# 29420-49-3
Source	Aldrich	3M	3M
Expiration Date	02/16/2017	02/27/2017	01/18/2017
Storage Conditions	Frozen	Frozen	Frozen
Chemical Lot Number	09713EC	332	2
TCR Number	TCR-757	TCR-123	TCR-282
Physical Description	Liquid, room temp	White powder	Good/white crystals
Purity	98.7%	95%	97.3%
Solubility	No information available	>20%	54,400 ppm

Reference Substance	PFHS	PFOS
Chemical Name	Perfluorohexanesulfonate	Perfluorooctanesulfonate
Chemical Formula	$C_6F_{13}SO_3^-$	$C_8F_{17}SO_3^-$
Identifier	Potassium Salt CAS# 3871-99-6	Potassium Salt CAS# 2795-39-3
Source	3M	3M
Expiration Date	02/12/2017	08/31/2016
Storage Conditions	Frozen	Frozen
Chemical Lot Number	NB 120067-69	217
TCR Number	TCR-083	TCR-1166
Physical Description	White powder	White powder
Purity	98.6%	86.9%
Solubility	No information available	680 ppm

AMEND TO READ:

6. REFERENCE SUBSTANCE

Reference Substance	PFBA	PFOA[1]	PFBS
Chemical Name	Perfluorobutanoic Acid	Perfluorooctanoic Acid	Perfluorobutanesulfonate

Reference Substance	PFHS	PFOS[1]
Chemical Name	Perfluorohexanesulfonate	Perfluorooctanesulfonate

(1) Two reference substances will be used for PFOA and PFOS, one containing primarily linear isomers, and the second containing branched isomers.

REASON:
To allow for the use of reference substances that may be identified by TCR number or lot number other that those stated in the protocol.

PROTOCOL READS:

9.1. Preparatory and Analytical method
Samples will be prepared and analyzed using ETS-8-154: "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates In Water By Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry".

AMEND TO READ:

9.1. Preparatory and Analytical method
Samples will be prepared and analyzed by LC/MS/MS following ETS 8-154.3 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates In Water By Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry".

Alternately, samples may be analyzed by ETS-8-044.0 "Determination of Perfluorinated Compounds In Water by High Performance Liquid Chromatography/Mass Spectrometry Direct Injection Analysis" or ETS-8-110.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water, Soil and Sediment by LC/MS/MS".

REASON:
Method ETS-8-154 is typically used for samples that contain perfluorinated compounds in the concentration range from 0.025 ng/mL to 25 ng/mL. Previous analysis of groundwater samples from the Cordova area (Interim report #1) produced sample results at levels that were greater than 25 ng/mL. The reason for this amendment is to allow for the use of methods ETS-8-110 and ETS-8-044, which are better suited for the analysis of samples of varying concentrations.

CONFIDENTIAL

3M Protocol E07-0149
Amendment #2

Amendment Approval

Gary Hohenstein, Sponsor Representative — 11/20/09 Date

Susan Wolf, Principal Investigator — 11/19/2009 Date

William Reagen, EHS Opns Environmental Lab Management — 19 Nov 2009 Date

Jaisimha Kesari P.E., DEE, Study Director — 12/3/09 Date

ATTACHMENT C: EXTRACTION AND ANALYTICAL METHODS

3M Environmental Laboratory

Method

Analysis of Fluorochemicals in Extracts Using HPLC-Electrospray/Mass Spectrometry/Mass Spectrometry

Method Number: ETS-8-110.01

Adoption Date: 08 Sept 1999

Revision Date: Upon signing

Effective Date: 10/13/04

Approved By:

William K. Reagen
Manager

09/10/04
Date

1 Scope and Application

The method describes the analysis of fluorochemicals (typically perfluoronated acids, alcohols, amides, and sulfonates) using HPLC-mass spectrometry/mass spectrometry.

Specific analytes, ions (parent and daughter) matrices, solvents, solutions, quality controls, internal standards(s), and other parameters will be defined in the protocol or the sample preparation worksheet(s).

Applicable Compounds: Electrospray ionizable fluorochemicals.

Matrices: To include, but not limited to, water, aqueous solutions, organic solvents, soils, tissues, and serum. The matrix will be defined by the protocol or sample preparation worksheet(s).

2 Method Summary

This method describes the analysis of electrospray ionizable fluorochemicals using HPLC-electrospray tandem mass spectrometry (HPLC-ES/MS/MS). The analysis is performed by following the mass selection of a single ion characteristic of a particular fluorochemical, such as the perfluorooctanesulfonate (PFOS) anion, m/z = 499 or perfluorooctanoate (PFOA), m/z = 413. A compound specific product ion is created through high-energy collisions, and the creation of the product ion is quantitatively monitored.

For example, the transition from 499 > 99 (characteristic of FSO_3^-) is monitored for PFOS and the 413 > 119 (characteristic of $C_2F_5^-$) transition is monitored for PFOA. This selectivity afforded by this technique reduces background interferences and enhances analytical sensitivity.

3 Definitions

3.1 Atmospheric Pressure Ionization (API):

Commercially available LC/MS and LC/MS/MS systems allow for various methods of ionization by utilizing a variety of sources, probes, and interfaces. These include but are not limited to: Electrospray Ionization (ESI), Atmospheric Pressure chemical Ionization (APcI). The ionization in these processes occurs at atmospheric pressure (i.e., not under a vacuum).

3.2 Electrospray Ionization (ES, ESI):

A method of ionization performed at atmospheric pressure, whereby ions in solution are transferred to the gas phase via tiny charged droplets. These droplets are produced by the application of a strong electrical field. Typically, this is for the analysis of fluorochemicals.

3.3 Mass Spectrometer (MS) or Tandem Mass Spectrometer (MS/MS):

Single mass spectrometer (MS) or Tandem mass spectrometers (MS/MS) systems are equipped with quadrupole mass selective detectors. Ions are selectively discriminated by mass to charge ratio (m/z) and subsequently detected. A single MS may be employed for ion detection or tandem MS (MS/MS) may be employed to obtain specific fragmentation information.

4 Warnings and Cautions

4.1 Health and Safety

Always wear appropriate personal protective equipments such as protective gloves, eye protection, and appropriate clothing when working with biological matrices, solvents, chemicals

and instrumentation. For potential hazard information refer to material safety data sheets, packing material, the 3M Environmental Laboratory's Chemical Hazard Review, the 3M Guide to Laboratory Practices or other information as appropriate.

4.2 Cautions

The analyst must be familiar with the laboratory equipment and potential hazards including, but not limited to, the use of biological materials, solvents, high temperatures, pressurized gas and solvent lines, high voltage, and vacuum systems. Refer to the appropriate equipment procedure or operator manual for additional information and cautions.

5 Interferences

To minimize interferences when analyzing samples, use containers and instruments parts that will not contaminate the sample(s) or extract(s).

Co-extracted matrix components may enhance or suppress the measured analyte signal in the mass spectrometer. These effects are minimized using an SPE technique, but the precision and accuracy of spike results must be evaluated for possible effects of co-extracted matrix interferences that may be present.

6 Instrumentation, Supplies, and Materials

6.1 Instrumentation

Equipment listed below may be modified in order to optimize the system. Document the actual configuration in the raw data and in the Analytical Equipment Systems documentation or other equivalent documentation system.

Agilent HPLC 1100 system:

Pump: Quaternary Pump, Agilent®, Model G1311A, or Binary Pump, Agilent®, Model 1312 or equivalent.

Solvent Degasser, Agilent®, Model G1322A or equivalent.

Autosampler, Agilent®, Model G1313A, or Thermostated Autosampler, Agilent®, Model G1329A or equivalent.

Autosampler thermostat (used with the Thermostated Autosampler), Agilent®, Model G1330A or equivalent.

Column Compartment (Temperature Controlled), Agilent®, Model G1316A or equivalent.

Diode Array Detector, (DAD) Agilent®, Model # G1315A or equivalent.

Controller, Hand Held, Agilent®, Model # G1323A or equivalent.

Ultima™, Tandem MS, Micromass®, or Quattro II, Tandem MS, Micromass®, or equivalent.

Capillary Pump, Waters®, Model CAPLC, or equivalent.

Capillary Autosampler, Spark-Holland, Model SPF, or equivalent.

Computer Hard Drive, Compaq® AP200 or better, or equivalent.

System control/data analysis software: MassLynx™, Version 3.4 or better, or equivalent.

Other instrumentation as needed, document as appropriate.

6.2 Supplies

Tubing: PEEK, stainless steel, etc.

Ferrules and other connective parts.

Autovials and caps.

6.3 Materials

High purity grade nitrogen gas regulated to approximately 100 psi (or other suitable gas system).

High purity grade collision gas, argon.

HPLC analytical columns.

Guard Columns.

Crimpers

7 Reagents and Standards

7.1 Reagents

Methanol, HPLC grade or equivalent.

ASTM Type I Water or equivalent. All water used in this method should be ASTM Type I or equivalent, and may be provided by a Milli-Q TOC Plus System or by another system or vendor, provided there is adequate documentation of the source.

Ammonium Acetate, reagent grade or equivalent. When preparing different amounts than those listed, adjust accordingly.

2.0 mmol/L ammonium acetate solution. Weigh approximately 0.300 g of ammonium acetate to a 2 L volumetric flask. Add water to the mark and mix until all solids are dissolved. Store at room temperature.

Other solvents and solutions may be used as stated in the protocol or in the sample preparation worksheet(s). Document all solvents and solutions used in the appropriate logs or media.

7.2 Calibration Standards

Typically, a minimum of 6 standards are analyzed with each group of samples.

8 Sample Handling

Standards, sample extracts, and prepared samples may be stored in capped autovials, capped 15 mL centrifuge tube, or other suitable containers until analysis.

If analysis will be delayed, standards, sample extracts, and prepared samples may be refrigerated at approximately 4°C until the analysis can be performed. Appropriate sample storage conditions will be evaluated on a case-by-case basis, and will be addressed in the protocol or by the analyst in the raw data and/or prep worksheets.

9 Quality Control

The following quality control is recommended for this method. If an approved protocol or extraction procedure is used for the samples, any quality procedures described therein takes precedence over the quality control in this method. For non-GLP studies without protocols or

extraction methods, document the chosen quality control on the sample preparation worksheets and or the final report.

9.1 Blanks

9.1.1 Solvent Blank

Solvent blanks are not extracted. They are analyzed with each sample set to determine contamination or carryover. The solvent used should, in some approximation, represent the solvent used for the standard curve and/or the sample extracts. Solvent blank area counts should be less than 50% of the area count of the lowest calibration standard.

Solvent blanks should be analyzed prior to each calibration curve. If carryover is a problem consecutive solvent blanks may be necessary. Solvent blank area counts should be less than 50% of the area count of the lowest calibration standard prior to the injection of unknown samples.

9.1.2 Method Blank

Method blanks are extracted and analyzed with each group of samples, consisting of water or another commercially available solvent.

The protocol or project lead will define the number and frequency of these samples. Method blanks should have area counts less than 50% of the area count of the lowest calibration standard.

9.1.3 Matrix Blank

Matrix blanks are extracted and analyzed with each group of samples. These groups would include tissue and sera samples. Endogenous levels of target analytes contained in the sample matrix are evaluated by taking an aliquot of blank (unexposed) matrix and carrying it through the entire extraction process alongside the samples.

The protocol or project lead will define the number and frequency of these samples. Matrix blanks should have area counts less than 50% of the area count of the lowest calibration standard. If this is not possible in cases where there are detectable levels of endogenous target analyte present in the matrix, then the endogenous levels should be taken into account when evaluating sample data and discussed in the raw data.

9.2 Surrogate

A surrogate standard may be used for quality control. The surrogate is used to qualitatively evaluate the entire analytical procedure including sample preparation and analysis. The surrogate should be spiked at the beginning of the sample preparation and should fall within the low to mid-range of the calibration curve.

9.3 Post-Extraction Control Sample

Post-extraction control samples may be prepared for each set of extracted samples (if applicable to the study) and analyzed to determine extraction stability. The samples are prepared by adding additional target analyte to the final extract, and after any dilution.

Post-extraction control sample duplicates may be prepared periodically to measure the precision associated with the analysis. These samples should be analyzed in the same run as the unspiked sample.

Post-extraction control sample concentrations should fall in the mid-range of the initial calibration curve or should be prepared at 1.5-5 times the endogenous concentration of the analyte. Spike concentrations should fall in the low-range of the initial calibration curve if extremely low levels are expected

10 Calibration and Standardization

10.1 Instrument Calibration

Analyze the standard curves prior to each set of samples. The standard curve may be plotted by linear regression ($y = mx + b$) or quadratic fit ($y = ax^2 + bx + c$); weighted 1/x or unweighted, using MassLynx™ or other suitable software. The calibration curves may include but should not be forced through zero. The mathematical method used to calculate the calibration curve should be applied consistently throughout a study. Any change should be thoroughly documented in the raw data.

If the calibration curve does not meet acceptance criteria perform routine maintenance or prepare a new standard curve (if necessary) and reanalyze.

For purposes of accuracy when quantitating low levels of analyte, it may be necessary to use the low end of the calibration curve rather than the full range. Example: when attempting to quantitate approximately 10 ppb of analyte, generate a calibration curve consisting of the standards from 5 ppb to 100 ppb rather than the full range of the curve (5 ppb to 1000 ppb). This will reduce inaccuracy attributed to linear regression weighting of high concentration standards.

High and/or low points may be excluded from the calibration curves to provide a better fit over the linear range appropriate to the data or because they did not meet the pre-determined acceptance criteria. Low-level curve points should also be excluded if their area counts are not at least twice that of the method blanks. Any curve point may be rejected due to a bad injection or if a possible prep error can be deduced by the raw data. Justification for exclusion of calibration curve points will be noted in the raw data.

A minimum of 6 points will be used to construct the calibration curve.

10.2 Internal Standard

An internal standard (IS) may be used to quantify the target analytes by establishing a relationship between the ratio of analyte response to IS response and a known concentration of the analyte of interest. The IS should be spiked at an amount that will fall within the mid-range of the calibration curve. The IS should be added after the extraction process and before analysis

10.3 Continuing Calibration Verification (CCV)

Continuing calibration verifications (CCV) are analyzed to verify the continued accuracy of the calibration curve.

Analyze a mid-range calibration standard (at a minimum) after every tenth sample, not including solvent blanks, with a minimum of one per sample set. Calibration verification injections must be within ±25% to be considered acceptable. The calibration curve and the last passing CCV will then bracket acceptable samples. Multiple CCV levels may be used. The CCVs used should be documented in the raw data.

11 Procedures

For instrument set-up, refer to the manual that pertains to the specific instrument. Actual parameters will be recorded on the instrument printouts. The procedures that follow are from Micromass® instruments using MassLynx™. Conditions may be adjusted, provided adequate QC is used.

11.1 Tune Page

The Tune Page controls many of the MS system parameters. Open up the Tune Page and review the parameters, change if necessary. Selected parameters and their recommended initial settings are as follows:

Tune Page Initial Conditions:

Source	Setting	Analyzer	Setting
Capillary	2	LM Res 1	15
Cone	20	HM Res 1	15
Source Block Temp	150	Ion Energy (Ienergy) 1	2
Desolvation Temp	250	LM Res 2	14
Analyzer	Setting	HM Res 2	14
Multiplier	650	Ion Energy (Ienergy) 2	2

Ensure that the instrument is operating and that the appropriate gases are on.

11.2 Inlet Editor

The Inlet Editor contains all of the parameters for the LC. Open the Inlet Editor, use the existing method or load a different method. Review the parameters and change if necessary. Selected parameters and their recommended initial settings are as follows:

LC Pump Initial Conditions:

Parameter	Setting
Solvent A	90
Solvent B	10
Flow Rate (mL/min)	0.300
Stop Time (mins)	10
Max Pressure (bar)	400
Oven Temp Left (°C)	30
Oven Temp Right (°C)	30

Gradient Initial Conditions:

Time	B%
0.00	10%
1.00	10%
5.50	95%
7.50	95%
8.00	10%

Autosampler Initial Conditions:

Parameter	Setting
Draw Speed	200.0
Eject Speed (uL/min)	200
Draw Position (mm)	0.00
Stop Time (mins)	10.00
Injection Volume (uL)	10.0

It is recommended, for consistency, that the aqueous solution is in Pump A and the organic solvent is always in Pump B.

Save the method with an appropriate method name.

Check the solvent levels in the reservoirs and refill if necessary.

Load the method and turn on the solvent flow.

11.3 Function list editor

Create a MS acquisition method appropriate for the data needed. See the Micromass® MassLynx™ "Guide To Data Acquisition" for additional information. Selected parameters and their recommended initial settings follow. Ion information is not listed due to the numerous possible ions of interest and their daughter ions.

MS Initial Conditions:

Parameter	Setting
Solvent Delay	0.00
Mode	ESP-
Interchannel Delay (Secs)	0.07
Span (Daltons)	0.00
Start Time (Mins)	0.00
End Time (Mins)	10.0
Repeats	1

12 Data Analysis and Calculations

Calculations (including, but not limited to):

Calculate matrix spike percent recoveries using the following equation:

$$\%\,Recovery = \frac{Observed\ Result - Background\ Result}{Expected\ Result} * 100$$

Calculate percent difference using the following equation:

$$\%Difference = \frac{Calculated\ Concentration - Expected\ Concentration}{Expected\ Concentration} * 100$$

13 Method Performance

Criteria listed in a protocol will supercede this method. Document any changes from the method in the protocol, raw data and/or the report.

If criteria listed in this method performance section are not met, maintenance may be performed on the system and samples reanalyzed or other actions as determined by the analyst. Document all actions in the raw data.

If data is to be reported when performance criteria have not been met, the data must be footnoted on tables and discussed in the text of the report.

13.1 System Suitability

A minimum of three system suitability samples will be injected at the beginning of each analytical run. Typically these samples are run prior to the calibration curve. The system suitability injections must have area counts with an RSD of ≤5% and a retention time RSD of ≤2%.

13.2 Quantitation

The coefficient of determination (r^2) value for the calibration curve must be greater than or equal to 0.990. Each point in the curve must be within ±25% of the theoretical concentration with the exception of the LLOQ, which may be within ±30%.

13.3 Lower Limit of Quantitation

The Lower Limit of Quantitation (LLOQ) is analyte and matrix specific. For most analytes, the LLOQ concentration is selected as the lowest acceptable non-zero standard in the calibration curve.

Solvent, matrix and method blank area counts must be < ½ that of the lowest standard used in the calibration curve. This is not always possible for matrix blanks, where there may be detectable levels of target analytes. The endogenous levels, if any should be taken into account when evaluating sample data and discussed in the raw data.

14 Pollution Prevention and Waste Management

Sample extract waste and flammable solvent is disposed in high BTU containers, and glass pipette waste is disposed in broken glass containers located in the laboratory.

15 Records

Record data per the laboratory's standard operating procedures.

16 Attachments

None.

17 References

ETS-9-024, "Operation and Maintenance of Micromass® Triple Quadrupole Mass Spectrometers fitted with Atmospheric Pressure Ionization Source".

18 Affected Documents

None.

19 Revisions

Revision Number	Summary of Changes
1	Updated to the new format. Changed the title to allow for more types of matrices to be tested. Revised the method to include the new requirements of the new format. Removed or modified the definitions in Section 3.

3M Environmental Laboratory

Method

Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates In Water By Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry

Method Number: ETS-8-154.3

Adoption Date: 28 Apr 2000

Revision Date: Upon Signing

Effective Date: 08/24/07

Approved By:

William K. Reagen
Manager

08/24/07
Date

1 Scope and Application

This method describes the extraction of target analytes from water matrices using solid phase extraction (SPE) followed by separation, identification, and quantitation using high-performance liquid chromatography mass spectrometry (HPLC/MS) or tandem mass spectrometry (HPLC/MS/MS). This method has been validated for perfluorooctane sulfonate (PFOS), perfluorooctane sulfonylamide (FOSA), and perfluorooctanoate (PFOA) in groundwater, surface water, and drinking water samples. This method is considered a ***performance-based*** method and may be applied to the determination of other perfluorinated acids, alcohols, amides, and sulfonates in similar matrices, as long as the defined QC elements are satisfied and with the understanding that the method is not validated for compounds outside the scope of the original protocol.

This method is based in part on the 3M Environmental Laboratory report E01-0454, "Method of Analysis for the Determination of Perfluorooctane sulfonate (PFOS), Perfluorooctane sulfonylamide (PFOSA), and Perfluorooctanoate (POAA) in Water", as developed and validated by Exygen Research (formerly Centre Analytical Laboratories, Inc.). This method was originally validated using a 40 mL sample aliquot for extraction and a final elution volume of 5 mL. These volumes may be changed on a per project basis to meet the data quality objectives set forth by the project lead. Acceptable recoveries of laboratory quality control samples will demonstrate that the extraction and elution volumes do not impact the validity of the method.

Sample collection is ***not*** covered under this analytical procedure.

2 Method Summary

Water samples are collected from a site of interest and shipped to the analytical facility. Perfluorinated acids, alcohols, amides, and sulfonates are extracted from aliquots of the water samples using solid phase extraction (SPE) cartridges. The compounds are eluted from the SPE cartridge, using methanol. Separation, identification, and measurement are accomplished by high-performance liquid chromatography/ tandem mass spectrometry (HPLC/MS/MS) analysis. High-performance liquid chromatography/mass spectrometry (HPLC/MS) may be used if the defined QC elements are satisfied and meets the data quality objectives outlined in the general project outline for the given project.

The concentration of each identified component is measured by comparing the MS response of the quantitation ion (or summed total of multiple ions) produced by that compound to the MS response of the quantitation ion (or summed total of multiple ions) produced by the same compound in an extracted calibration standard (external standard).

3 Definitions

3.1 SPE cartridge

A column containing an open solvent reservoir, retaining frit, sorbent bed, retaining frit, and luer tip. The sorbent bed is bonded silica which is designed to selectively retain or elute the compounds of interest depending on the solvent conditions. The compounds of interest can be separated from the water matrices and introduced into an appropriate solvent for analysis.

3.2 Analytical Sample

A portion of an extracted laboratory sample prepared for analysis.

3.3 Calibration Standard

An aqueous solution prepared by spiking a known volume of the Working Standard (WS) into a predetermined amount of ASTM type I water, HPLC grade reagent water, or other suitable water (i.e. matrix water), and extracting the solution according to this method. The calibration standard solutions are used to calibrate the instrument response with respect to analyte concentration.

3.4 Laboratory Duplicate Sample (LDS)

A laboratory duplicate sample is a separate aliquot of a sample, taken in the analytical laboratory that is extracted and analyzed separately with identical procedures. Analysis of LDSs compared to that of the first aliquot give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.5 Field Blank (FB)/Trip Blank

ASTM Type I water, HPLC grade reagent water, or other suitable water, is placed in a sample container in the laboratory and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation and all analytical procedures. The purpose of the FB is to determine if test substances or other interferences are present in the field environment. This sample is also referred to as a Trip Blank.

3.6 Field Duplicate Sample (FDS)

A sample collected in duplicate at the same time from the same location as the sample. The FDS is placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analysis of the FDS compared to that of the first sample gives a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.7 Field Matrix Spike (FMS)

A sample to which known quantities of the target analytes are added to the sample bottle in the field or in the laboratory before the bottles are sent to the field. A known, specific volume of sample must be added to the sample container without rinsing. This may be accomplished by making a "fill to this level" line on the outside of the sample container. The FMS should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. If the expected range of analyte concentrations is unknown, multiple spikes at varying levels may be prepared to increase the likelihood that a spike at an appropriate level is made. Typically a low and a high spike are prepared for each sampling location. The FMS is analyzed to ascertain if matrix effects or sample holding time contributes bias to the analytical results.

3.8 Field Spike Control Sample (FSCS)/Trip Blank Matrix Spike

An aliquot of ASTM Type I water (HPLC grade reagent water or other suitable water may used) to which known quantities of the target analytes are added in the field at the time of sample collection (at an appropriate concentration to be determined by the project lead) or in the laboratory prior to the shipment of the collection bottles. The FSCS is extracted and analyzed exactly like a sample to determine whether a loss of analyte could be attributed to holding time, sample storage and/or shipment issues. A low and high FSCS may be appropriate when expected sample concentrations are not known. This sample may also be referred to as Trip Blank Matrix Spike.

3.9 Laboratory Control Sample (LCS)

An aliquot of ASTM Type I water (HPLC grade reagent water or other suitable water may used) to which known quantities of the target analytes are added in the laboratory at the time of sample extraction. At least two levels are included, one generally at the low end of the calibration curve

and one near the mid to upper range of the curve. The LCSs are extracted and analyzed exactly like a laboratory sample to determine whether the method is in control. LCSs should be prepared each day samples are extracted.

3.10 Laboratory Matrix Spike (LMS)

A laboratory matrix spike is an aliquot of a sample to which known quantities of target analytes are added in the laboratory. The LMS is extracted and analyzed exactly like a laboratory sample to determine whether the sample matrix contributes bias to the analytical results. The endogenous concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LMS corrected for these concentrations.

3.11 Laboratory Sample

A portion or aliquot of a sample received from the field for testing.

3.12 Limit of Quantitation (LOQ)

The lower limit of quantitation (LLOQ) for a dataset is the lowest concentration that can be reliably quantitated within the specified limits of precision and accuracy during routine operating conditions. To simplify data reporting, the LLOQ is generally selected as the lowest non-zero standard in the calibration curve that meets method criteria. Sample LLOQs are matrix-dependent.

The upper limit of quatitation (ULOQ) for a dataset is the highest concentration that can be reliably quantitatied within the specified limits of precision and accuracy during routine operating conditions. The highest standard in the calibration curve that meets method criteria is defined as the ULOQ.

3.13 Method Blank

An aliquot of ASTM Type I water (HPLC grade reagent water or other suitable water may used) that is treated exactly like a laboratory sample including exposure to all glassware, equipment, solvents, and reagents that are used with other laboratory samples. The method blank is used to determine if test substances or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.14 Sample

A sample is a small portion collected from a larger quantity of material intended to represent the original source material.

3.15 Stock Standard Solution (SSS)

A concentrated solution of a single analyte prepared in the laboratory with an assayed reference compound.

3.16 Surrogate

A compound similar to the target analyte(s) in chemical composition and behavior that is not normally found in the sample(s). A surrogate compound is typically a target analyte with at least one atom containing an isotopically labeled substitution. If used, surrogate(s) are added to all samples and quality control samples (except solvent blanks and half of the prepared method blanks). Surrogate(s) are added to quantitatively evaluate the entire analytical procedure including sample collection, extraction, and analysis. Inclusion of a surrogate analyte is an optional quality control measure and is NOT required. The project lead should indicate in the general project outline/protocol whether or not a surrogate compound will be part of the analysis.

3.17 Working Standard (WS)

A solution of several analytes prepared in the laboratory from SSSs and diluted as needed to prepare calibration standards and other required analyte solutions.

4 Warnings and Cautions

4.1 Health and Safety

The acute and chronic toxicity of the standards for this method have not been precisely determined; however, each should be treated as a potential health hazard.

Unknown samples may contain high concentrations of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure.

The laboratory is responsible for maintaining a safe work environment and a current awareness of local regulations regarding the handling of the chemicals used in this method. A reference file of material safety data sheets (MSDS) should be available to all personnel involved in these analyses.

4.2 Cautions

The analyst must be familiar with the laboratory equipment and potential hazards including, but not limited too, the use of solvents, pressurized gas and solvent lines, high voltage, and vacuum systems. Refer to the appropriate equipment procedure or operator manual for additional information and cautions.

5 Interferences

During extraction and analysis, be aware of potential contaminant sources from reagents and solid phase extraction devices.

All materials used in the analyses shall be demonstrated to be free from interferences under conditions of analysis by running method blanks.

Parts and supplies that contain Teflon® should be avoided or minimized due to the possibility of interference and/or contamination. These may include, but are not limited to: wash bottles, Teflon® lined caps, autovial caps, HPLC parts, etc.

The use of disposable micropipettes or pipettes to aliquot standard solutions is recommended to make calibration standards and matrix spikes.

6 Instrumentation, Supplies, and Materials

Note: Brand names, suppliers, and part numbers are for illustrative purposes only. Equivalent performance may be achieved using apparatus and materials other than those specified here. Demonstration of equivalent performance (quality control samples meeting method acceptance criteria) is the responsibility of the laboratory performing the analysis.

6.1 Instrumentation

Balance, analytical (display at least 0.0001g), Mettler

HPLC/MS/MS or HPLC/MS system, as described in Section 10.

6.2 Supplies and Materials.

Sample collection bottles—LDPE (e.g., Nalgene™) narrow-mouth bottles with screw cap. **Note:** Do not use Teflon bottles or Teflon lined caps.

Coolers or boxes for sample shipment.

AutoTrace automated SPE workstation, Caliper Life Sciences. A manual SPE manifold with vacuum pump may be used if desired. Supplies for the manual SPE manifold are included below.)

Vacuum pump, Büchi.

Visiprep vacuum manifold, Supelco.

15mL disposable polypropylene centrifuge tubes, VWR.

50mL disposable polypropylene centrifuge tubes, VWR

Sep Pak Vac 6cc (1g) tC18 cartridges (part # WAT 036795), Waters. (Cartridges with other amount or types of sorbent material may be used depending on the data quality objectives of the project.)

15 mL disposable culture tubes (17 x 100 mm), VWR (Cat. No. 60818-626).

Disposable micropipettes (50–100µL, 100–200µL), Drummond.

Class A pipettes and volumetric flasks, various.

LDPE narrow-mouth bottles, Nalgene (volumes may vary depending on analytical needs).

2 mL clear HPLC vial kit (cat # 5181–3400), Agilent/Hewlett Packard.

Standard lab equipment (graduated cylinders, disposable tubes, etc.), various.

7 Reagents and Standards

Note: Suppliers and catalog numbers are for illustrative purposes only. Equivalent performance may be achieved using chemicals obtained from other suppliers. Do not use a lesser grade of chemical than those listed.

7.1 Chemicals

Methanol (MeOH), HPLC grade, JT Baker, Catalog No. JT9093–2.

Ammonium Acetate, Reagent grade, Sigma-Aldrich, Catalog No. A–7330.

ASTM Type I Water, prepared in-house (HPLC grade reagent water or other suitable water may also be used)

Sodium Thiosulfate, Reagent grade, JT Baker.

7.2 Standards

PFBA, Heptafluorobutyric Acid, (C_4 Perfluorinated Acid)

NFPA, Nonafluoropentanoic Acid (C_5 Perfluorinated Acid)

PFHA, Perfluorohexanoic Acid (C_6 Perfluorinated Acid)

TDHA, Tridecafluoroheptanoic Acid, (C_7 Perfluorinated Acid)

PFOA, Ammonium perfluorooctanoate, (C_8 Perfluorinated Acid)

C_9 Acid, Heptadecafluorononanoic Acid

PFDA, Nonadecafluorodecanoic Acid (C_{10} Perfluorinated Acid)

C_{11} Acid, Perfluoroundecanoic Acid

C_{12} Acid, Perfluorododecanoic Acid

FBSA, Perfluorobutanesulfonamide

FOSA, Perfluorooctanesulfonylamide

PFBS, Potassium Perfluorobutanesulfonate

PFHS, Perfluorohexanesulfonate

PFOS, Potassium perfluorooctanesulfonate

THPFOS, 1H, 1H, 2H, 2H-perfluorooctanesulfonic Acid

THPFDS, IH, IH, 2H, 2H-perfluorodecanesulfonic Acid

PFOA [1,2-^{13}C], ^{13}C isotopically labeled perfluorooctanoic acid, Perkin Elmer

PFOS [$^{18}O_2$], $^{18}O_2$ isotopically labeled Ammonium Perfluorooctanesulfonate

Others as required.

7.3 Reagent Preparation

250 mg/mL sodium thiosulfate solution — Dissolve 25g of sodium thiosulfate in 100 mL reagent water.

2 mM ammonium acetate solution (Analysis)—Weigh 0.3 g of ammonium acetate and dissolve in 2.0 L of reagent water.

Note: Alternative volumes may be prepared as long as the ratios of the solvent to solute ratios are maintained.

7.4 Stock Standard Solution (SSS) and Working Standard Solution Preparation

The following standard preparation procedure serves as an example. Weighed amounts and final volumes may be changed to suit the needs of a particular study. For example, µL volumes may be spiked into volumetric flasks when diluting stock solutions to appropriate levels.

100 µg/mL target analyte SSSs—Weigh out 10 mg of analytical standard ***(corrected for percent salt and purity)*** and dilute to 100mL with methanol or other suitable solvent, in a 100mL volumetric flask. Transfer to a 125mL LDPE bottle or other suitable container. Prepare a separate solution for each analyte. Expiration dates and storage conditions of stock solutions should be assigned in accordance with laboratory standard operating procedure. An example of purity and salt correction is given below for PFOS.

$$\text{salt correction factor} = \frac{\text{molecular weight of anion}}{\text{moclecular weight of salt}}$$

$$\text{PFOS } (K^+)\text{salt correction factor} = \frac{499}{538} = 0.9275$$

10 mg $C_8F_{17}SO_3^-K^+$ with purity 90% = 8.35mg $C_8F_{17}SO_3^-$ (10 mg*0.90*0.9275=8.35 mg)

1 µg/mL (1000 ng/mL) mixed working standard—Add 1.0mL each of the 100µg/mL SSSs to a 100mL volumetric flask and bring up to volume with solvent.

0.1 µg/mL (100 ng/mL) mixed working standard—Add 10.0mL of the 1.0µg/mL-mixed working standard solution to a 100mL volumetric flask and bring up to volume with solvent.

0.01 µg/mL (10 ng/mL) mixed standard—Add 10.0mL of the 0.1µg/mL-mixed working standard solution to a 100mL volumetric flask and bring up to volume with solvent.

Storage Conditions—Store all SSSs and working standards in accordance with laboratory standard operating procedure or in a refrigerator at 4°±2°C for a maximum period of 6 months from the date of preparation.

7.5 Calibration Standards

Using the working standards described above, prepare calibration solutions in ASTM Type I water, or other suitable water, using the following table as a guideline. Note: Volumes of water and working standards may be adjusted to meet the data quality objectives addressed in the general project outline. Calibration levels other than those listed below can be prepared as needed.

Concentration of WS, ng/mL	*Volume of WS, µL*	*Final Volume of Calibration Standard (mL of ASTM Type I Water, or other suitable water)*	*Final Concentration of Calibration Standard, ng/mL (ppb) in ASTM Type I Water, or other suitable water*
100	10	40	0.025
100	20	40	0.050
100	40	40	0.100
100	100	40	0.250
1000	20	40	0.500
1000	40	40	1.00
1000	100	40	2.50
10000	20	40	5.00
10000	40	40	10.0
10000	100	40	25.0

The calibration standards are processed through the entire extraction procedure (Section 11), identical to the laboratory samples. The concentration of the calibration standard in the final extract depends on the volume extracted and the final elution volume.

Final sample concentration factor = Volume extracted (mL)/Elution Volume (mL)

Storage Conditions— Store all extracted calibration standards in 15mL polypropylene tubes or in labeled autovials at 4°±2°C. After analysis, archive all extracted standards with the sample extracts in accordance with laboratory standard operating procedures.

8 Sample Collection Bottle Preparation

For most projects, sample collection bottles are prepared by 3M Environmental Laboratory personnel before they are shipped to the collection site. Typically, four separate collection bottles are associated with a single collection site: sample, field duplicate sample, low field matrix spike, and high field matrix spike. Depending on the scope of the project, additional replicates of the field sample and field matrix spikes may be added. Also, it is not uncommon for additional mid-level field matrix spikes to be collected if the expected sample concentrations are truly unknown or could span a large concentration range.

Low-density polyethylene (LDPE) wide-mouth Nalgene bottles are used for the sample collection containers. (Volumes of the bottles may vary depending on how much sample is required to meet

data quality objectives.) The interiors of the Nalgene bottles may be rinsed multiple times with acetone and methanol and allowed to dry before adding the appropriate spikes. Note: rinsing of the bottles is optional and is not required. The project lead will communicate what the final collection volume will be and a corresponding "fill to here" line is drawn on the exterior of the bottle using a permanent marker. Typically, placement of the "fill to here" line is done by using a marker template. Alternatively, bottles with pre-marked volume indicators may be purchased.

For the sample bottles designated for matrix spikes, an appropriate volume of matrix spiking solution is added to the empty bottle. The volume of spike solution added should produce the desired final concentration of target analytes once the bottle is filled with sample to the "fill to here line". (If a surrogate is included for the project, the surrogate compound should also be added to the matrix spikes). The matrix spiking solution should be prepared in a suitable solvent and contains all of the target (and surrogate if applicable) analytes; however, multiple spike solutions may be used if an appropriate mixed component standard is not available. The matrix spiking solution is often the same as the working standards used to create the calibration standards. An example of a bottle spike is given below.

"Fill to here" volume = 450 mL (A 500 mL Nalgene bottle is used)

Desired Field Spike Concentration = 0.1 ng/mL

45µL of a 1 µg/mL spiking solution (containing the target analytes) is added to the bottle and the bottle cap promptly sealed

If analysis of a surrogate analyte is included in the project objectives, the preparatory analyst should also add an appropriate volume of surrogate standard solution to all the bottles designated as samples or field duplicate samples.

All bottles should be clearly labeled to indicate its intended use as a sample/field sample duplicate, low spike, or high spike. If each location has different designated spike levels, the label should also clearly indicate the sample location designation. Generally, a set of bottles for a given collection site are then grouped together in plastic bags for organizational purposes.

For each collection event, at least one set of trip blank and trip blank matrix spikes are prepared. The number of trip blank sets required for a given project will be communicated by the project lead. For the trip blank, the surrogate spike is added to the bottle (if included) and then ASTM Type I water (HPLC grade reagent water or other suitable water may used) is added to the "fill to here" line. The bottle cap is replaced and tape may be placed around the outer edge of the cap. Trip blank matrix spikes are prepared by adding the appropriate volume of matrix spiking solution, filling the bottle to the desired volume with the appropriate water and replacing and sealing the cap.

The preparatory analyst should document bottle preparation in a Note to File or on a sample preparation worksheet. The Note to File should include the following information: date prepared, total number of bottles prepared, number of sample sites, the standard identification numbers and spike volumes used to prepare spiked bottles, the "fill to here" volume, and any other pertinent information needed for reconstructibility of the data. The Note to File will be included in the final data package for the project.

9 Quality Control and Data Quality Objectives

Analytical results of the FB, FMS, LMS, FD, and FSCS should be evaluated at the conclusion of the study to help interpret the quality of sample data. Analytical results for these control/duplicate samples must be reported with the sample data.

9.1 Solvent Blanks

Solvent blanks are analyzed with each sample set to determine contamination or carryover. In general, solvent blanks should have area counts that are less than 50% of the area count of the lowest calibration standard.

Solvent blanks should be analyzed prior to and following each calibration curve, each set of system suitability samples, and after no more than 10 unknown sample extracts. If instrument carryover is a problem, consecutive solvent blanks may be necessary. In this case, the area counts of the solvent blanks should return to <50% of the lowest calibration standard prior to the injection of further standards or samples.

9.2 Method Blanks

A method blank consists of an aliquot of ASTM Type I water (HPLC grade reagent water, or other suitable water may be used) equal in volume to the samples, and extracted in the same manner as the samples. At least five method blanks should be prepared and analyzed each day that extractions are performed for a particular study or project. Method blanks must be interspersed throughout the extraction batch and analyzed interspersed throughout the analytical sequence.

The average area counts for each analyte must be less than 50% of the area count of the LOQ standard. The standard deviation of the area counts should be calculated. A specific %RSD acceptance criteria is not specified but is assessed on a batch basis. If the mean area counts of the method blank exceed 50% of the LOQ standard, then the LOQ must be raised to the first standard level in the curve that meets criteria. Method blanks may be eliminated if technical justification can be provided (e.g. the method blank was analyzed after an unexpectedly high level sample). If any method blanks are removed from the LOQ determination, document in the raw data and report as appropriate.

9.3 Laboratory Sample Replicates / Field Duplicate Sample

Depending on the scope of the project, all or selected samples may be extracted at least in duplicate, and in triplicate if difficulties were encountered in the sampling and/or holding conditions of the samples. If field sample replicates are collected, duplicate and triplicate extractions of an individual sample may not be required. The relative percent difference (RPD) of duplicate samples or relative standard deviation (RSD) should be less than 20% for the precision of sample preparation and analysis to be considered in control. Replicate samples not meeting the 20% method criteria will be reviewed and evaluated on a case by case basis.

9.4 Laboratory Matrix Spikes (LMSs)

LMSs are performed if FMSs have previously been performed for the sample matrix. Alternatively, LMSs are performed for a sample matrix if the FMS levels were not appropriate for determining spike recoveries relative to endogenous levels. Generally, each sample location represents a different sample and sample matrix. LMSs are prepared for each sample and analyzed to determine the matrix effect on spike recovery efficiency of each target analyte. Lab matrix spike recoveries should fall within ± 30% of expected values.

LMS concentrations should be prepared at approximately 0.5-10 times the endogenous concentration or approximately 4-10 times the LOQ concentration of each analyte.

9.5 Laboratory Control Spike (LCS)

Lab control spikes are prepared for each extraction batch to determine method accuracy and precision. LCSs should be prepared at a minimum of two levels and in triplicate. Low lab control spikes should be prepared at a concentration in the range of approximately four to ten times higher than the targeted LOQ and the high lab control spikes should be prepared at a concentration near the mid-point of the calibration curve. For each target analyte, the percent relative standard deviation (method precision) of the pooled control spikes must be less than or equal to 20% and the average recovery (method accuracy) must be 80-120%. Sample data for target analytes outside of the laboratory control spike acceptance criteria are not reportable.

9.6 Field Matrix Spikes (FMSs)

FMSs are prepared for each sample and analyzed to determine the matrix effect and sample holding time on spike recovery efficiency of each target analyte. Generally, each sample location represents a different sample and sample matrix.

FMS samples are a sample to which known quantities of the target analytes are added to the sample bottle in the field or in the laboratory before the bottles are sent to the field. Typically a low and a high FMS are prepared for each sampling location. The ratio of endogenous analyte to field spike concentration that is appropriate to assess accuracy is defined as approximately 0.5 to 10 times the expected sample concentration. For example, if the endogenous level of analyte in the sample is expected to be 1.0 ng/mL, the appropriate range for FMS used to assess accuracy of results would be approximately 0.5 ng/mL to 10 ng/mL. For samples that are expected to have endogenous analytes present at or below the targeted LOQ, the appropriate range for FMS would be approximately 4 to 10 times the LOQ concentration. For example, if the analyte LOQ is 0.025 ng/mL, the appropriate range for low level FMS would be 0.1 ng/mL to 0.25 ng/mL. If the expected range of analyte concentrations is unknown, multiple spikes at varying levels may be prepared to increase the likelihood that a spike at an appropriate level is made.

Field matrix spike method acceptance criteria are recoveries within ±30% of the expected value. If FMS recovery is outside of ±30% of the expected value or could not be assessed because the FMS was spiked at an inappropriate level, the sample result is reported as NR, where NR is defined as "Not Reportable". For data reportability, a sample may be re-extracted and re-analyzed or an alternate analytical method may be applied to the sample. Alternatively, resampling and reanalysis of a new sample may be completed. If re-extraction, resampling, and re-analysis fail to meet the FMS acceptance criteria, the sample result will be reported as "NR" (not reported due to noncompliant QC results).

Exceptions to the ±30% FMS acceptance criteria for data reportability are as follows:

1.) If FMS recovery could not be assessed because FMS's were at an inappropriate level, then Laboratory Matrix Spikes (LMS) may be substituted. If LMS recoveries are within ±30% the data are reportable but flagged as not meeting the FMS method acceptance criteria.

2.) If multiple FMS's were prepared on a sample and the closest FMS level to the reported sample meets the ±30% acceptance criteria but additional FMS's are outside the ±30% acceptance range, the data are reportable but flagged with an expanded uncertainty and as not meeting FMS method acceptance criteria.

3.) If the FMS recoveries are outside of the ±30% acceptance range but at least 20 acceptable historical reportable FMS sample results are available, the data may be reported but flagged with an expanded uncertainty and as not meeting FMS criteria.

10 Calibration and Standardization

10.1 Instrument Setup

Note: In this example, an Applied Biosystems Sciex API 4000 Tandem Mass Spectrometer (LC/MS/MS) is used. Other brands of LC/MS/MSs as well as single quadrupole mass spectrometers (LC/MS) may be used as long as the method criteria are met. Brand names, suppliers, part numbers, and models are for illustrative purposes only. Equivalent performance may be achieved using apparatus and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory. The operator must optimize and document the equipment and settings used.

Establish the LC/MS/MS system and operating conditions equivalent to the following:

Mass Spec: Applied Biosystems API 4000

Ion Source: Turbo Ion Spray (ABS)

Mode: Electrospray Negative

Scan Type: MRM (Multiple Reaction Monitoring)

Harvard infusion pump (Harvard Instruments), for tuning

Computer: Dell DHM

Software: Windows 2000 or Windows XP, Analyst 1.4.1

HPLC: Agilent Series 1100

Agilent Quaternary Pump

Agilent Vacuum Degasser

Agilent Autosampler

Agilent Column Oven

Note: One or more C18 HPLC analytical columns (2.1 mm x 100 mm, 5µm or 2.1 mm x 50 mm, 5µm) may be attached on-line after the purge valve and before the sample injection port to retard and separate any residue contaminants that may be in the mobile phase and/or HPLC system.

HPLC Column: Betasil C18 , 2.1mm x 100mm, 5µm (ThermoElectron Corporation)

Column Temperature: 35°C

Injection Volume: 5µL

Mobile Phase (A): 2mM Ammonium Acetate in ASTM Type I water (See 7.3)

Mobile Phase (B): Methanol

Liquid Chromatography Gradient Program.

Step Number	Total Time (min)	Flow Rate (µL/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0	300	80.0	20.0
1	1.0	300	80.0	20.0
2	14.5	300	10.0	90.0
3	15.5	300	10.0	90.0
4	16.5	300	80.0	20.0
5	20.0	300	80.0	20.0

Note: Other HPLC gradients may be used as long as the method criteria and project data quality objectives are met.

It may be necessary to adjust the HPLC gradient in order to optimize instrument performance. Columns with different dimensions (e.g. 2.1mm x 30mm) and columns from different manufacturers (Keystone Betasil C18 etc.) may be used.

Mass Transitions Monitored.

Analyte	Mass Transition Q1 (amu)	Mass Transition Q3 (amu)	Declustering Potential
PFBA (C_4 Acid)	213.0	169.0	-30
NFPA (C_5 Acid)	262.9	219.0	-30
PFHA (C_6 Acid)	313.0	268.7, 118.9	-45
TDHA (C_7 Acid)	362.9	318.7, 168.8, 118.9	-35
PFOA (C_8 Acid)	413.0	368.9, 219.0, 169.0	-45
C_9 Acid	463.0	418.7, 168.9, 218.9	-45
C_{10} Acid	512.9	468.8, 218.9, 269.1	-35
C_{11} Acid	563.0	518.7, 268.9, 218.8	-45
C_{12} Acid	613.0	568.7, 168.9, 318.7	-50
FBSA	297.7	78.0	-55
FOSA	497.9	77.9	-90
PFBS	298.9	98.9, 79.9	-65
PFHS	398.9	98.9, 80.0	-95
PFOS	498.9	80.0, 98.8, 130.0	-120
THPFOS	426.9	406.8, 80.9	-70
THPFDS	526.9	506.7, 81.0	-70
PFOA [1,2 ^{13}C]	414.9	369.8	-40
PFOS [$^{18}O_2$]	503.0	103.0, 84.0	-100

Multiple transitions for monitoring the analytes is an option as summing multiple transitions may provide quantitation of isomers that more closely matches NMR data and may have the added benefit of increased analyte signal. The use of one daughter ion is acceptable if method sensitivity is achieved, provided that retention time criteria are met to assure adequate specificity. While the daughter ions may be chosen at the discretion of the analyst, mass transition 99 is suggested for PFOS. Quantitation may be performed using the total ion chromatogram (TIC) for a given analyte. For example, the PFOA TIC would sum all three of the monitored transitions. Use of the suggested primary ion is recommended. Retention times may vary slightly, on a day-to-day basis, depending on the batch of mobile phase and the gradient, column, guard column(s) used etc. Drift in retention times is acceptable within an analytical run, as long as the drift continues through the entire analysis and the standards are interspersed throughout the analytical run.

10.2 Method Acquisition Parameters

The following values are provided as an example of method acquisition parameters for a single period, single experiment method using the Sciex instrumentation. Actual values may vary from instrument to instrument. Also, these values may be changed from time to time in order to optimize for greatest sensitivity. If a multiple period is used, each period may have different gas, temperature, and ion spray voltages.

Curtain Gas (CUR)	15.0
Collision Gas (CAD)	High
IonSpray Voltage (IS)	-4500
Temperature (TEM)	450.0
Gas 1 (Nebulizer) GS1	35.0
Gas 2 (Turbo Gas) GS2	45.0
Interface Heater (ihe)	ON
Entrance Potential (EP)	-10

10.3 Calibration Curve

Analyze the standard curve prior to each set of samples. The validated method specifies that the standard curve should be plotted using a linear fit, weighted 1/x or unweighted. However, the standard curve may also be plotted by quadratic fit ($y = ax^2 + bx + c$), weighted 1/x or unweighted, using suitable software. The calibration curves may include but should not be forced through zero. The mathematical method used to calculate the calibration curve should be applied consistently throughout a study. Any change should be thoroughly documented in the raw data.

If the calibration curve does not meet acceptance criteria, perform routine maintenance or prepare a new standard curve (if necessary) and reanalyze.

For purposes of accuracy when quantitating low levels of analyte, it may be necessary to use the low end of the calibration curve rather than the full range. For example, when attempting to quantitate approximately 0.05 ng/mL of analyte, generate a calibration curve consisting of the standards from 0.025 ng/mL to 10.0 ng/mL rather than the full range of the curve (0.025 ng/mL to 25.00 ng/mL). This will reduce inaccuracy attributed to linear regression weighting of high concentration standards.

High and/or low points may be excluded from the calibration curves to provide a better fit over the range appropriate to the data or because they did not meet the pre-determined acceptance criteria. Low-level curve points should also be excluded if their area counts are not at least twice that of the method and/or solvent blanks. Any curve point may be rejected due to a bad injection

or failing to meet accuracy requirements of ± 25% (and ± 30% for the LLOQ). Justification for exclusion of calibration curve points will be noted in the raw data. A minimum of 6 points will be used to construct the calibration curve. No more than 25% of the total extracted calibration standards should be excluded. This does not pertain to low and high levels that may need to be disabled to achieve an accurate curve in the concentration range of the samples, rather, this is intended to apply to mid curve points only.

10.4 Continuing Calibration Verification (CCV)

Continuing calibration verifications (CCV) are analyzed to verify the accuracy of the calibration curve. Analyze a mid-range calibration standard, one of the same standards used to construct the calibration curve, at a minimum after every tenth sample, not including solvent blanks, with a minimum of one per sample set. Calibration verification injections must be within ±25% to be considered acceptable. The calibration curve and the last passing CCV will then bracket acceptable samples. Multiple CCV levels may be used.

10.5 System Suitability

A minimum of three system suitability samples will be injected at the beginning of each analytical run, prior to the analysis of the calibration curve. Typically these samples are at a concentration near the mid-level of the calibration curve are repeated injections from one autosampler vial. The system suitability injections must have area counts with an RSD of ≤5% and a retention time RSD of ≤2% to be compliant.

11 Procedures

11.1 Extraction Scheme

The following steps represent a typical extraction scheme. Sample extraction volumes and final elution volumes may be adjusted to meet data quality objectives.

Allow samples to equilibrate to room temperature. Thoroughly mix samples by gently inverting the sample bottle.

Measure a 40mL representative aliquot of the well-mixed sample into a 50mL polypropylene centrifuge tubes (Spike the lab matrix spikes as required*, replace lid and mix well). Alternate volumes may be used depending on the scope of the project. Use a consistent sample volume for all extractions and document on the sample prep sheet.

Add 40µL of 250 mg/mL sodium thiosulfate solution to 40mL of sample. Adjust the amount of sodium thiosulfate solution added if alternate sample volumes are used. Thoroughly mix sample.

Note: * Samples may need to be prescreened to determine an appropriate matrix spike level (typically approximately 0.5 to 10 times the sample concentration). Alternatively the samples could be spiked at more than one level, allowing for the inappropriate spike level to be eliminated.

Condition the C18 SPE cartridges (1g, 6mL) by passing approximately 10mL methanol (at a minimum) followed by approximately 50mL (at a minimum) ASTM Type I water. Do not let column run dry. If column does run dry, recondition. SPE cartridges cannot be over conditioned.

Load the analytical sample onto the C18 SPE cartridge. Once the entire sample has loaded onto the cartridge, isolate the cartridge from the vacuum and wait until all samples on the vacuum manifold have been loaded. Discard eluate. Open manifold valves and pull a vacuum on the SPE cartridges for approximately 3 minutes to remove residual water from the SPE cartridge.

Elute with exactly 5 mL of 100% methanol. Collect eluate into graduated 15mL polypropylene centrifuge tubes. This is the target elution fraction (final volume approximately 4.5 mL as not all of the solvent will leave the SPE column. This will not affect the calculations in any way since the

curve is also extracted). Note: the elution volume may be altered to meet project needs. The same elution volume needs to be used for all samples, calibration standards, and QC samples.

Intersperse the method blanks throughout the extraction batch.

Transfer well-mixed aliquots of the final extract to labeled autovials.

Analyze a portion of the target elution fraction eluent using negative electrospray HPLC/MS/MS or HPLC/MS.

Note: Samples are concentrated by a factor of eight during the extraction; Initial Vol = 40mL → Final Vol. = 5mL.

11.2 Sample Analysis

Set up analysis sample queue. Method blanks must be interspersed throughout the analytical sequence.

Inject the same volume (between 5–25µL) of each standard, analytical sample and blank into the instrument (unless an on-instrument sample dilution is desired).

All sample extracts with a concentration > ULOQ must be diluted and reanalyzed. If dilution of the final extract fails to produce acceptable results (e.g. poor MS recoveries) dilute the original sample and re-extract.

12 Data Analysis and Calculations

The chromatography analysis software will typically calculate the amount of target analyte in the sample extracts using the established calibration curve.

Calculate the percent recovery of the LCS using the following equation:

$$\text{LCS\% recovery} = \frac{\text{LCS Concentration } (\frac{ng}{mL})}{\text{Spike Concentration } (\frac{ng}{mL})} * 100\%$$

Calculate the percent recovery of the FSCS using the following equation:

$$\text{FSCS\% recovery} = \frac{\text{FSCS Concentration } (\frac{ng}{mL})}{\text{Spike Concentration } (\frac{ng}{mL})} * 100\%$$

Calculate the percent recovery of the FMS using the following equation:

$$\text{FMS \% recovery} = \frac{\text{FMS Concentration } (\frac{ng}{mL}) - \text{Average Concentration of Sample/Sample Duplicate } (\frac{ng}{mL})}{\text{Spike Concentration } (\frac{ng}{mL})} * 100\%$$

13 Method Performance

Any method performance parameters that are not achieved must be considered in the evaluation of the data. Nonconformance to any specified parameters must be described and discussed if the project lead chooses to report the data.

If criteria listed in this method performance section are not met, maintenance may be performed on the system and samples reanalyzed, or other actions taken as determined by the analyst/project lead. Document all actions in the raw data.

If data are to be reported when performance criteria have not been met, the data must be footnoted on tables and discussed in the text of the report.

13.1 System Suitability

A minimum of three system suitability samples will be injected at the beginning of each analytical run. These samples are run prior to the calibration curve. The system suitability injections must have area counts with an RSD of ≤5% and a retention time RSD of ≤2% to be compliant.

13.2 Quantitation

Calibration Curve: The coefficient of determination (r^2) value for the calibration curve must be greater than or equal to 0.990. Each point in the curve must be within ±25% of the theoretical concentration with the exception of the LLOQ, which may be within ±30%.

Demonstration of Specificity: Specificity is demonstrated by chromatographic retention time (within 3% of standard) and the mass spectral response of unique ions.

13.3 Sensitivity

Solvent Blanks and Method Blanks: Solvent and method blank area counts must be < 50% that of the lowest standard used in the calibration curve. Solvent blanks analyzed after high level samples or calibration standards may have area counts greater than 50% of the lowest standard from instrument carryover as long as subsequent solvent blanks analyzed before the next sample/QC sample demonstrate that the instrument carryover is decreasing back to expected levels.

Limits of Quantitation (LOQ): The lower LOQ (LLOQ) is the lowest non-zero active standard in the calibration curve; the peak area of the LLOQ must be at least 2X that of the method blank(s). By definition, the measured value of the LLOQ must be within 30% of the theoretical value.

13.4 Accuracy

CCV Performance: Calibration verification injections must be within ±25% to be considered acceptable. The calibration curve and the last passing CCV will then bracket acceptable samples. Multiple CCV levels may be used. LCS recoveries should be within ±25% to be considered acceptable and to verify that the method is in control on a given day.

Lab Control Spikes: The average recovery of the pooled LCSs for each target analyte should be within 80-120% and the percent relative standard deviation of the recoveries must be less than or equal to 20%.

13.5 Precision

Reproducibility: Reproducibility of the method is defined by the results of duplicate or triplicate analysis of samples. A RPD or RSD of ≤ 20% will be considered acceptable for replicate lab control spikes. Because field sample duplicates account for variability in sample location (as well as variability in the extraction procedure), RPD or RSD values may be higher. Report the RPD or

RSD of sample/field duplicates and discuss in the report if greater than 20% and the impact on field matrix spike recoveries, if any.

13.6 Analytical Method Uncertainty / Data Acceptability

Analytical method uncertainty may be defined and reported in one of two ways. The first approach uses historical LCS data that is control charted and is used to evaluate method accuracy and precision, based on procedures defined in ETS-12-012. In ETS-12-012, a detailed estimation of uncertainty measurements was performed on ETS-8-231.2, "Solid Phase Extraction and Analysis of Perfluorinated Acids, Alcohol, Amides, Sulfonates and other Fluorinated Compounds by High Performance Liquid Chromatography/Mass Spectrometry". The procedure outlined in this method utilizes the same preparation and analysis steps as those identified in ETS-8-231.2. In that evaluation it was determined that the use of control charts was an acceptable approach to determining analytical method uncertainty. At least twenty data points are required when using this method for determining analytical uncertainty. The method uncertainty is defined as 2x the standard deviation of the percent recoveries of the pooled lab control spikes. While all LCS data points are control charted, only the most recent fifty data points are used for determining the method uncertainty.

When less than twenty LCS data points have been generated for a given analyte, the batch LCS and FMS QC determine the data acceptability. If FMSs meet the ±30% recovery criteria at a level appropriate to the endogenous level, and the LCS meet the ±20% recovery criteria, then the uncertainty of the data is deemed accurate to within 100±20%. If FMS do not meet the ±30% recovery criteria, and historical FMS data does not exist, the analytical uncertainty is evaluated on a case-by-case basis and a discussion included in the final analytical report.

14 Pollution Prevention and Waste Management

Sample extract waste and flammable solvent is discarded in high BTU containers, and glass pipette waste is discarded in broken glass containers located in the laboratory.

15 Records

Each data package generated for a study must include all supporting information for reconstructibility of the data. Information for the data package must include, but is not limited to the following items: study or project number, sample and standard prep sheets/records, instrument run log (instrument batch records, instrument acquisition method, summary pages), instrument results files, chromatograms, calibration curves, and data calculations.

16 Attachments

None.

17 References

"Method of Analysis for the Determination of Perfluorooctane sulfonate (PFOS), Perfluorooctane sulfonylamide (PFOSA), and Perfluorooctanoate (POAA) in Water", E. Wickremesinhe and J. Flaherty, Study Number 023–002, Centre Analytical Laboratories, Inc., State College, Pennsylvania, January 2000.

E01-0454: Validation report for the "Method of Analysis for the Determination of Perfluorooctane sulfonate (PFOS), Perfluorooctane sulfonylamide (PFOSA), and Perfluorooctanoate (POAA) in

Water", E. Wickremesinhe and J. Flaherty, Study Number 023–002, Centre Analytical Laboratories, Inc., State College, Pennsylvania.

18 Affected Documents

None.

19 Revisions

Revision Number	Summary of Changes
1	*Updated to the new format. Changed Title.* *Section 1: States the validation of 3 analytes, removes reference to EPA document that's no longer applicable.* *Section 2: Provided for the extraction of more than the 3 validated analytes, allows the use of a LC/MS system, not only the LS/MS/MS previously mentioned.* *Section 3: Revised definitions for field matrix spike, field control spike, LLOQ, method blank, and MDL.* *Section 5: Reworded the interferences, added recommendation to use disposable pipettes.* *Section 6: Re-categorized and pared down.* *Section 7: Changed storage time to 6 months. Added more calibration points to the table.* *Section 8:Added statement addressing labeling requirements and spiking procedures. Expanded section 8.8.* *Section 9: New Section* *Section 10: Changed some of the parameters in the tables. Allowed for use of different instrumentation. Added information from section 12 of previous version, extensively revised.* *Section 11 (section 9 in previous version): Clarification of wash step, stated exact volume of eluate is 5 mL, revised standardization process, removed requirement to use LC/MS/MS.* *Section 12 (section 13 in previous version: no changes* *Section 13 (section 14 in previous version): Extensively rewritten.* *Section 14 (section 15 in previous version): no changes* *Section 15 (section 16 in previous version): Minor changes to recording requirements.* *Section 16 (section 17 in previous version): Removed attachment.* *Section 17 (section 18 in previous version): Removed reference to EPA document that no longer applied to this SOP.* *Section 18: New section.*
2	*Section 1: Added emphasis that the method is a performance-based method. Added statement that this method does not cover sample collection.* *Section 2: Removed statement that samples are shipped cold as this is no longer the practice. Removed reference to specific C18 SPE cartridge. Added statement that other extraction and elution volumes may be used.* *Section 3. Minor edits/clarifications to several definitions. Added surrogate and SPE cartridge as a definition. Removed references to LOD and MDL as this*

laboratory does not report LCD and/or MDL values.
Section 6. Added reference to automated SPE workstation.
0Section 7. Added additional analytes and updated standard preparation examples.
Section 8. Removed verbiage that pertained to sample collection as the 3M Environmental Lab personnel typically do not collect samples. Addressed sample bottle preparation.
Section 9. Changed method blank criteria, LMS and FMS acceptance criteria from 100±25% to 100±30%, and LCS criteria where pooled recoveries are used to determine acceptance.
Section 10. Updated all example instrument conditions for Sciex instrumentation – removed Micromass references
Section 11. Clarified extraction procedure – removed inclusion of a 40% methanol wash step.
Section 12. Minor edits.
Section 13. Added section on determination of analytical uncertainty and changed performance criteria as appropriate.

3 *Section 11.1. Added use of sodium thiosulfate*

3M Environmental Laboratory

Method

Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis

Method Number: ETS-8-044.0

Adoption Date: Upon Signing

Effective Date: 04/13/07

Approved By:

William K. Reagen, Laboratory Manager — Date: 4/12/07

1 Scope and Application

This method is to be used to quantify Perfluorobutanoic Acid (PFBA), Perfluoropentanoic Acid (PFPeA), Perfluorohexanoic Acid (PFHA), Perfluoroheptanoic Acid (PFHpA), Perfluorooctanoic Acid (PFOA), Perfluorononanoic Acid (PFNA), Perfluorodecanoic Acid (PFDA), Perfluoroundecanoic Acid (PFUnA), Perfluorododecanoic Acid (PFDoA), Perfluorobutanesulfonate (PFBS), Perfluorohexanesulfonate (PFHS), and Perfluorooctanesulfonate (PFOS) by High Performance Liquid Chromatography coupled to a tandem Mass Spectrometric Detector (LC/MS/MS) in clean water samples. Water samples containing heavy particulate may require preparation by an alternate method such as ETS-8-154 "Determination of Perfluorinated Acids, Alcohols, Amides, and Sulfonates In Water By Solid Phase Extraction and High Performance Liquid Chromatography/Mass Spectrometry".

This method is considered a ***performance-based*** method. Data is considered acceptable as long as the defined QC elements are satisfied.

Sample collection is not covered under this analytical procedure.

2 Method Summary

Clean aqueous samples are analyzed by direct injection using LC/MS/MS. Samples containing heavy particulate may not be suitable for analysis by this method. Samples containing suspended particulate should be centrifuge prior to removing a sample aliquot, or filtered.

This is a performance-based method. Method accuracy is determined for each sample set using multiple laboratory control spikes at multiple concentrations. This method also requires that the precision and accuracy for each sample be determined using field matrix spikes to verify that the method is applicable to each sample matrix. Sample results for spikes outside of 70% to 130%, may be flagged as such (with expanded accuracy statements), or will not be reported due to non-compliant quality control samples.

Fortification levels for field matrix spikes and for laboratory matrix spikes should be at least 50% of the endogenous level and less than 10 times the endogenous level to be used to determine the statement of accuracy for analytical results.

3 Definitions

3.1 Calibration Standard

A solution prepared by spiking a known volume of the Working Standard (WS) into a predetermined amount of ASTM Type I, HPLC grade water, or other suitable water, and analyzed according to this method. Calibration standards are used to calibrate the instrument response with respect to analyte concentration.

3.2 Laboratory Duplicate Sample (LDS, or Lab Dup)

A laboratory duplicate sample is a separate aliquot of a sample taken in the analytical laboratory that is analyzed separately with identical procedures. Analysis of LDSs compared to that of the first aliquot give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.3 Field Blank (FB)/Trip Blank

ASTM Type I, HPLC grade water, or other suitable water, placed in a sample container in the laboratory and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation and all analytical procedures. The purpose of the FB is to determine if test substances or other interferences are present in the field environment. This sample is also referred to as a Trip Blank.

3.4 Field Duplicate Sample (FDS, Field Dup)

A sample collected in duplicate at the same time from the same location as the sample. The FDS is handled under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analysis of the FDS compared to that of the first sample gives a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

3.5 Field Matrix Spike (FMS)

A sample to which known quantities of the target analytes are added to the sample bottle in the laboratory before the bottles are sent to the field for collection of aqueous samples. A known, specific volume of sample must be added to the sample container without rinsing. This may be accomplished by making a "fill to this level" line on the outside of the sample container. The FMS should be spiked between approximately 50% and 10 times the expected analyte concentration in the sample. If the expected range of analyte concentrations is unknown, multiple spikes at varying levels may be prepared to increase the likelihood that a spike at an appropriate level is made. The FMS is analyzed to ascertain if any matrix effects, interferences, or stability issues may complicate the interpretation of the sample analysis.

3.6 Trip Blank Spike (Field Spike Control Sample, FSCS)

An aliquot of ASTM Type I, HPLC grade water, or other suitable water, to which known quantities of the target analytes are added in the laboratory prior to the shipment of the collection bottles. The FSCS is extracted and analyzed exactly like a study sample to help determine if the method is in control and whether a loss of analyte could be attributed to holding time, sample storage and/or shipment issues. A low and high FSCS are appropriate when expected sample concentrations are not known or may vary. At least one separate, un-spiked sample must be taken at the same time and place as each FMS.

3.7 Laboratory Control Sample (LCS)

An aliquot of control matrix to which known quantities of the target analytes are added in the laboratory at the time of sample extraction. At least two levels are included, one generally at the low end of the calibration curve and one near the mid to upper range of the curve. The LCSs are extracted and analyzed exactly like a laboratory sample to determine whether the method is in control. LCSs should be prepared each day samples are extracted.

3.8 Laboratory Matrix Spike (LMS)

A laboratory matrix spike is an aliquot of a sample to which known quantities of target analytes are added in the laboratory. The LMS is analyzed exactly like a laboratory sample to determine whether the sample matrix contributes bias to the analytical results. The endogenous concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LMS corrected for these concentrations. LMSs are optional for analysis of aqueous samples.

3.9 Laboratory Sample

A portion or aliquot of a sample received from the field for testing.

3.10 Limit of Quantitation (LOQ)

The lower limit of quantitation (LLOQ) for a dataset is the lowest concentration that can be reliably quantitated within the specified limits of precision and accuracy during routine operating conditions. To simplify data reporting, the LLOQ is generally selected as the lowest non-zero standard in the calibration curve that meets method criteria. Sample LLOQs are matrix-dependent.

The upper limit of quantitation (ULOQ) for a dataset is the highest concentration that can be reliably quantitated within the specified limits of precision and accuracy during routine operating conditions. The highest standard in the calibration curve that meets method criteria is defined as the ULOQ.

3.11 Method Blank

An aliquot of control matrix that is treated exactly like a laboratory sample including exposure to all glassware, equipment, solvents, and reagents that are used with other laboratory samples. The method blank is used to determine if test substances or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.12 Sample

A sample is an aliquot removed from a larger quantity of material intended to represent the original source material.

3.13 Stock Standard Solution (SSS)

A concentrated solution of a single-analyte prepared in the laboratory with an assayed reference compound.

3.14 Surrogate

A compound similar in chemical composition and behavior to the target analyte(s), but is not normally found in the sample(s). A surrogate compound is typically a target analyte with at least one atom containing an isotopically-labeled substitution. If used, surrogate(s) are added to all samples and quality control samples. Surrogate(s) are added to quantitatively evaluate the entire analytical procedure including sample collection, preparation, and analysis. Inclusion of a surrogate analyte is an optional quality control measure and is NOT required.

3.15 Working Standard (WS)

A solution of several analytes prepared in the laboratory from SSSs and diluted as needed to prepare calibration standards and other required analyte solutions.

4 Warnings and Cautions

4.1 Health and Safety

The acute and chronic toxicity of the standards for this method have not been precisely determined; however, each should be treated as a potential health hazard. The analyst should wear gloves, a lab coat, and safety glasses to prevent exposure to chemicals that might be present.

The laboratory is responsible for maintaining a safe work environment and a current awareness of local regulations regarding the handling of the chemicals used in this method. A reference file of material safety data sheets (MSDS) should be available to all personnel involved in these analyses.

4.2 Cautions

The analyst must be familiar with the laboratory equipment and potential hazards including, but not limited to, the use of solvents, pressurized gas and solvent lines, high voltage, and vacuum systems. Refer to the appropriate equipment procedure or operator manual for additional information and cautions.

5 Interferences

During sample preparation and analysis, major potential contaminant sources are reagents and glassware. All materials used in the analyses shall be demonstrated to be free from interferences under conditions of analysis by running method blanks.

Parts and supplies that contain Teflon® should be avoided or minimized due to the possibility of interference and/or contamination. These may include, but are not limited to: wash bottles, Teflon® lined caps, autovial caps, HPLC parts, etc.

The use of disposable micropipettes or pipettes to aliquot standard solutions is recommended to make calibration standards and matrix spikes.

6 Instrumentation, Supplies, and Equipment

6.1 Instrumentation and Equipment

A high performance liquid chromatograph capable of pumping up to two solvents and equipped with a variable volume injector capable of injecting 5-100 µL connected to a tandem Mass Spectrometer (LC/MS/MS).

Analytical balance capable of reading to 0.0001g

A device to collect raw data for peak integration and quantitation

15-mL and 50-mL disposable polypropylene centrifuge tubes.

Gas tight syringes, 25µL, 50µL, 100µL, 250µL, 500µL, 1000µL.

1 mL plastic HPLC autovial.

Disposable pipettes, polypropylene or glass as appropriate

Centrifuge capable of spinning 15-mL and 50-mL polypropylene tubes at 3000 rpm.

6.2 Chromatographic System

Guard Column: Prism RP, 4.6 mm x 50 mm, 5 µm

Analytical Column: Betasil C18, 4.6 mm x 100 mm, 5 µm

Temperature: 10°C

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

Mobile Phase (B): Methanol

Gradient Program:

Time (min)	% A	% B	Flow Rate (mL/min)
0.0	97	3	1.0
0.5	97	3	1.0
11.0	5	95	1.0
13.5	5	95	1.0
13.6	97	3	1.0
17.0	97	3	1.0

Injection Volume: 100 µL.

Quantitation: Peak Area – quadratic curve fit, 1/x weighted.

Run Time: ~ 17 minutes.

The previous information is intended as a guide; alternate conditions and equipment may be used provided that data quality objectives are met.

6.3 MS/MS System

6.3.1 Mode: Electrospray Negative ion, MRM mode, monitoring the following transitions:

Analyte	Transition Monitored
PFBA	213 → 169
PFPeA	263 → 219
PFHA	313 → 269 and 313 → 119
PFHpA	363 → 319, 363 → 169 and 363 → 119
PFOA	413 → 369, 413 → 219 and 413 → 169
PFNA	463 → 419, 463 → 169 and 463 → 219
PFDA	513 → 469, 513 → 219 and 513 → 269
PFUnA	563 → 519, 563 → 269 and 563 → 219
PFDoA	613 → 569, 613 → 169 and 613 → 319
PFBS	299 → 80 and 299 → 99
PFHS	399 → 80 and 399 → 99
PFOS	499 → 80, 499 → 99 and 499 → 130

Multiple transitions for monitoring the analytes is an option, as summing multiple transitions may provide quantitation of isomers that more closely matches NMR data and may have the added benefit of increased sensitivity. The use of one daughter ion is acceptable if method sensitivity is achieved, provided that retention time criteria are met to assure adequate specificity.

The previous information is intended as a guide, alternate instruments and equipment may be used.

7 Reagents and Standards

7.1 Chemicals

Water – Milli-Q, HPLC grade, or other suitably appropriate sources

Methanol – HPLC grade

Ammonium Acetate – A.C.S. Reagent Grade

7.2 Standards

Perfluorobutanoic Acid (PFBA – C4 acid); Oakwood Products, Inc

Perfluoropentanoic Acid (PFPeA – C5 acid, also known as NFPA, nonafluoropentanoic acid); Alfa Aesar

Perfluorohexanoic Acid (PFHA – C6 acid); Oakwood Products, Inc

Perfluoroheptanoic Acid (PFHpA – C7 acid, also known as TDHA, tridecafluoroheptanoic acid); Oakwood Products, Inc

Perfluorooctanoic Acid (PFOA – C8 acid); 3M

Perfluorononanoic Acid (PFNA – C9 acid); Oakwood Products, Inc

Perfluorodecanoic Acid (PFDA – C10 acid); Oakwood Products, Inc

Perfluoroundecanoic Acid (PFUnA – C11 acid); Oakwood Products, Inc

Perfluorododecanoic Acid (PFDoA – C12 acid); Oakwood Products, Inc

Perfluorobutanesulfonate (PFBS – C4 sulfonate); 3M

Perfluorohexanesulfonate (PFHS – C6 sulfonate); 3M

Perfluorooctanesulfonate (PFOS – C8 sulfonate); 3M

The previous information is intended as a guide. Reagents and standards from alternate sources may be used.

7.3 Reagent Preparation

2 mM Ammonium acetate solution (Analysis)—Weigh 0.3 g of Ammonium acetate and dissolve in 2.0 L of reagent water.

Note: Alternative volumes may be prepared as long as the ratios of the solvent to solute ratios are maintained.

7.4 Stock Standard Solution (SSS) and Working Standard Solution Preparation

The following standard preparation procedure serves as an example. Weighed amounts and final volumes may be changed to suit the needs of a particular study. For example, µL volumes may be spiked into volumetric flasks when diluting stock solutions to appropriate levels.

100 µg/mL target analyte SSSs—Weigh out 10 mg of analytical standard ***(corrected for percent salt and purity)*** and dilute to 100mL with methanol or other suitable solvent, in a 100mL volumetric flask. Transfer to a 125mL LDPE bottle or other suitable container. Prepare a separate solution for each analyte. Expiration dates and storage conditions of stock solutions should be assigned in accordance with laboratory standard operating procedure. An example of purity and salt correction is given below for PFOS.

$$\text{salt correction factor} = \frac{\text{molecular weight of anion}}{\text{moclecular weight of salt}}$$

$$\text{PFOS } (K^+)\text{salt correction factor} = \frac{499}{538} = 0.9275$$

10 mg $C_8F_{17}SO_3^-K^+$ with purity 90% = 8.35mg $C_8F_{17}SO_3^-$ (10 mg*0.90*0.9275=8.35 mg)

5 µg/mL (5000 ng/mL) mixed working standard—Add 0.5mL each of the 100µg/mL SSSs to a 10mL volumetric flask and bring up to volume with solvent.

250 ng/mL mixed working standard—Add 1.25mL of the 5 µg/mL -mixed working standard solution to a 25mL volumetric flask and bring up to volume with solvent.

125 ng/mL mixed standard—Add 625µL of the 5 µg/mL-mixed working standard solution to a 25mL volumetric flask and bring up to volume with solvent.

Storage Conditions—Store all SSSs and working standards in accordance with laboratory standard operating procedure or in a refrigerator at 4°±2°C for a maximum period of 6 months from the date of preparation.

7.5 Calibration Standards

Using the working standards described above, prepare calibration solutions in ASTM Type I water, HPLC water, or other suitable water, using the following table as a guideline. Note: Volumes of water and working standards may be adjusted to meet the data quality objectives addressed in the general project outline. Calibration levels other than those listed below can be prepared as needed.

Concentration of WS, ng/mL	*Volume of WS, µL*	*Final Volume of Calibration Standard (mL of ASTM Type I Water, or other suitable water)*	*Final Concentration of Calibration Standard, ng/mL (ppb) in ASTM Type I Water, or other suitable water*
125	10	50	0.025
125	15	50	0.0375
125	20	50	0.050
125	30	50	0.075
250	20	50	0.100
250	50	50	0.250
250	100	50	0.500
250	200	50	1.00
5000	25	50	2.50
5000	50	50	5.00
5000	100	50	10.0

8 Sample Handling

8.1 Water Sample Preparation

This method is applicable to clean water samples. Samples containing heavy particulate may not be suitable for analysis by this method. Samples containing suspended particulate should be centrifuge prior to removing a sample aliquot, or filtered.

Thoroughly mix sample before removing an aliquot and placing in a labeled plastic autovial. Plastic is preferred over the use of glass autovials, to prevent the possibly of fluorochemical sticking to the glass.

Dilute sample, if necessary, with ASTM Type I, HPLC water, or other suitable water.

Prepare method QC samples and multiple method blanks and aliquot into labeled plastic autovials.

Prepare at least five method blanks.

9 Sample Analysis - LC/MS/MS

Analyze the standard curve prior to each set of samples. The standard curve may be plotted using a linear fit, weighted 1/x or unweighted, or by quadratic fil ($y = ax^2 + bx + c$), weighted 1/x or unweighted, using suitable software. The calibration curves may include but should not be forced through zero. The mathematical method used to calculate the calibration curve should be applied consistently throughout a study. Any change should be thoroughly documented in the raw data.

High and/or low points may be excluded from the calibration curves to provide a better fit over the range appropriate to the data or because they did not meet the pre-determined acceptance criteria. Low-level curve points should also be excluded if their area counts are not at least twice that of the method and/or solvent blanks. The coefficient of determination (r^2) value for the calibration curve must be greater than or equal to 0.990. Each point in the curve must be within ±25% of the theoretical concentration with the exception of the LLOQ, which may

be within ±30%. Justification for exclusion of calibration curve points will be noted in the raw data. A minimum of 6 points will be used to construct the calibration curve.

If the calibration curve does not meet acceptance criteria, perform routine maintenance or prepare a new standard curve (if necessary) and reanalyze.

Continuing calibration verifications (CCV) are analyzed to verify the accuracy of the calibration curve. Analyze a mid-range calibration standard, one of the same standards used to construct the calibration curve, at a minimum after every tenth sample, not including solvent blanks, with a minimum of one per sample set. Calibration verification injections must be within ±25% to be considered acceptable. The calibration curve and the last passing CCV will then bracket acceptable samples. Multiple CCV levels may be used.

Samples containing analytes that are quantitated above the concentration of the highest standard in the curve should be further diluted and reanalyzed.

10 Quality Control

10.1 Data Quality Objectives

This method and required quality control samples is designed to generate data accurate to ±30% with a targeted LOQ of 0.025 ng/mL. Any deviations from the quality control measures spelled out below will be documented in the raw data and footnoted in the final report.

10.2 Method Blanks

Method blanks must be prepared with each analysis batch. At least five method blanks must be prepared. Method blanks may be injected multiple times, but no more than 3 injections should be removed from a single method blank. At a minimum, method blanks are analyzed prior to instrument calibration, prior to the analysis of CCV samples, and at the end of the analytical run.

The mean area count for each analyte in the method blanks must be less than 50% of the area count of the LOQ standard. The standard deviation of the area counts of these method blanks should be calculated and reported. If the mean area counts of the method blanks exceed 50% of the LOQ standard, then the LOQ must be raised to the first standard level in the curve that meets criteria, or alternatively, the method blanks must be evaluated statistically to determine outliers, or technical justification to eliminate one or more results should be made.

10.3 Sample Replicates

Samples duplicates are collected in the field. The relative percent difference, RPD, should be reported. RPD results greater than 20% will be flagged in the report, but will not be excluded from reporting. The requirement for replicates excludes field blanks.

10.4 Surrogate Spikes

Surrogate spikes are not required but may be used on project specific requirements.

10.5 Lab Control Sample

Triplicate lab control spikes at a minimum of two different concentrations are to be prepared with each preparation batch. Low lab control spikes should be prepared at concentrations in the range of five to ten times higher than the targeted LOQ and high lab control spikes should be prepared at concentrations near the mid-point of the curve. The relative standard deviation of the control spikes evaluated independently at each concentration level must be less than or equal to 20% and the average recovery must be 80-120%. If the above criteria are not met, the entire set of samples should be re-injected or re-prepared as appropriate.

10.6 Field Matrix Spikes / Lab Matrix Spikes

Recoveries of field matrix spikes and laboratory matrix spikes are anticipated to be between 70% and 130% of the fortified levels. Sample results for spikes outside of 70% to 130%, may be flagged as such (with expanded accuracy statements), or will not be reported due to non-compliant quality control samples.

The targeted fortification levels should be at least 50% of the endogenous level and less than 10 times the endogenous level to be used without justification to determine the statement of accuracy for analytical results.

The average of the sample and the field duplicate should be used to calculate the recovery.

11 Data Analysis and Calculations

Use the following equation to calculate the amount of analyte found (in ng/mL, based on peak area) using the standard curve (linear regression parameters) generated by an appropriate software program:

$$\text{Analyte found (ng/mL)} = \frac{\text{(Peak Area - Intercept)}}{\text{Slope}} \times \text{DF}$$

DF = factor by which the final volume was diluted, if necessary.

For samples fortified with known amounts of analyte prior to extraction, use the following equation to calculate the percent recovery.

$$\text{Recovery} = \frac{\text{Total analyte found (ng/mL) - Average analyte found in sample (ng/mL)}}{\text{Analyte added (ng/mL)}} \times 100$$

12 Method Performance

Any method performance parameters that are not achieved must be considered in the evaluation of the data. Nonconformance to any specified parameters must be described and discussed if the Technical Manager (non-GLP study) or Study Director (GLP study) chooses to report the data.

If criteria listed in this method performance section are not met, maintenance may be performed on the system and samples reanalyzed, or other actions taken as appropriate. Document all actions in the raw data.

If data are to be reported when performance criteria have not been met, the data must be footnoted on tables and discussed in the text of the report.

12.1 System Suitability

System Suitability standards are not a required component of this method. If required by protocol or by the technical manager, a minimum of three system suitability samples are injected at the beginning of each analytical run prior to the calibration curve. Typically these samples are at a concentration near the mid level of the calibration curve and are repeated injections from one autosampler vial. The system suitability injections must have area counts with an RSD of ≤5% and a retention time RSD of ≤2% to be compliant.

12.2 Quantitation

Calibration Curve: The coefficient of determination (r^2) value for the calibration curve must be greater than or equal to 0.990. Each point in the curve must be within ±25% of the theoretical concentration with the exception of the LLOQ, which may be within ±30%.

CCV Performance: The calibration standards that are interspersed throughout the analytical sequence are evaluated as continuing calibration verifications in addition to being part of the calibration curve. The accuracy of each curve point must be within 25% of the theoretical value (within 30% for lowest curve point). Samples that are bracketed by CCVs not meeting these criteria must be reanalyzed.

Demonstration of Specificity: Specificity is demonstrated by chromatographic retention time (within 4% of standard) and the mass spectral response of unique ions.

12.3 Sensitivity

The targeted limit of quantitation for all analytes is 0.025 ng/mL. The LOQ for any specific analyte may vary depending on the evaluation of appropriate blanks and the accuracy of the low-level calibration curve points. Refer to Section 10 for additional details.

12.4 Accuracy

This method and required quality control samples are designed to generate data that are accurate to +/-30%. Section 10 contains additional information regarding the required accuracy of laboratory control spikes, field matrix spikes and laboratory matrix spikes.

12.5 Precision

Samples should be collected in duplicate in the field. The relative percent difference, RPD, should be reported. RPD results greater than 20% will be flagged in the report, but will not be excluded from reporting. The requirement for replicates excludes field blanks or rinse blanks.

Section 10 contains additional information regarding the required precision of laboratory control spikes.

13 Pollution Prevention and Waste Management

Waste generated when performing this method will be disposed of appropriately. The original samples will be archived at the 3M Environmental Laboratory in accordance with internal procedures.

14 Records

Each data package generated for a study must include all supporting information for reconstruction of the data. Information for the data package must include, but is not limited to the following items: study or project number, sample and standard prep sheets/records, instrument run log (instrument batch records, instrument acquisition method, summary pages), instrument results files, chromatograms, calibration curves, and data calculations.

15 Affected Documents

None.

16 Revisions

Revision Number	Summary of Changes

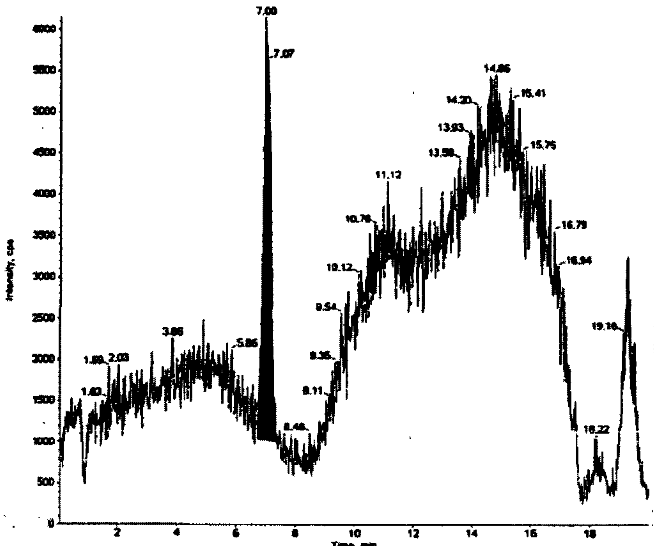
ATTACHMENT D: SAMPLE CHROMATOGRAMS AND CALIBRATION CURVES

Sample Name: "g091211a015" Sample ID: "08008-83-7" File: "g091211a.wiff"
Peak Name: "PFBA" Mass(es): "213.0/169.0 amu"
Comment: "1.0 ng/mL FC std in 90:10 MeOH:H2O" Annotation: ""

Sample Index: 15
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: 0.965 ng/mL
Acq. Date: 12/11/2009
Acq. Time: 1:57:57 PM

Modified: No
Proc. Algorithm: IntelliQuan - IQA
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.96 sec
Smoothing Width: 0 points
RT Window: 20.0 sec
Expected RT: 7.00 min
Use Relative RT: No

Int. Type: Valley
Retention Time: 7.00 min
Area: 33422 counts
Height: 5.13e+003 cps
Start Time: 6.71 min
End Time: 7.26 min

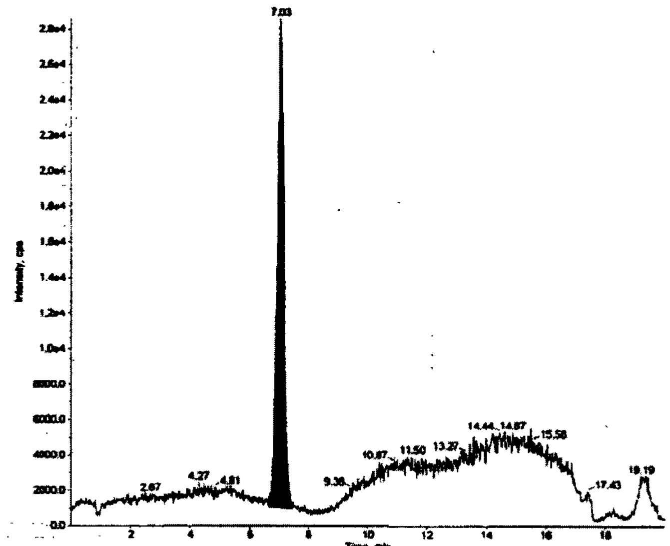


Sample Name: "g091211a029" Sample ID: "LCS-091202-3" File: "g091211a.wiff"
Peak Name: "PFBA" Mass(es): "213.0/169.0 amu"
Comment: "Spike LCS" Annotation: ""

Sample Index: 29
Sample Type: QC
Concentration: 5.00 ng/mL
Calculated Conc: 5.13 ng/mL
Acq. Date: 12/11/2009
Acq. Time: 9:01:01 PM

Modified: Yes
RT Window: 20.0 sec
Expected RT: 7.00 min
Use Relative RT: No

Int. Type: Manual
Retention Time: 7.03 min
Area: 429439 counts
Height: 2.76e+004 cps
Start Time: 6.53 min
End Time: 7.39 min

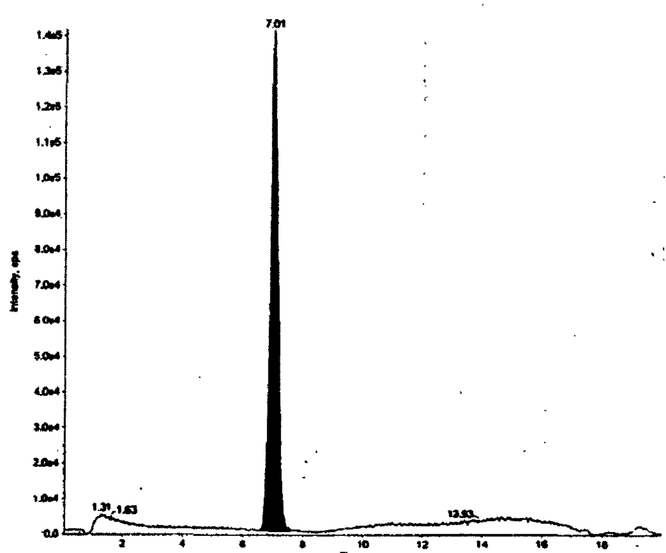


Sample Name: "g091211a077" Sample ID: "E07-0149-143" File: "g091211a.wiff"
Peak Name: "PFBA" Mass(es): "213.0/169.0 amu"
Comment: "COL-GW-MW3-04-0 (dil 1:10)" Annotation: ""

Sample Index: 77
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 292. ng/mL
Acq. Date: 12/12/2009
Acq. Time: 1:46:07 PM

Modified: Yes
RT Window: 20.0 sec
Expected RT: 7.00 min
Use Relative RT: No

Int. Type: Manual
Retention Time: 7.01 min
Area: 2342925 counts
Height: 1.41e+005 cps
Start Time: 6.60 min
End Time: 7.62 min

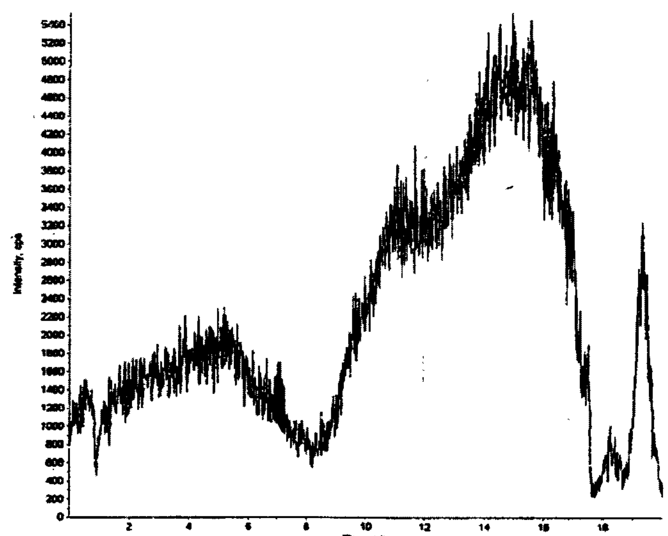


Sample Name: "g091211a102" Sample ID: "Solvent blank" File: "g091211a.wiff"
Peak Name: "PFBA" Mass(es): "213.0/169.0 amu"
Comment: "08004-37 - 90:10 MeOH:water" Annotation: ""

Sample Index: 102
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.0737 ng/mL
Acq. Date: 12/12/2009
Acq. Time: 10:30:28 PM

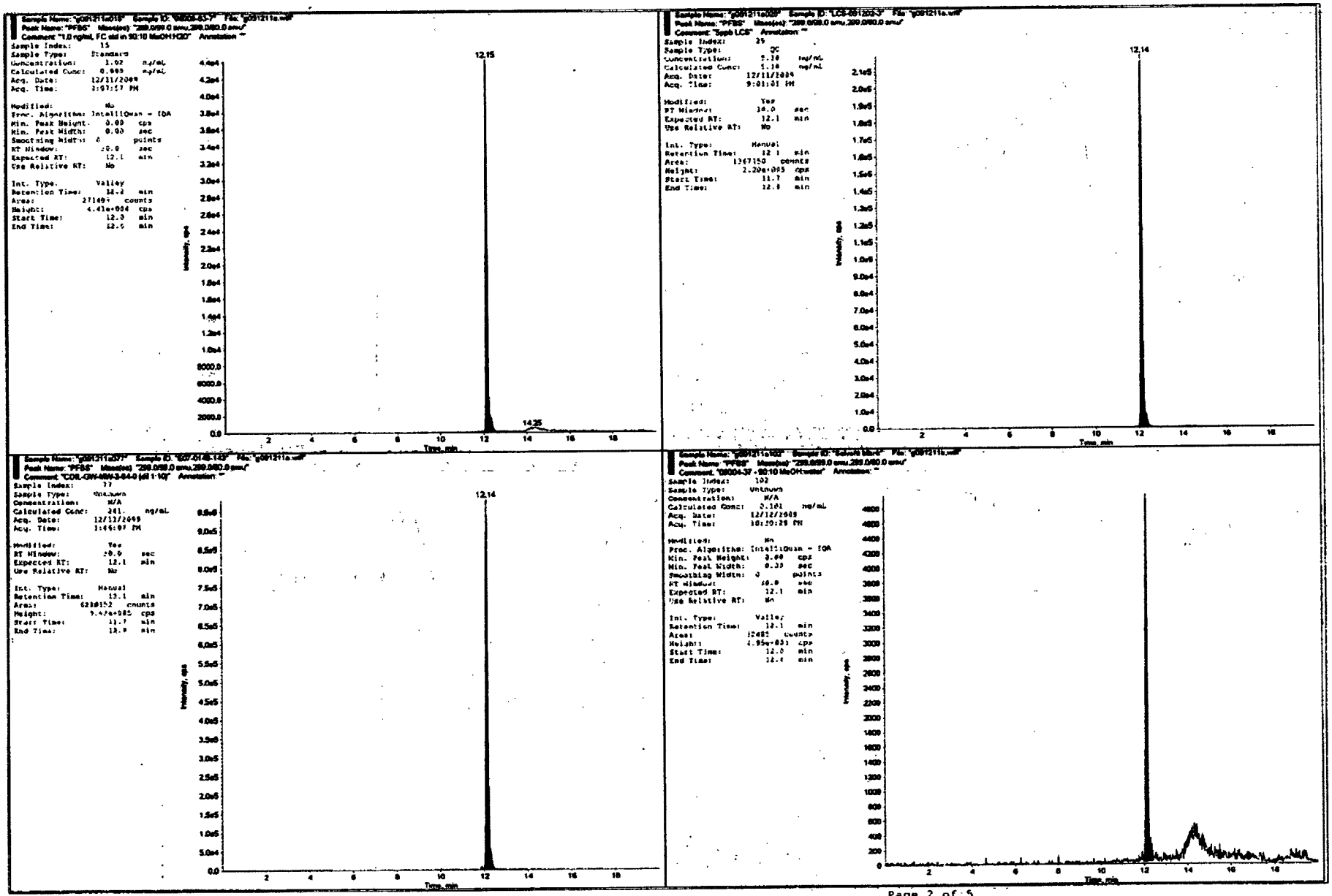
Modified: Yes
RT Window: 30.0 sec
Expected RT: 7.00 min
Use Relative RT: No

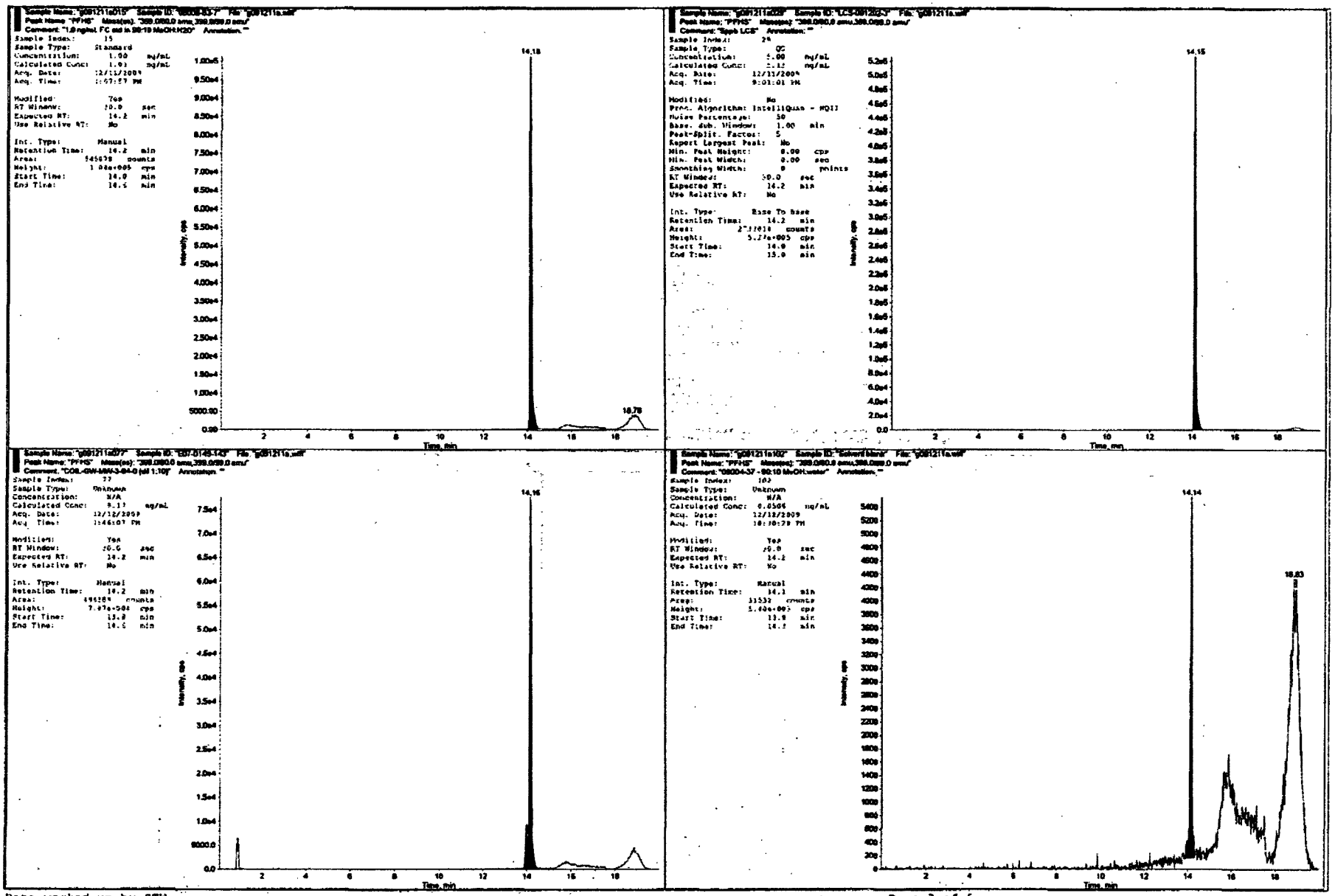
Int. Type: Manual
Retention Time: 7.01 min
Area: 10734 counts
Height: 7.03e+002 cps
Start Time: 6.94 min
End Time: 7.27 min



Data worked up by STW
Printing Time: 11:32:14 AM
Printing Date: Thursday, January 28, 2010

Page 1 of 5


Sample Index: 15
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: 0.989 ng/mL
Acq. Date: 12/11/2009
Acq. Time: 3:07:57 PM
Modified: No
Proc. Algorithm: IntelliQuan - EOA
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 0 points
RT Window: 20.0 sec
Expected RT: 12.1 min
Use Relative RT: No
Int. Type: Valley
Retention Time: 12.2 min
Area: 27149* counts
Height: 4.41e+004 cps
Start Time: 12.0 min
End Time: 12.5 min
12.15
14.25
Intensity, cps
Time, min
Sample Index: 25
Sample Type: QC
Concentration: 5.00 ng/mL
Calculated Conc: 5.10 ng/mL
Acq. Date: 12/11/2009
Acq. Time: 9:01:01 PM
Modified: Yes
RT Window: 10.0 sec
Expected RT: 12.1 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 12.1 min
Area: 1367150 counts
Height: 2.20e+005 cps
Start Time: 11.7 min
End Time: 12.8 min
12.14
Intensity, cps
Time, min
Sample Index: 17
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 241. ng/mL
Acq. Date: 12/12/2009
Acq. Time: 1:46:07 PM
Modified: Yes
RT Window: 20.0 sec
Expected RT: 12.1 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 12.1 min
Area: 6280152 counts
Height: 9.42e+005 cps
Start Time: 11.7 min
End Time: 12.9 min
12.14
Intensity, cps
Time, min
Sample Index: 102
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.101 ng/mL
Acq. Date: 12/12/2009
Acq. Time: 10:20:29 PM
Modified: No
Proc. Algorithm: IntelliQuan - EOA
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.20 sec
Smoothing Width: 0 points
RT Window: 20.0 sec
Expected RT: 12.1 min
Use Relative RT: No
Int. Type: Valley
Retention Time: 12.1 min
Area: 2485 counts
Height: 1.95e+003 cps
Start Time: 12.0 min
End Time: 12.4 min
Intensity, cps
Time, min

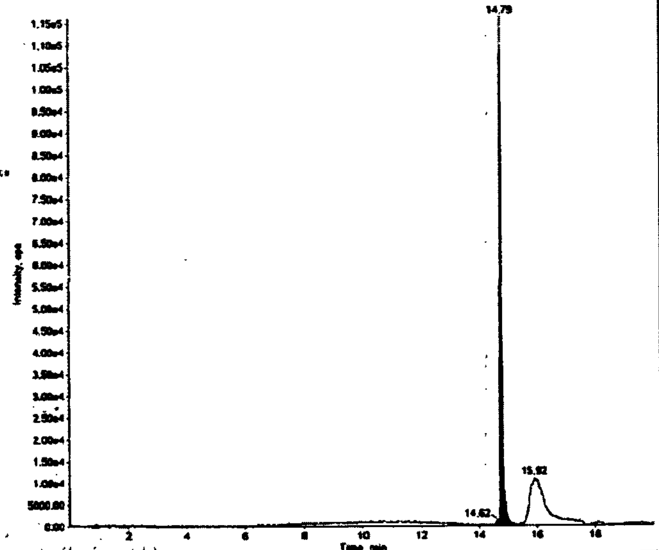

Sample Name: "g091211a015" Sample ID: "0806-83-7" File: "g091211a.wiff"
Peak Name: "PFHS" Mass(es): "399.0980,0 amu,399.0980,0 amu"
Comment: "1.0 ng/mL FC std in 90:10 MeOH:H2O" Annotation: ""
Sample Index: 15
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: 1.03 ng/mL
Acq. Date: 12/11/2009
Acq. Time: 11:07:27 PM
Modified: Yes
RT Window: ±0.8 sec
Expected RT: 14.2 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 14.2 min
Area: 545638 counts
Height: 1.04e+005 cps
Start Time: 14.0 min
End Time: 14.5 min
14.18
16.78
Intensity, cps
Time, min
Sample Name: "g091211a029" Sample ID: "LCS-091202-7" File: "g091211a.wiff"
Peak Name: "PFHS" Mass(es): "399.0980,0 amu,399.0980,0 amu"
Comment: "5ppb LCS" Annotation: ""
Sample Index: 29
Sample Type: QC
Concentration: 5.00 ng/mL
Calculated Conc: 5.12 ng/mL
Acq. Date: 12/11/2009
Acq. Time: 9:01:01 PM
Modified: No
Peak Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 5
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 0 points
RT Window: ±0.8 sec
Expected RT: 14.2 min
Use Relative RT: No
Int. Type: Base To Base
Retention Time: 14.2 min
Area: 2"31018 counts
Height: 5.27e+005 cps
Start Time: 14.0 min
End Time: 15.0 min
14.15
Intensity, cps
Time, min
Sample Name: "g091211a027" Sample ID: "057-0149-143" File: "g091211a.wiff"
Peak Name: "PFHS" Mass(es): "399.0980,0 amu,399.0980,0 amu"
Comment: "COC-GW-MW3-84-0 (dil 1:10)" Annotation: ""
Sample Index: 27
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 9.17 ng/mL
Acq. Date: 12/12/2009
Acq. Time: 1:46:07 PM
Modified: Yes
RT Window: ±0.8 sec
Expected RT: 14.2 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 14.2 min
Area: 496189 counts
Height: 7.87e+004 cps
Start Time: 13.8 min
End Time: 14.5 min
14.16
Intensity, cps
Time, min
Sample Name: "g091211a102" Sample ID: "Solvent Blank" File: "g091211a.wiff"
Peak Name: "PFHS" Mass(es): "399.0980,0 amu,399.0980,0 amu"
Comment: "0806-57 - 90:10 MeOH:water" Annotation: ""
Sample Index: 102
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.0506 ng/mL
Acq. Date: 12/13/2009
Acq. Time: 10:30:29 PM
Modified: Yes
RT Window: ±0.8 sec
Expected RT: 14.2 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 14.1 min
Area: 31532 counts
Height: 5.40e+003 cps
Start Time: 13.9 min
End Time: 14.7 min
14.14
16.83
Intensity, cps
Time, min

Sample Name: "g091211a015" Sample ID: "08008-63-7" File: "g091211a.wiff"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu,413.0/219.0 amu,413.0/169.0 amu"
Comment: "1.0 ng/mL FC std in 90:10 MeOH:H2O" Annotation: ""

Sample Index: 15
Sample Type: Standard
Concentration: 1.01 ng/mL
Calculated Conc: 1.04 ng/mL
Acq. Date: 12/11/2009
Acq. Time: 4:07:37 PM

Modified: No
Proc. Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 2
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 0 points
RT Window: 30.5 sec
Expected RT: 14.8 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 14.8 min
Area: 243141 counts
Height: 1.16e+005 cps
Start Time: 14.5 min
End Time: 15.2 min

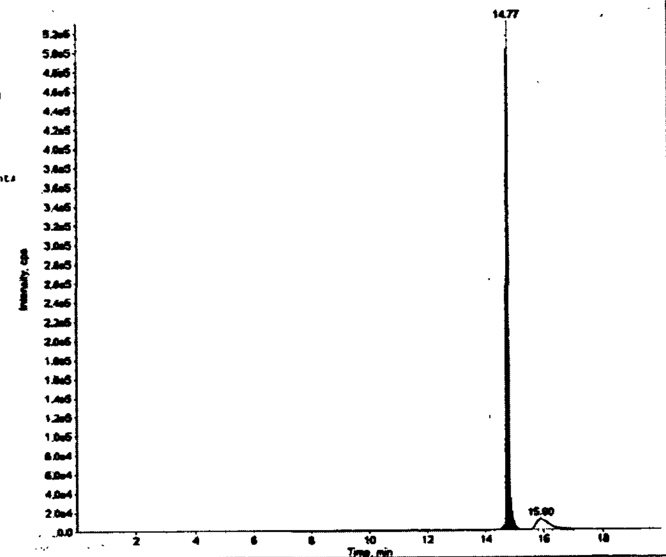


Sample Name: "g091211a029" Sample ID: "LCS-091209-3" File: "g091211a.wiff"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu,413.0/219.0 amu,413.0/169.0 amu"
Comment: "5ppb LCS" Annotation: ""

Sample Index: 29
Sample Type: QC
Concentration: 5.05 ng/mL
Calculated Conc: 5.14 ng/mL
Acq. Date: 12/11/2009
Acq. Time: 9:01:01 PM

Modified: No
Proc. Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 1
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 0 points
RT Window: 30.0 sec
Expected RT: 14.8 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 14.8 min
Area: 2604395 counts
Height: 5.31e+005 cps
Start Time: 14.5 min
End Time: 15.2 min

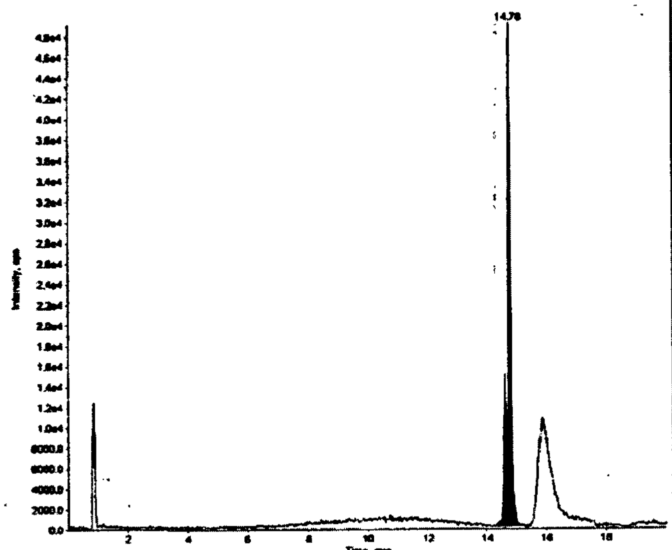


Sample Name: "g091211a077" Sample ID: "E07-0149-143" File: "g091211a.wiff"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu,413.0/219.0 amu,413.0/169.0 amu"
Comment: "COL-GW-4893-8-0 (dil 1:10)" Annotation: ""

Sample Index: 77
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 5.40 ng/mL
Acq. Date: 12/12/2009
Acq. Time: 1:45:47 PM

Modified: Yes
RT Window: 30.0 sec
Expected RT: 14.8 min
Use Relative RT: No

Int. Type: Manual
Retention Time: 14.8 min
Area: 358753 counts
Height: 4.96e+004 cps
Start Time: 14.3 min
End Time: 15.1 min

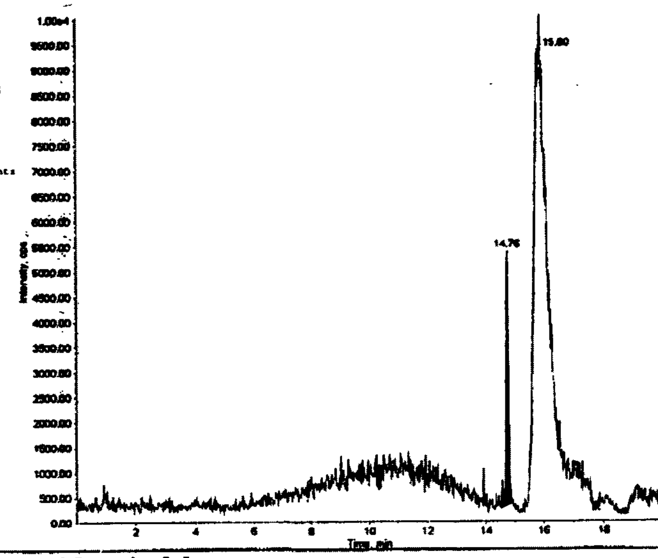


Sample Name: "g091211a102" Sample ID: "Solvent blank" File: "g091211a.wiff"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu,413.0/219.0 amu,413.0/169.0 amu"
Comment: "08004-37 - 90:10 MeOH:water" Annotation: ""

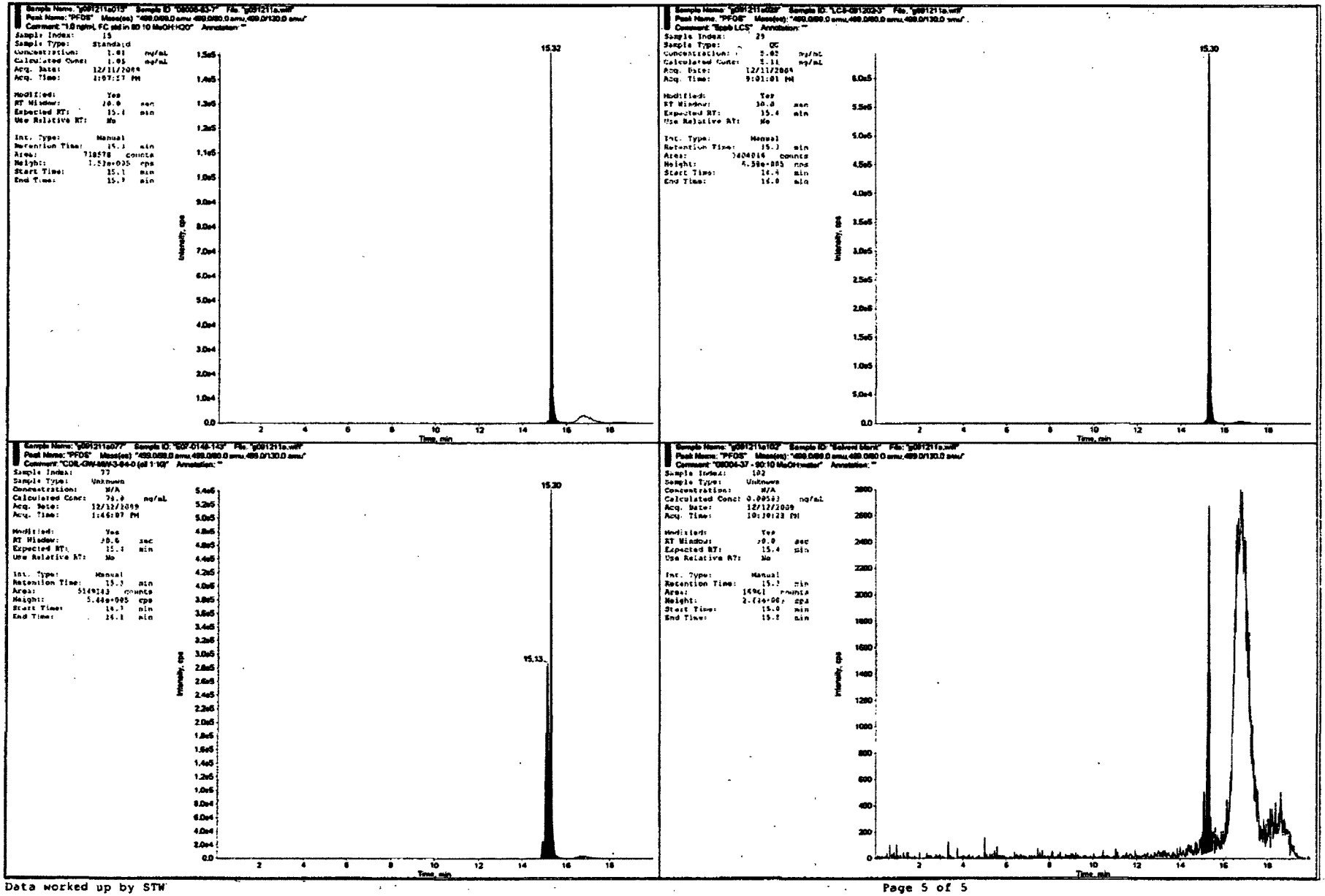
Sample Index: 102
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.0355 ng/mL
Acq. Date: 12/12/2009
Acq. Time: 10:10:28 PM

Modified: No
Proc. Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 3
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 0 points
RT Window: 30.0 sec
Expected RT: 14.8 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 14.8 min
Area: 24519 counts
Height: 5.08e+003 cps
Start Time: 14.5 min
End Time: 14.9 min

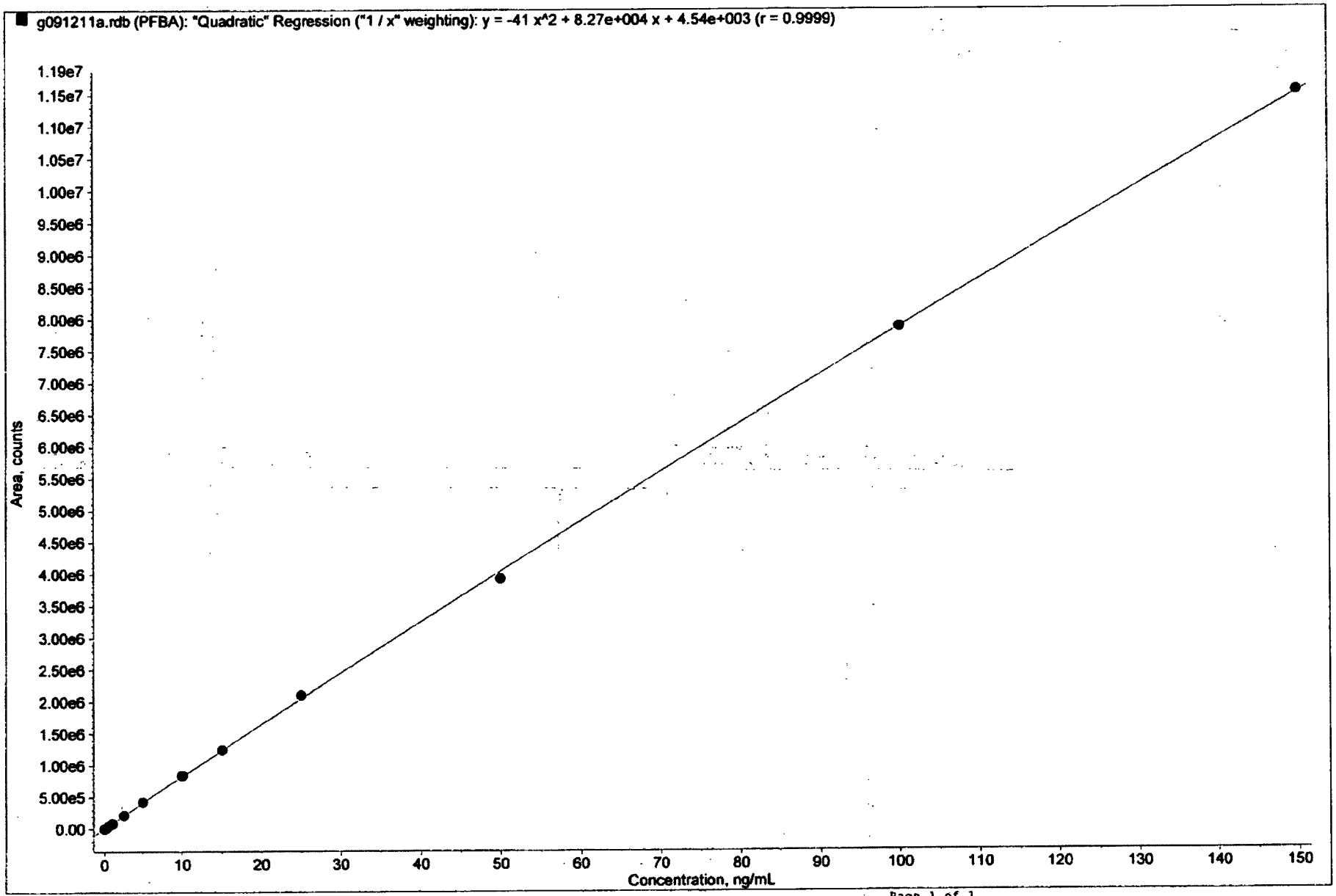


Data worked up by STW
Printing Time: 11:32:14 AM
Printing Date: Thursday, January 28, 2010



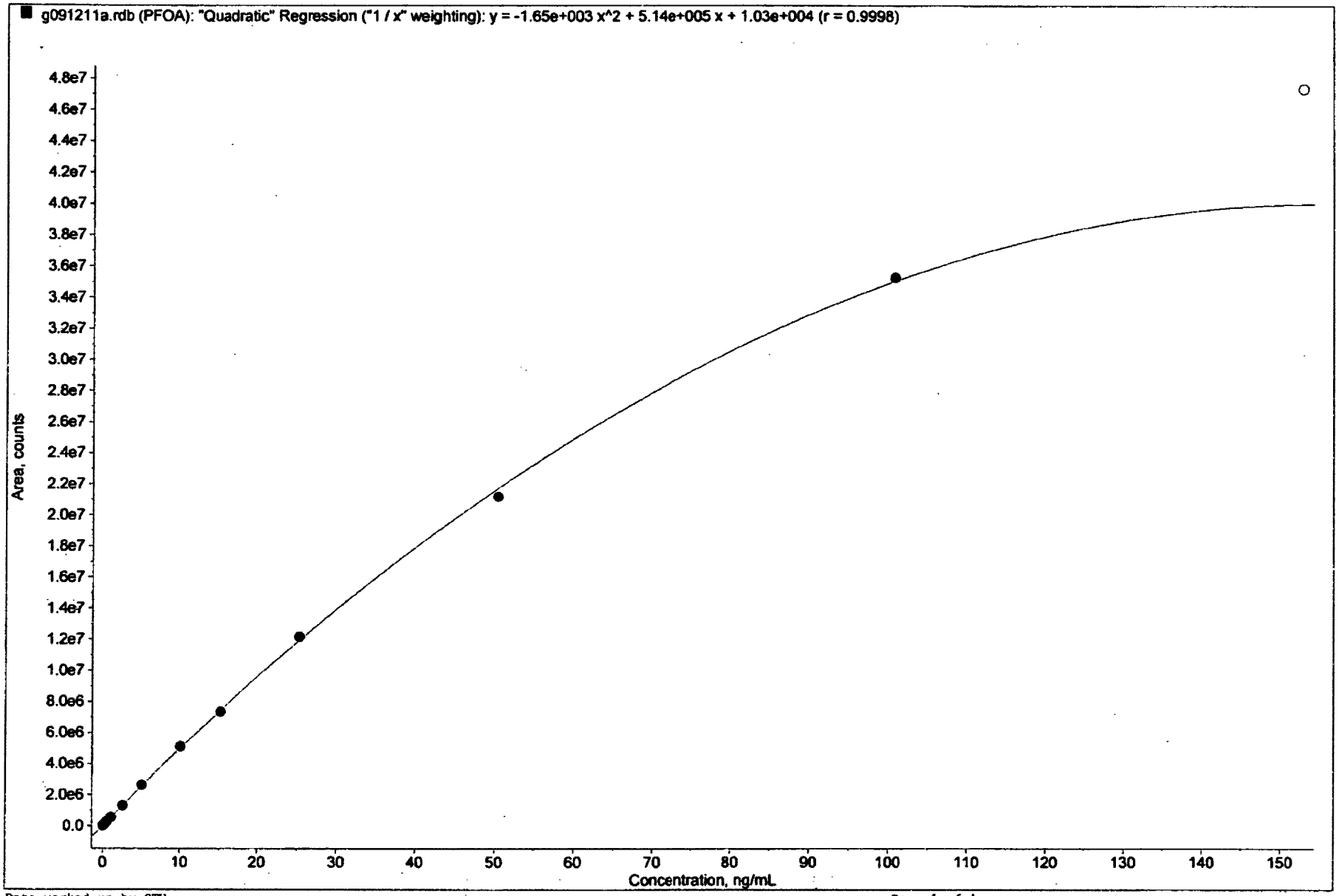
Data worked up by STW
Printing Time: 11:32:14 AM
Printing Date: Thursday, January 28, 2010

Page 5 of 5



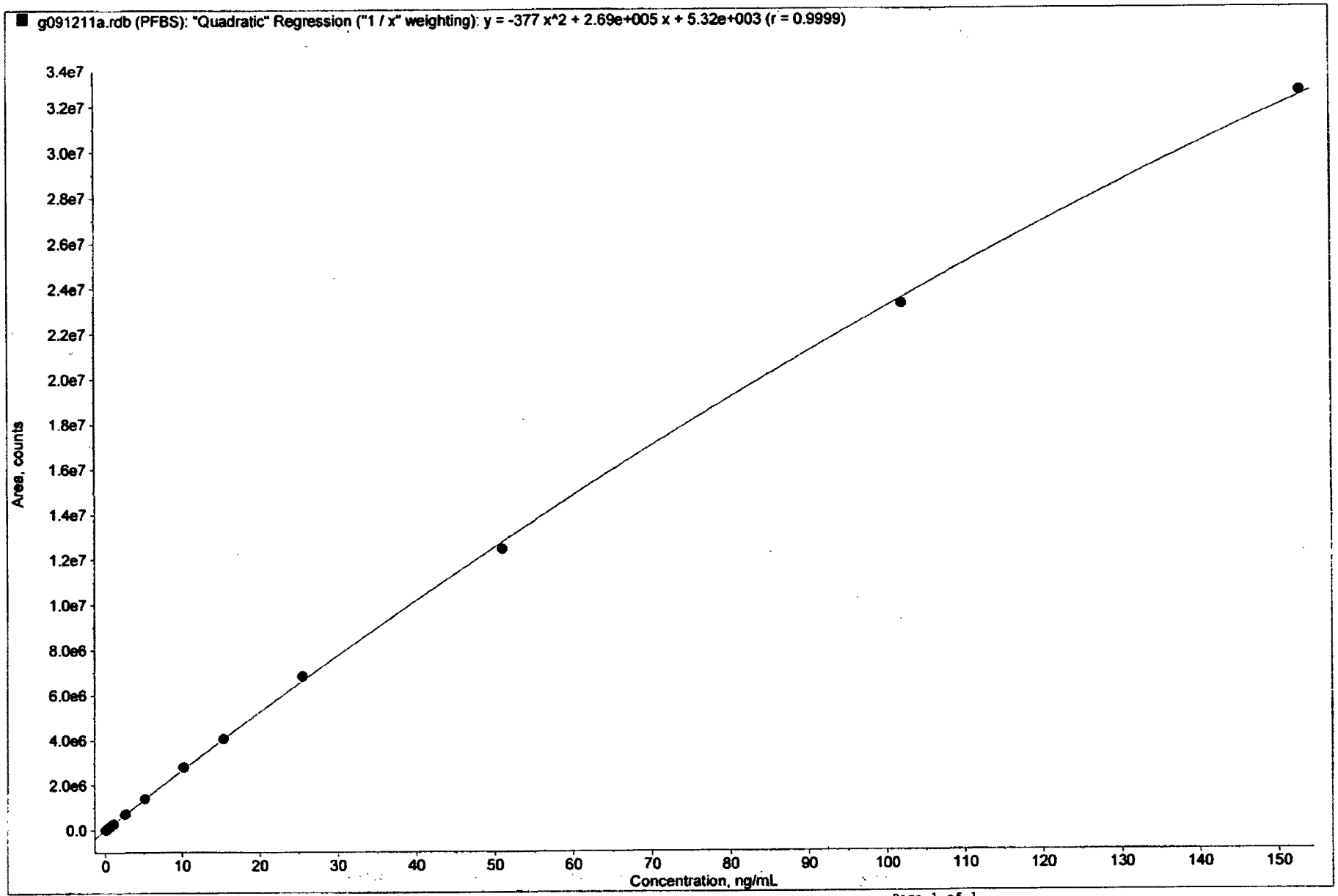
Data worked up by STW
Printing Time: 11:32:38 AM
Printing Date: Thursday, January 28, 2010

Page 1 of 1



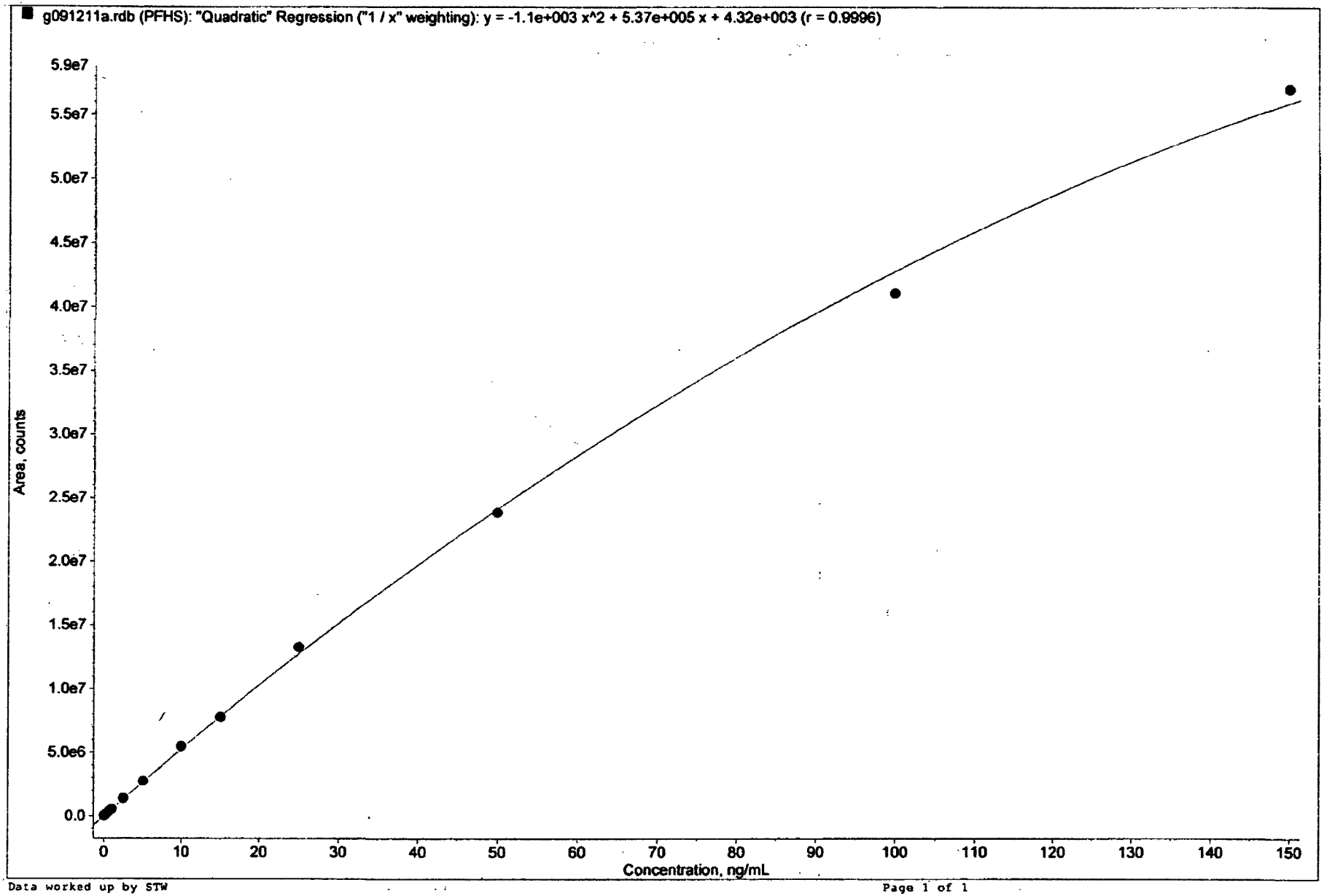
Data worked up by STW
Printing Time: 11:32:54 AM
Printing Date: Thursday, January 28, 2010

Page 1 of 1



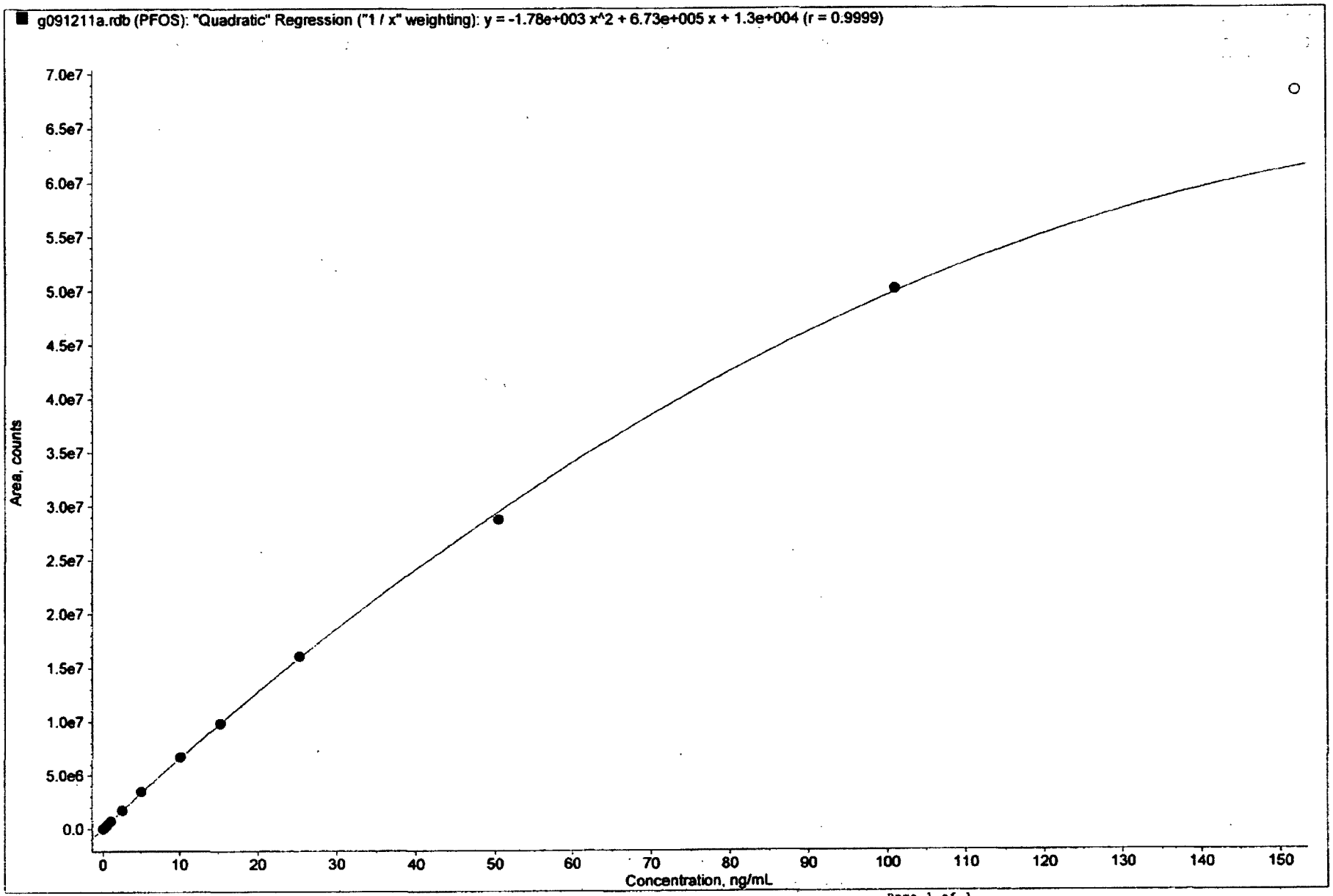
Page 1 of 1

Data worked up by STW
Printing Time: 11:33:10 AM
Printing Date: Thursday, January 28, 2010



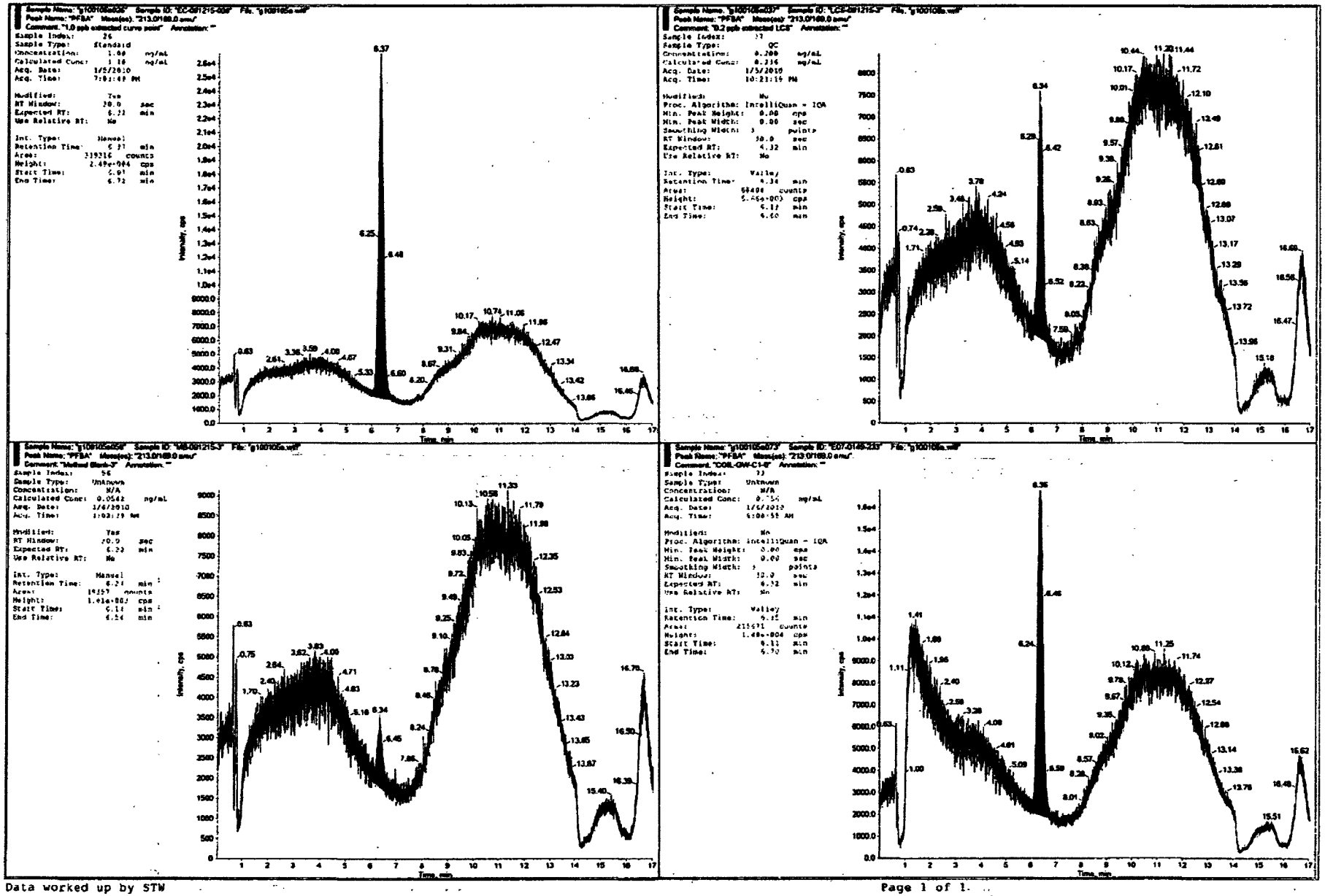
Data worked up by STW
Printing Time: 11:33:28 AM
Printing Date: Thursday, January 28, 2010

Page 1 of 1



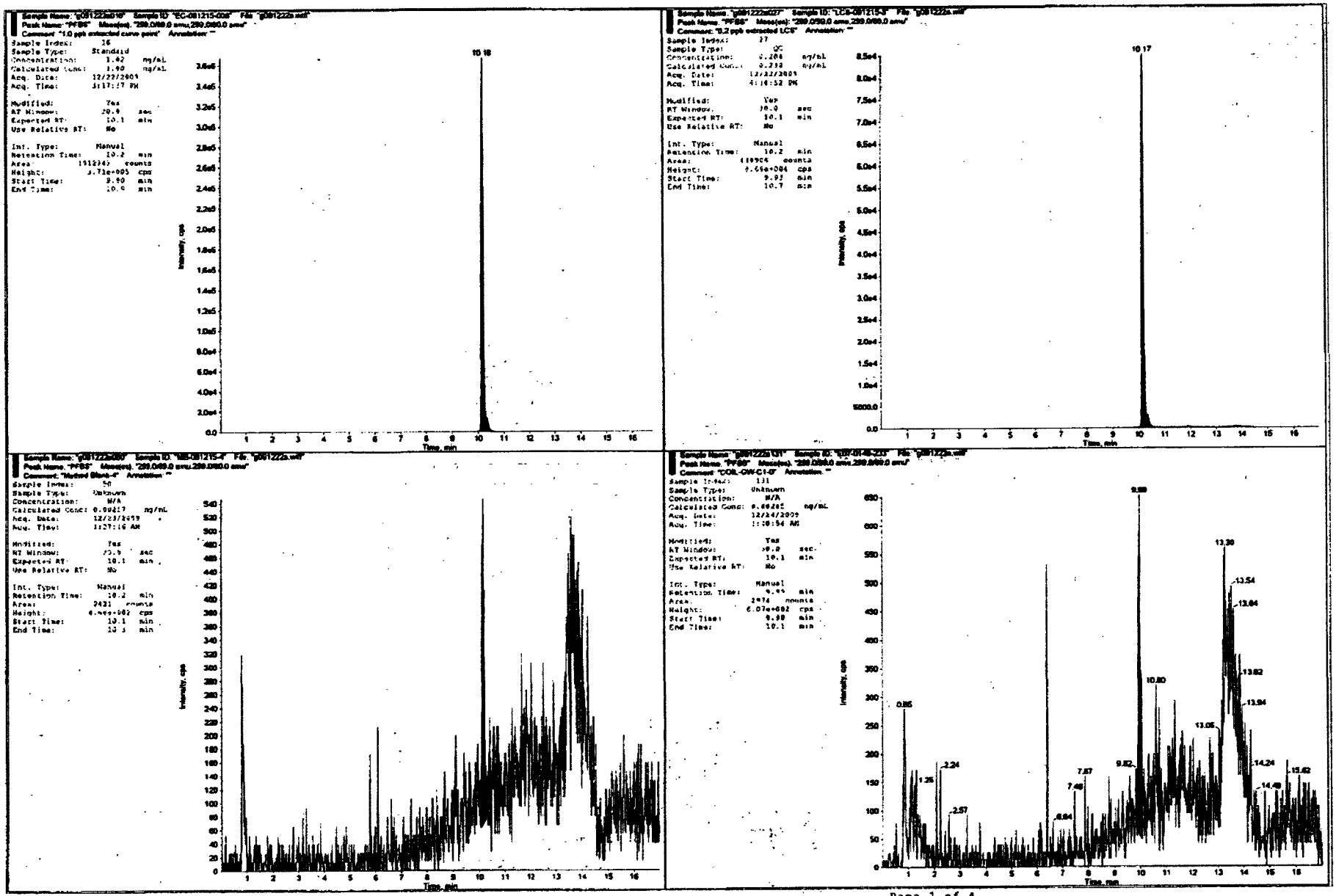
Page 1 of 1

Data worked up by STW
Printing Time: 11:33:44 AM
Printing Date: Thursday, January 28, 2010

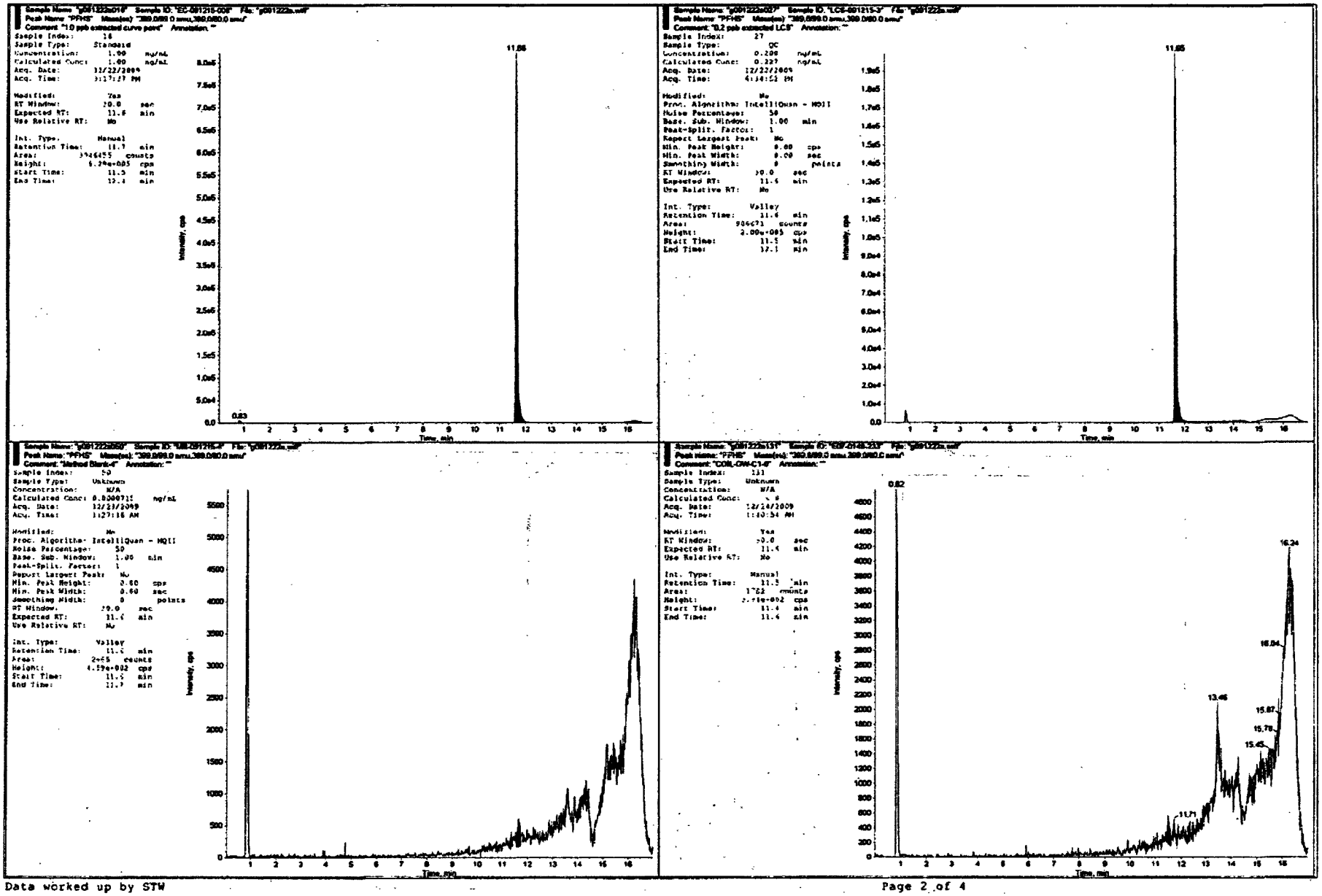


Data worked up by STW
Printing Time: 11:38:58 AM
Printing Date: Thursday, January 28, 2010

Page 1 of 1.



Data worked up by STW
Printing Time: 11:36:48 AM
Printing Date: Thursday, January 28, 2010

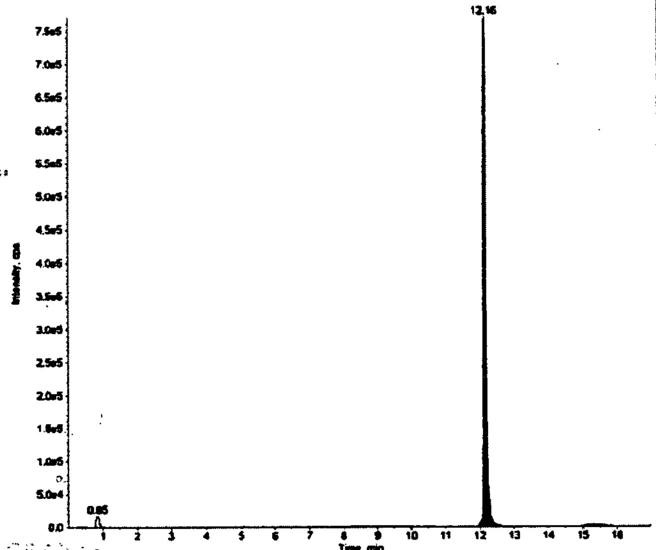

Sample Name: "g091222a016" Sample ID: "EC-091215-008" File: "g091222a.wiff"
Peak Name: "PFHS" Mass(es): "399.9899.0 amu,399.9980.0 amu"
Comment: "1.0 ppb extracted curve point" Annotation: ""
Sample Index: 16
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: 1.00 ng/mL
Acq. Date: 12/22/2009
Acq. Time: 9:17:27 PM
Modified: Yes
RT Window: ±0.8 sec
Expected RT: 11.6 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 11.7 min
Area: 3946455 counts
Height: 6.39e+005 cps
Start Time: 11.5 min
End Time: 12.4 min
11.66
0.83
Intensity, cps
Time, min
Sample Name: "g091222a027" Sample ID: "LCS-091215-3" File: "g091222a.wiff"
Peak Name: "PFHS" Mass(es): "399.9899.0 amu,399.9980.0 amu"
Comment: "0.2 ppb extracted LCS" Annotation: ""
Sample Index: 27
Sample Type: QC
Concentration: 0.200 ng/mL
Calculated Conc: 0.227 ng/mL
Acq. Date: 12/22/2009
Acq. Time: 4:14:52 PM
Modified: No
Proc. Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 1
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 9 points
RT Window: ±0.8 sec
Expected RT: 11.6 min
Use Relative RT: No
Int. Type: Valley
Retention Time: 11.6 min
Area: 906671 counts
Height: 2.00e+005 cps
Start Time: 11.5 min
End Time: 12.1 min
11.65
Intensity, cps
Time, min
Sample Name: "g091222a050" Sample ID: "LB2-091215-4" File: "g091222a.wiff"
Peak Name: "PFHS" Mass(es): "399.9899.0 amu,399.9980.0 amu"
Comment: "Method Blank-4" Annotation: ""
Sample Index: 50
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.0300715 ng/mL
Acq. Date: 12/23/2009
Acq. Time: 1:27:16 AM
Modified: No
Proc. Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 1
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 9 points
RT Window: ±0.8 sec
Expected RT: 11.6 min
Use Relative RT: No
Int. Type: Valley
Retention Time: 11.6 min
Area: 2e+05 counts
Height: 4.57e+002 cps
Start Time: 11.5 min
End Time: 11.9 min
Intensity, cps
Time, min
Sample Name: "g091222a131" Sample ID: "EB-091218-2" File: "g091222a.wiff"
Peak Name: "PFHS" Mass(es): "399.9899.0 amu,399.9980.0 amu"
Comment: "CCBL_CAM-C1-6" Annotation: ""
Sample Index: 131
Sample Type: Unknown
Concentration: N/A
Calculated Conc: N/A
Acq. Date: 12/24/2009
Acq. Time: 1:10:54 AM
Modified: Yes
RT Window: ±0.8 sec
Expected RT: 11.6 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 11.5 min
Area: 1753 counts
Height: 2.41e+002 cps
Start Time: 11.4 min
End Time: 11.6 min
0.82
16.24
16.04
13.46
15.87
15.78
15.45
11.71
Intensity, cps
Time, min

Sample Name: "g091222a016" Sample ID: "EC-091215-008" File: "g091222a.wiff"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu,413.0/219.0 amu,413.0/169.0 amu"
Comment: "1.0 ppb extracted curve point" Annotation: ""

Sample Index: 16
Sample Type: Standard
Concentration: 1.01 ng/mL
Calculated Conc: 0.869 ng/mL
Acq. Date: 12/22/2009
Acq. Time: 3:17:37 PM

Modified: No
Proc. Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 4
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 0 points
RT Window: 26.0 sec
Expected RT: 12.1 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 12.2 min
Area: 3729944 counts
Height: 7.70e+005 cps
Start Time: 11.9 min
End Time: 12.8 min

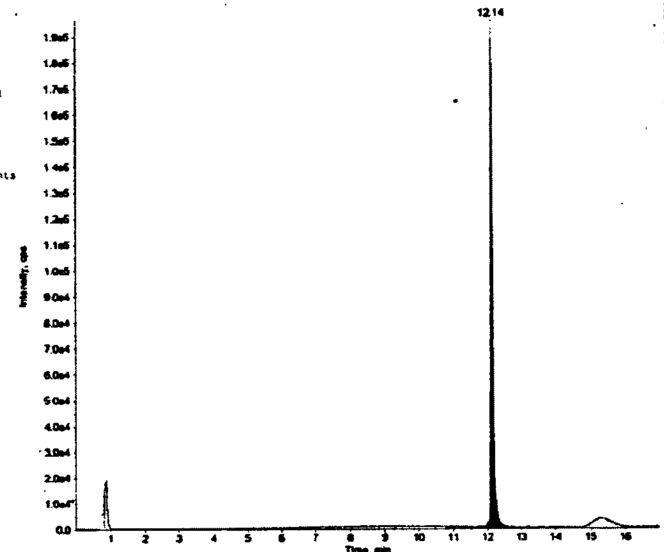


Sample Name: "g091222a027" Sample ID: "LCS-091215-5" File: "g091222a.wiff"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu,413.0/219.0 amu,413.0/169.0 amu"
Comment: "0.2 ppb extracted LCS" Annotation: ""

Sample Index: 27
Sample Type: QC
Concentration: 0.202 ng/mL
Calculated Conc: 0.214 ng/mL
Acq. Date: 12/22/2009
Acq. Time: 4:34:52 PM

Modified: No
Proc. Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 4
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 0 points
RT Window: 30.0 sec
Expected RT: 12.1 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 12.1 min
Area: 495347 counts
Height: 1.96e+005 cps
Start Time: 11.9 min
End Time: 12.7 min

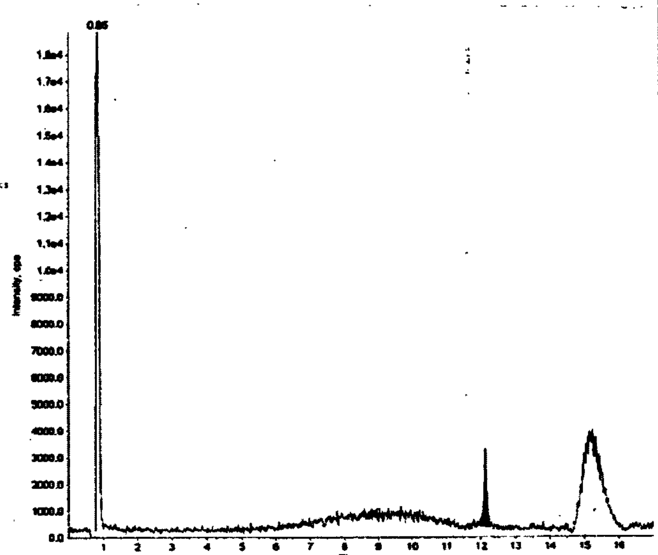


Sample Name: "g091222a050" Sample ID: "MB-091216-4" File: "g091222a.wiff"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu,413.0/219.0 amu,413.0/169.0 amu"
Comment: "Method Blank-4" Annotation: ""

Sample Index: 50
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.0130 ng/mL
Acq. Date: 12/23/2009
Acq. Time: 1:27:16 AM

Modified: No
Proc. Algorithm: IntelliQuan - MQII
Noise Percentage: 50
Base. Sub. Window: 1.00 min
Peak-Split. Factor: 4
Report Largest Peak: No
Min. Peak Height: 0.00 cps
Min. Peak Width: 0.00 sec
Smoothing Width: 0 points
RT Window: 28.9 sec
Expected RT: 12.1 min
Use Relative RT: No

Int. Type: Base To Base
Retention Time: 12.1 min
Area: 15541 counts
Height: 2.91e+003 cps
Start Time: 11.9 min
End Time: 12.3 min

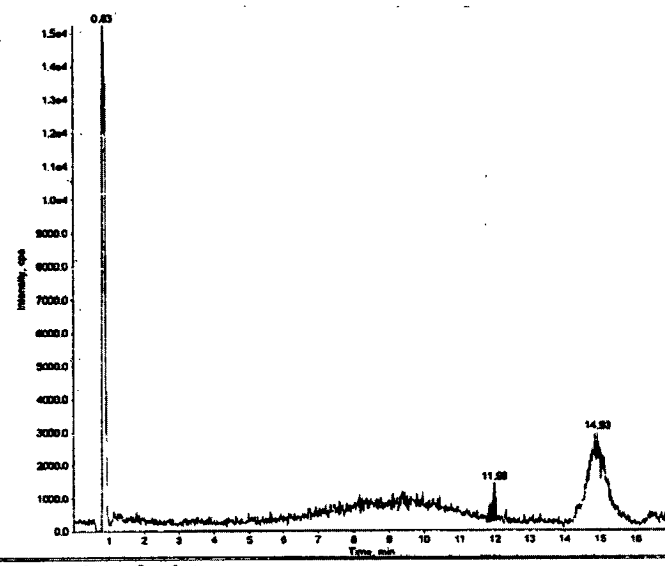


Sample Name: "g091222a131" Sample ID: "E07-0149-220" File: "g091222a.wiff"
Peak Name: "PFOA" Mass(es): "413.0/369.0 amu,413.0/219.0 amu,413.0/169.0 amu"
Comment: "COIL-GW-C1-0" Annotation: ""

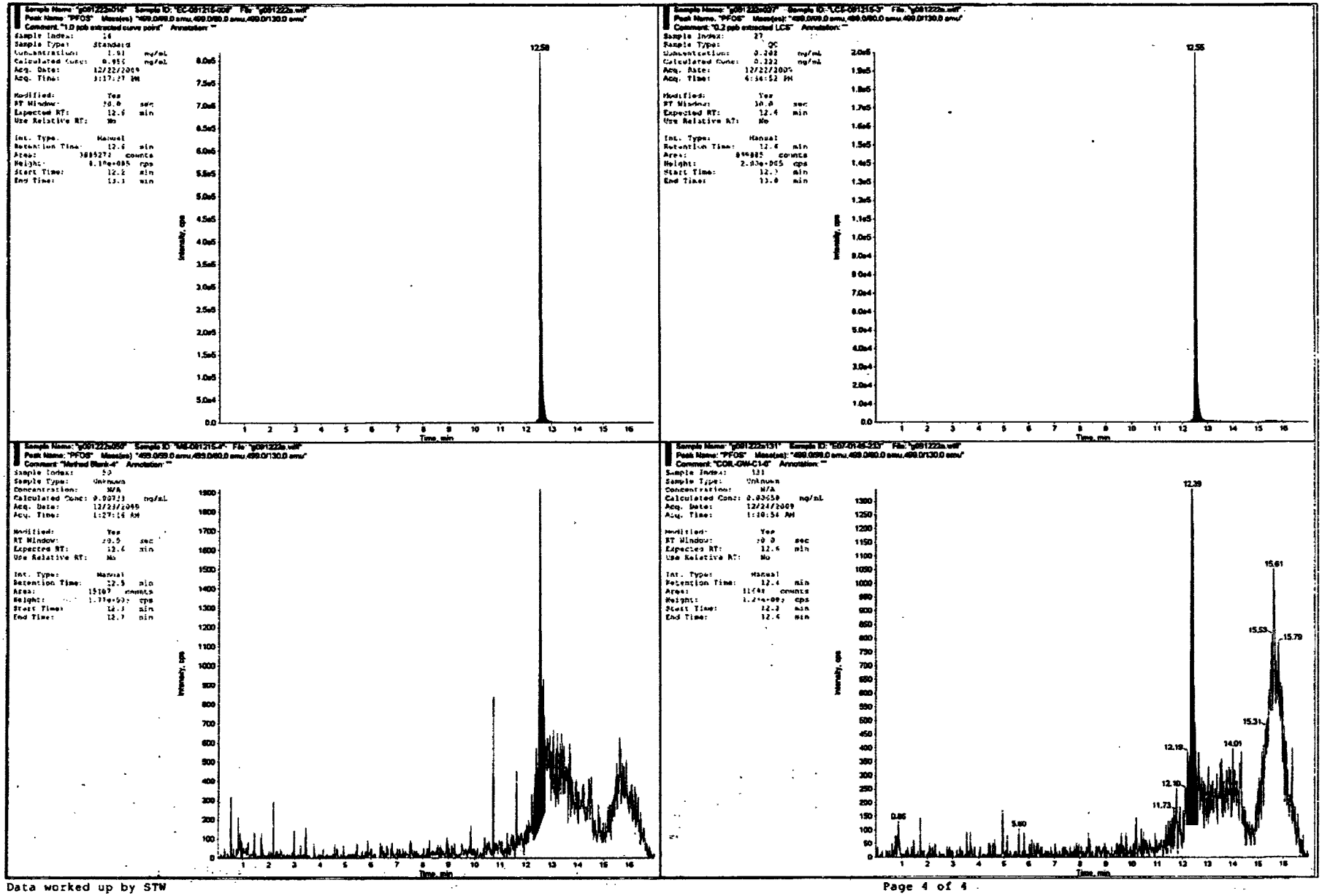
Sample Index: 131
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.0163 ng/mL
Acq. Date: 12/24/2009
Acq. Time: 1:40:34 AM

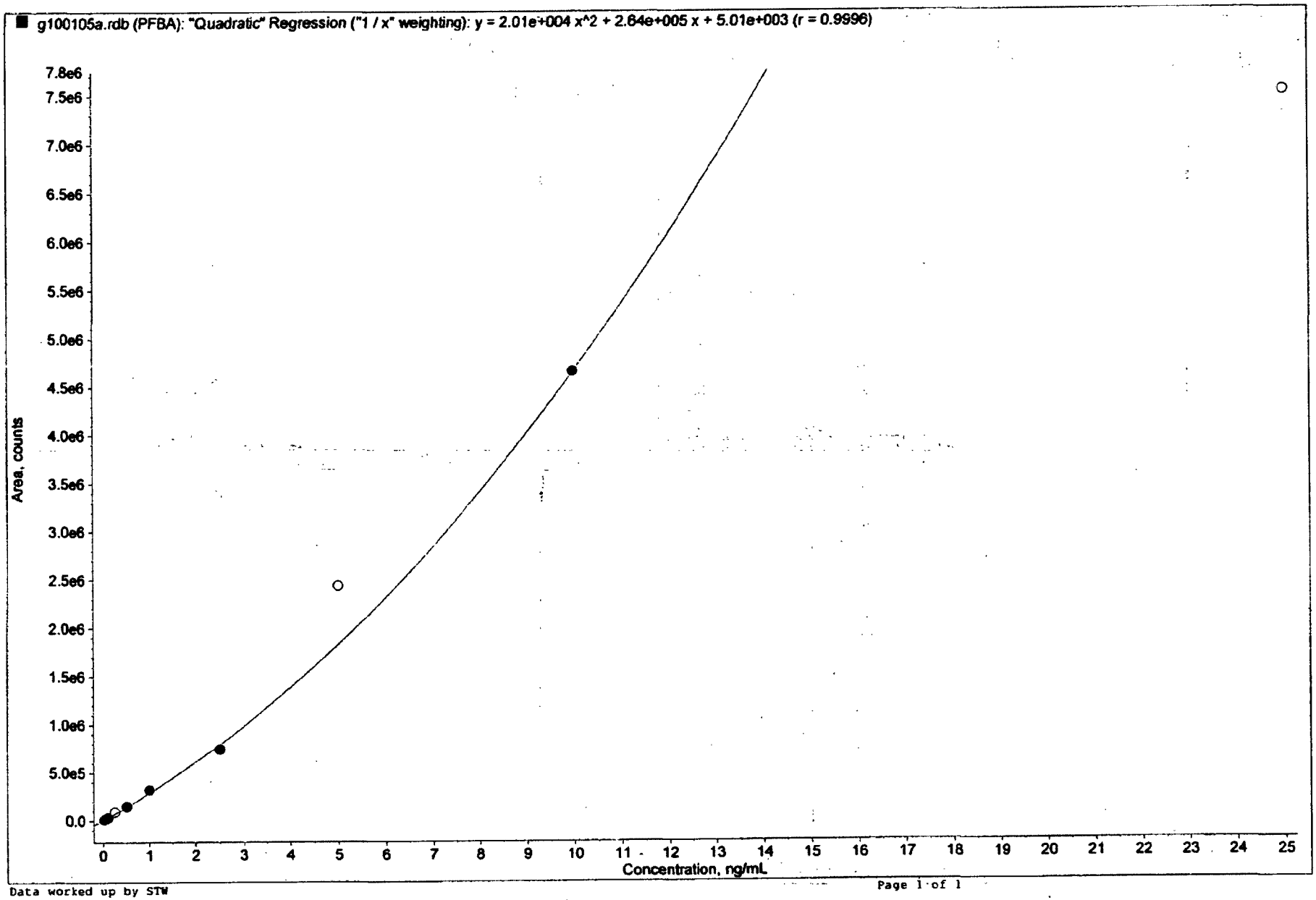
Modified: Yes
RT Window: 30.0 sec
Expected RT: 12.1 min
Use Relative RT: No

Int. Type: Manual
Retention Time: 12.0 min
Area: 6095 counts
Height: 1.15e+003 cps
Start Time: 11.8 min
End Time: 12.1 min



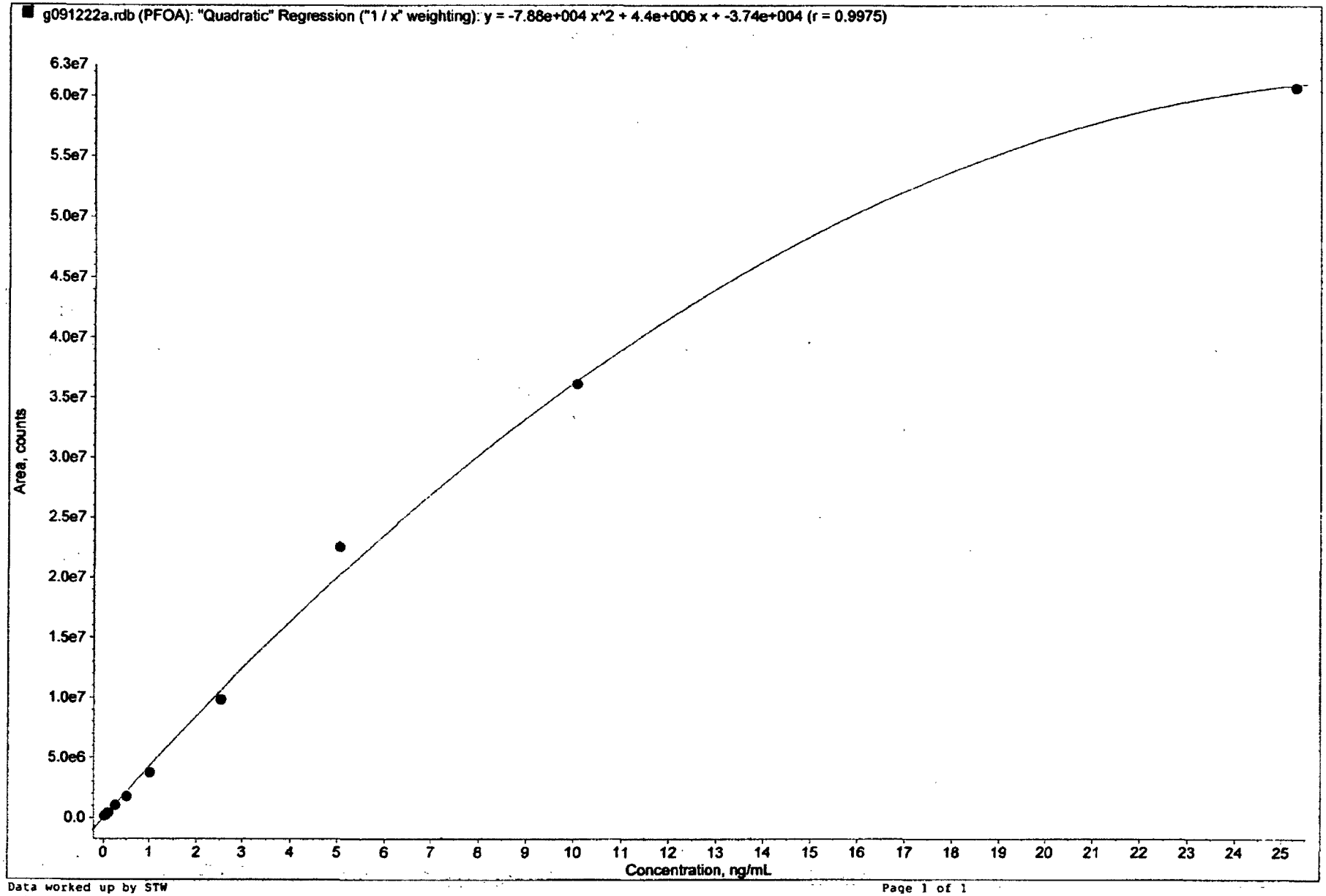
Data worked up by STW
Printing Time: 11:36:48 AM
Printing Date: Thursday, January 28, 2010


Sample Name: "g091222a016" Sample ID: "EC-091216-004" File: "g091222a.wiff"
Peak Name: "PFOS" Mass(es): "499.0/499.0 amu,499.0/80.0 amu,499.0/130.0 amu"
Comment: "1.0 ppb extracted curve point" Annotation: ""
Sample Index: 16
Sample Type: Standard
Concentration: 1.00 ng/mL
Calculated Conc: 0.955 ng/mL
Acq. Date: 12/22/2009
Acq. Time: 3:17:27 PM
Modified: Yes
RT Window: 30.0 sec
Expected RT: 12.6 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 12.6 min
Area: 3885272 counts
Height: 8.19e+005 cps
Start Time: 12.2 min
End Time: 13.1 min
12.58
Intensity, cps
Time, min
Sample Name: "g091222a027" Sample ID: "LCS-091216-3" File: "g091222a.wiff"
Peak Name: "PFOS" Mass(es): "499.0/499.0 amu,499.0/80.0 amu,499.0/130.0 amu"
Comment: "0.2 ppb extracted LCS" Annotation: ""
Sample Index: 27
Sample Type: QC
Concentration: 0.200 ng/mL
Calculated Conc: 0.222 ng/mL
Acq. Date: 12/22/2009
Acq. Time: 6:56:52 PM
Modified: Yes
RT Window: 30.0 sec
Expected RT: 12.6 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 12.6 min
Area: 896885 counts
Height: 2.93e+005 cps
Start Time: 12.3 min
End Time: 13.0 min
12.55
Intensity, cps
Time, min
Sample Name: "g091222a050" Sample ID: "ME-091215-4" File: "g091222a.wiff"
Peak Name: "PFOS" Mass(es): "499.0/99.0 amu,499.0/80.0 amu,499.0/130.0 amu"
Comment: "Method Blank-4" Annotation: ""
Sample Index: 50
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.00733 ng/mL
Acq. Date: 12/23/2009
Acq. Time: 1:29:16 AM
Modified: Yes
RT Window: 29.5 sec
Expected RT: 12.6 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 12.5 min
Area: 15107 counts
Height: 1.77e+05 cps
Start Time: 12.3 min
End Time: 12.7 min
Intensity, cps
Time, min
Sample Name: "g091222a131" Sample ID: "E07-0149-2SS" File: "g091222a.wiff"
Peak Name: "PFOS" Mass(es): "499.0/99.0 amu,499.0/80.0 amu,499.0/130.0 amu"
Comment: "SOIL-GW-C14" Annotation: ""
Sample Index: 131
Sample Type: Unknown
Concentration: N/A
Calculated Conc: 0.03650 ng/mL
Acq. Date: 12/24/2009
Acq. Time: 1:10:54 AM
Modified: Yes
RT Window: 29.8 sec
Expected RT: 12.6 min
Use Relative RT: No
Int. Type: Manual
Retention Time: 12.4 min
Area: 11748 counts
Height: 1.24e+004 cps
Start Time: 12.2 min
End Time: 12.6 min
12.39
15.61
15.53
15.79
15.31
14.01
12.19
12.10
11.73
0.86
5.80
Intensity, cps
Time, min



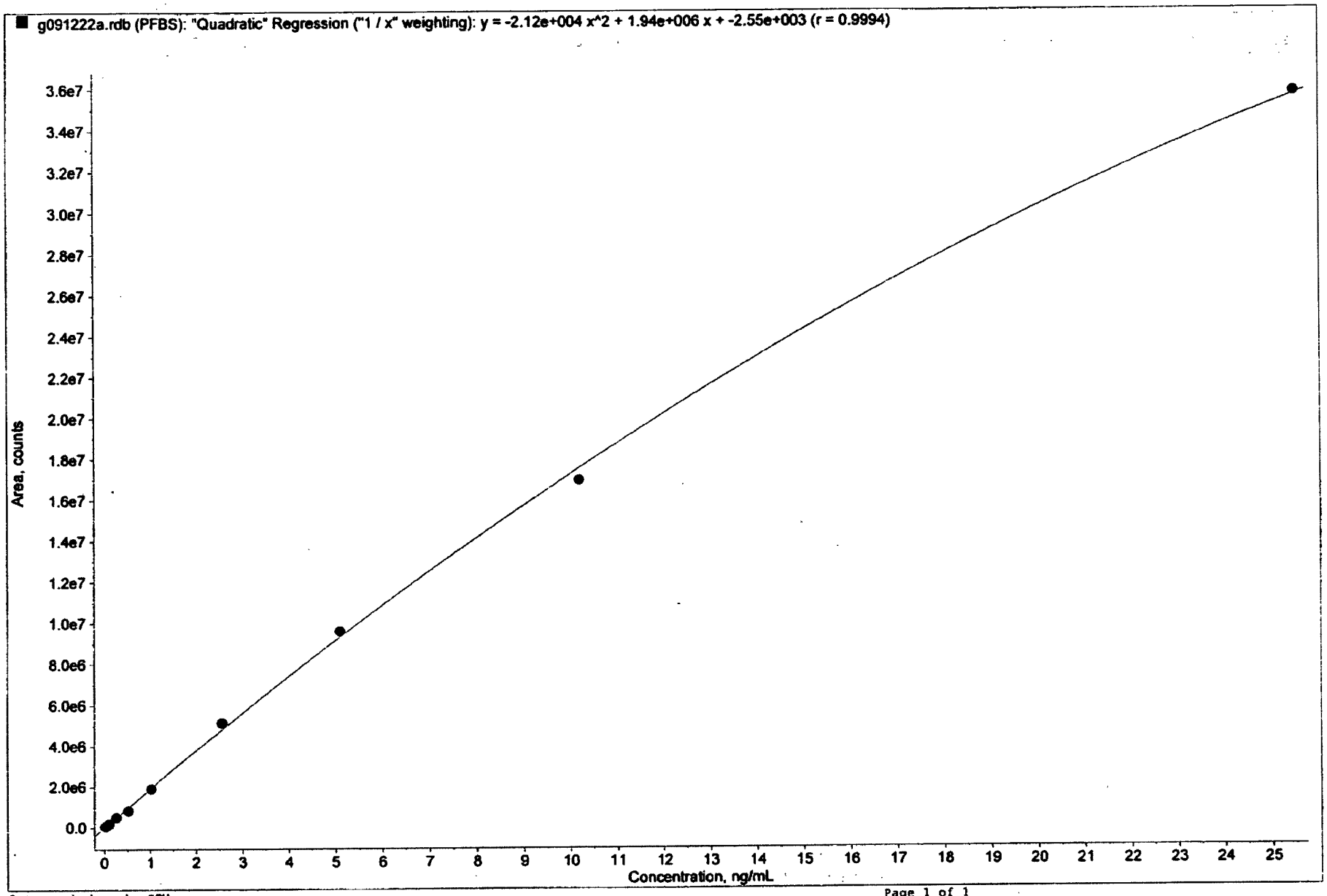
Page 1 of 1

Data worked up by STW
Printing Time: 11:37:28 AM
Printing Date: Thursday, January 28, 2010

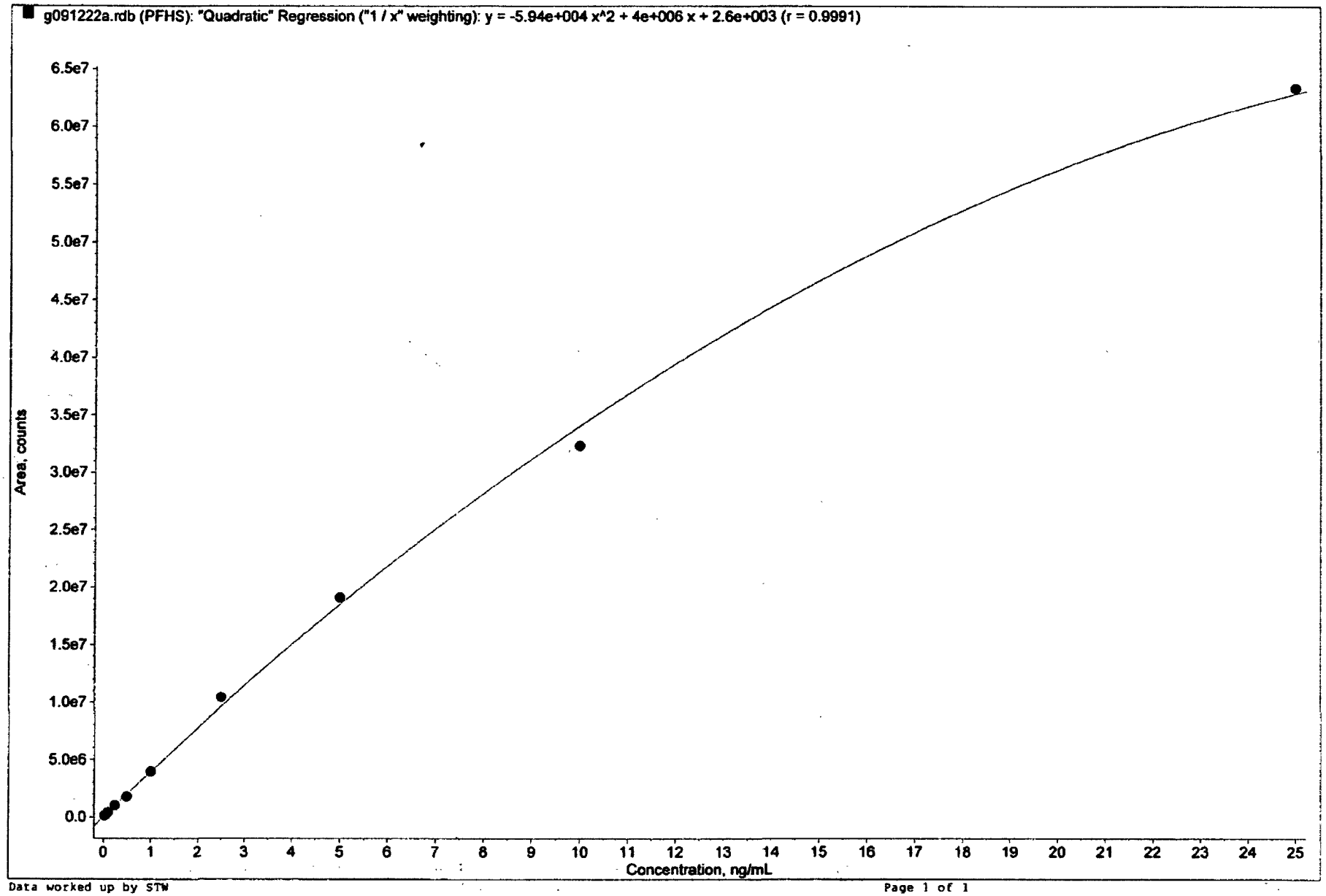


Data worked up by STW
Printing Time: 11:35:08 AM
Printing Date: Thursday, January 28, 2010

Page 1 of 1

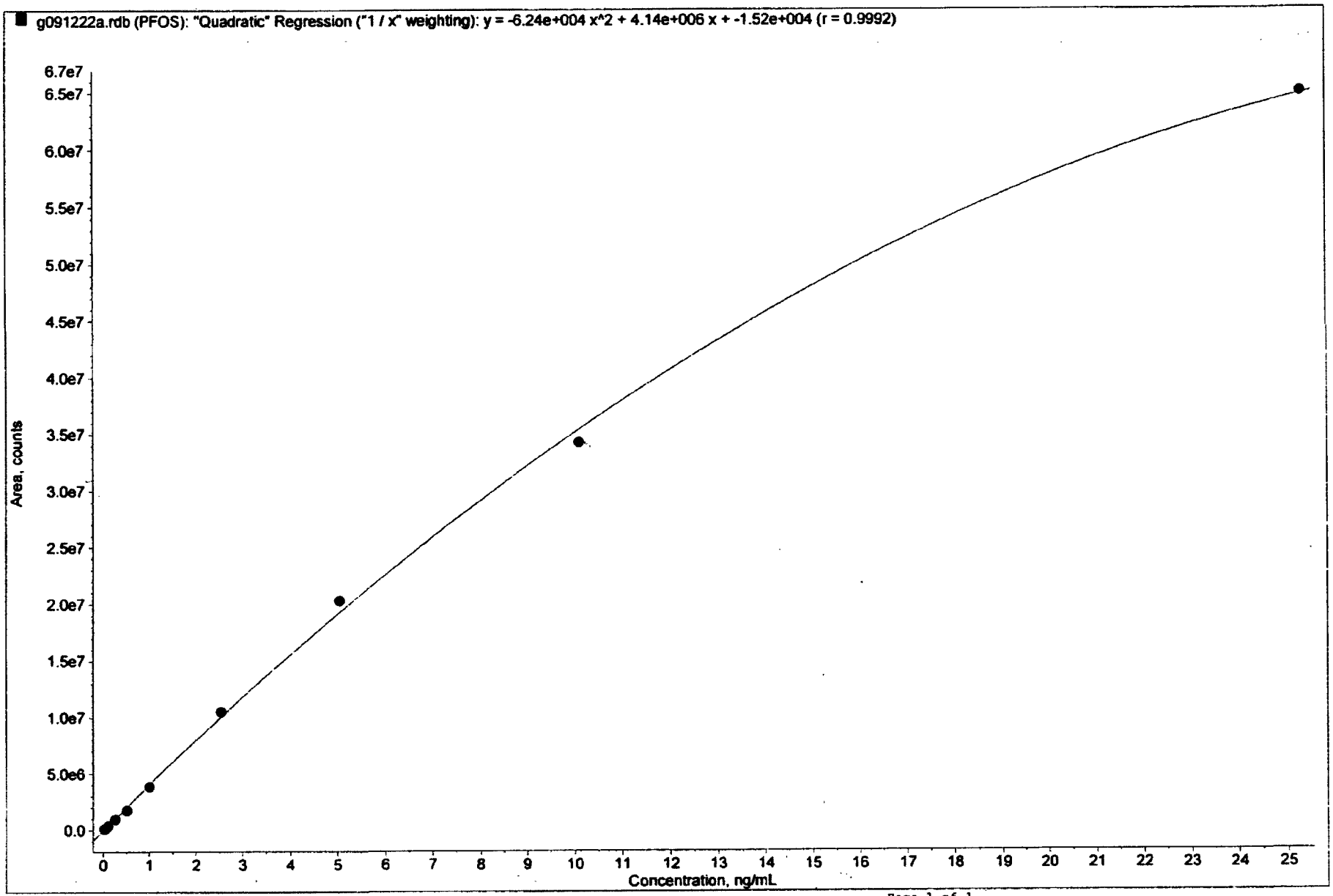


Data worked up by STW
Printing Time: 11:35:20 AM
Printing Date: Thursday, January 28, 2010



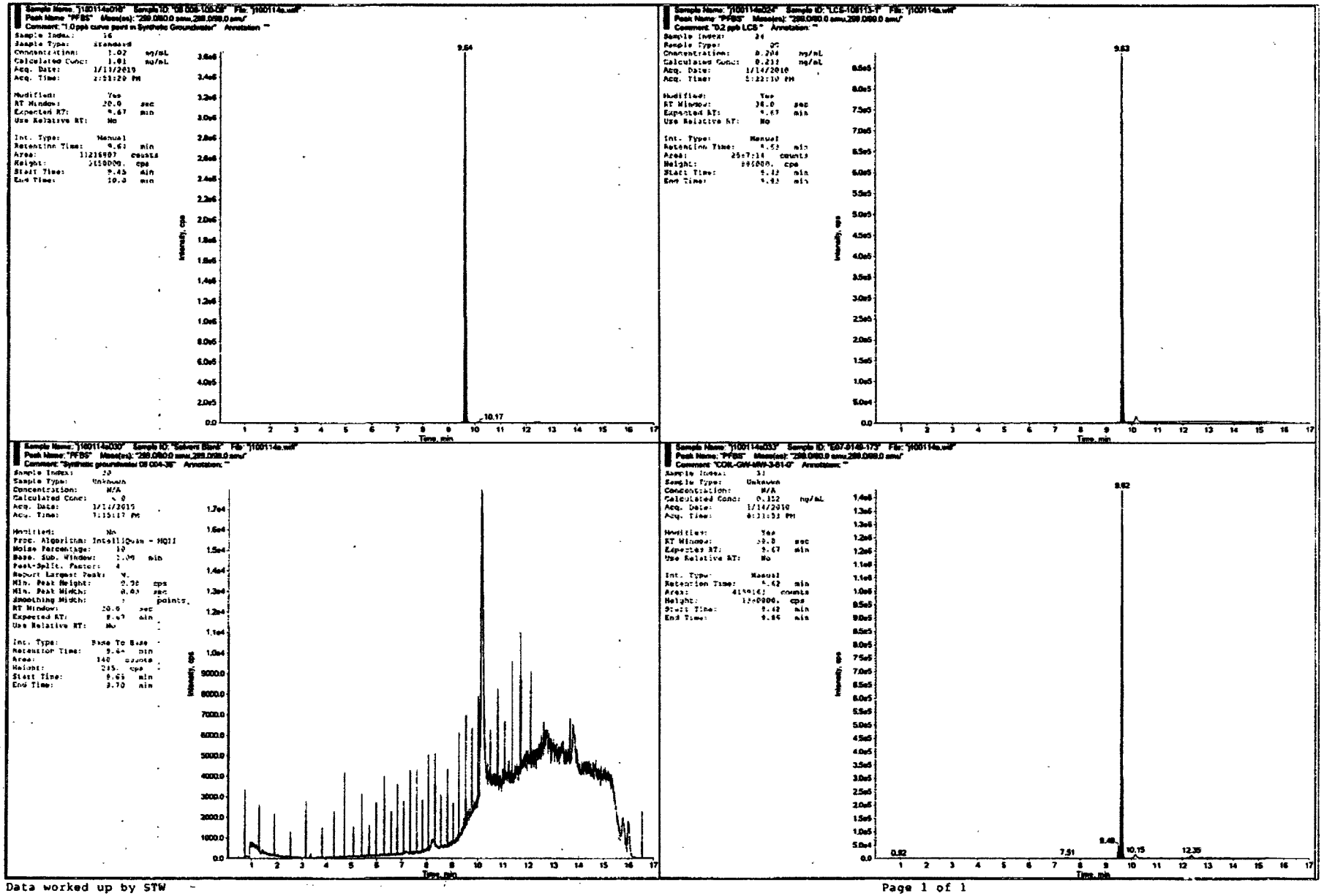
Data worked up by STW
Printing Time: 11:35:36 AM
Printing Date: Thursday, January 28, 2010

Page 1 of 1



Page 1 of 1

Data worked up by STW
Printing Time: 11:35:51 AM
Printing Date: Thursday, January 28, 2010

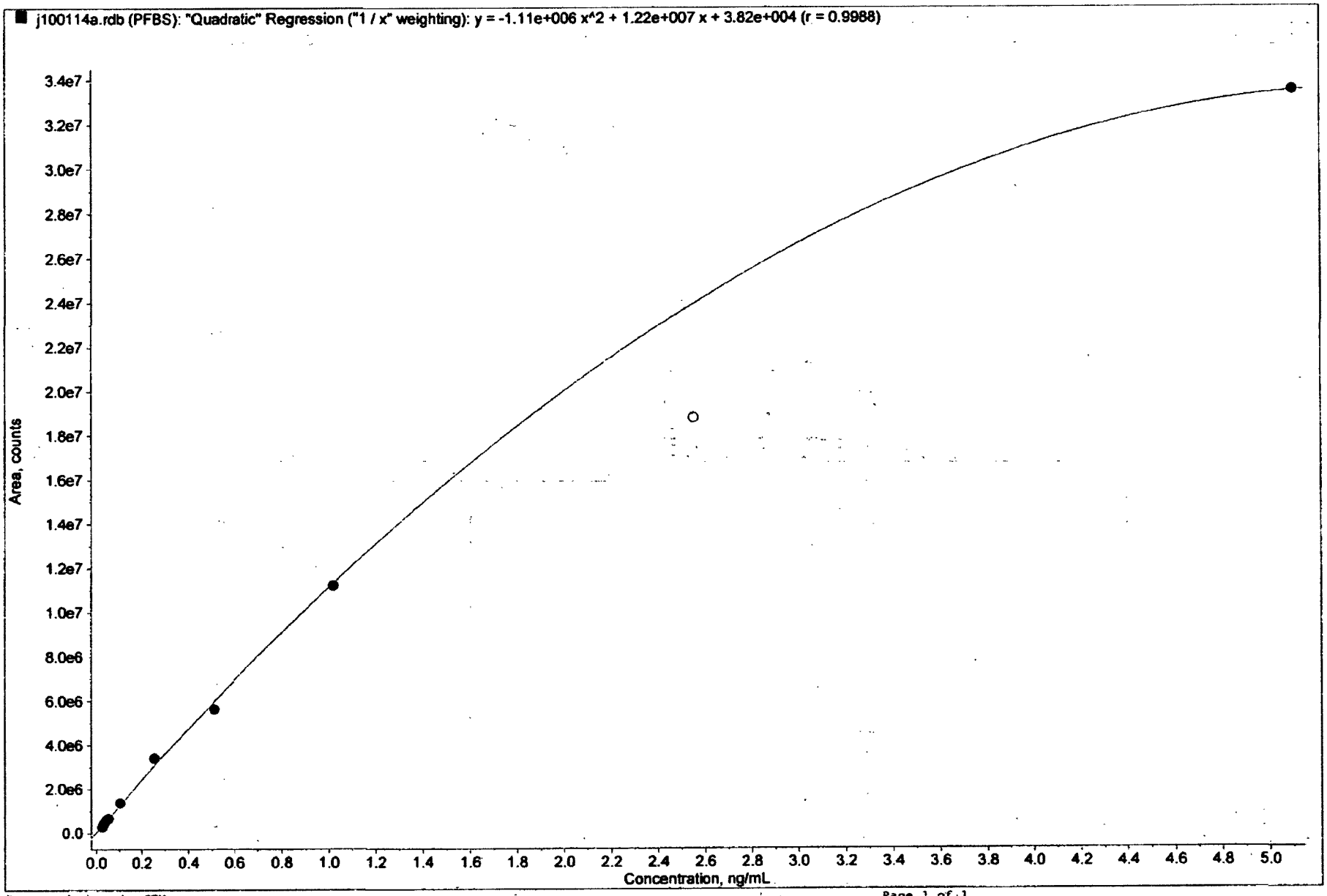


Data worked up by STW

Printing Time: 11:42:08 AM

Printing Date: Thursday, January 28, 2010

Page 1 of 1



Page 1 of 1

Data worked up by STW
Printing Time: 11:40:40 AM
Printing Date: Thursday, January 28, 2010

ATTACHMENT E: PROTOCOL AND METHOD DEVIATIONS

3M Confidential

CONFIDENTIAL

RECORD OF DEVIATION/NONCONFORMANCE

I. Identification

Study / Project No.	Date(s) of Occurrence:	Document Number:
E07-0149 – Interim Report #2	Sequence g091222a and g100105a	ETS-8-154.3

Deviation type (Check one): ☐ SOP ☐ Protocol ☐ Equipment Procedure ☐ GPO ☑ Method ☐ Other:

II. Description (attach extra pages as needed)

Method Requirements:

1. CCV recovery within ± 25% of true value (section 10.4).
2. LCS average recovery of 100% ± 20%.
3. FMS recoveries within ± 30% (section 9.6).

Actual procedure/process:

(1) Sequence g091222a - One of the eleven CCVs for PFOA had a recovery of 72.4%.
(2) LCS of branched PFOS and PFBA both had an average recovery of 126%.
(3) PFOS FMS LS recovery for location COIL-GW-C1 was 66.9%

III. Actions Taken (such as amendment issued, SOP revision, etc.)

Corrective Action (☐ Yes ☑ No) Reference:

Acceptability of the nonconforming work:

1) Eleven CCVs were analyzed during the course of the run with one CCV for PFOA recovering at 72.4%. Curve criteria, LCS criteria, and FMS recovery criteria were all met for PFOA. Samples bracketed by the CCV will be noted in the report.
2) The branched PFOS when quantitated against calibration curve point prepared from linear PFOS have been quantitating around 15-20% higher. The average recovery of 126% for both PFOS and PFBA were within the acceptance criteria of ± 30% for FMS samples.
3) The other two FMS samples for PFOS for location COIL-GW-CI met method acceptance criteria with recoveries of 80.2% and 85.4%. Since the low spike was the most appropriate as compared to the sample concentration, the analytical uncertainty will be adjusted accordingly.

Actions: ☐ Halting of Work ☐ Client Notification ☐ Work Recall ☐ Withholding of Report
☑ Other: Deviations will be noted in final report.

Project Lead/PAI Approval: Susan Wolf — Date: 1/18/10

Study Director (if GLP): — Date: 2/12/10

Sponsor Approval (for GLP protocol deviations): NA — Date: NA

Technical Reviewer (optional): — Date:

Laboratory Department Manager Approval: — Date: 29 Jan 2010

IV. Authorization to Resume Work

Where halting of work occurred, resumption of work must first be approved by Laboratory Management

Laboratory Department Manager Approval: NA — Date: NA

Deviation No. 1

(assigned by Study Director or Team Leader at the end of study or project)

3M Confidential

CONFIDENTIAL

RECORD OF DEVIATION/NONCONFORMANCE

I. Identification

Study / Project No.	Date(s) of Occurrence:	Document Number:
E07-0149 – Interim Report #2	Sequence g091211a and g100107a	ETS-8-110.1

Deviation type (Check one): ☐ SOP ☐ Protocol ☐ Equipment Procedure ☐ GPO ☑ Method ☐ Other:

II. Description (attach extra pages as needed)

Method Requirements:

1. CCV recovery within ± 25% of true value (section 10.3)

Actual procedure/process:

(1) One of the seventeen CCVs for PFBA and PFBS did not meet method acceptance criteria with recoveries 163% and 141%, respectively.

III. Actions Taken

(such as amendment issued, SOP revision, etc.)

Corrective Action (☐ Yes ☑ No) Reference:

Acceptability of the nonconforming work:

1) QC criteria were reviewed and met method acceptance criteria: curve accuracy, LCS and sample FMS recoveries. Samples bracketed by the PFBA and PFBS CCV not meeting method acceptance criteria, will be noted in the report.

Actions: ☐ Halting of Work ☐ Client Notification ☐ Work Recall ☐ Withholding of Report
☑ Other: CCV deviation will be noted in final report.

Project Lead/PAI Approval: Susan Wolf — Date: 1/28/10

Study Director (If GLP): — Date: 2/12/10

Sponsor Approval (for GLP protocol deviations): NA — Date: NA

Technical Reviewer (optional): — Date:

Laboratory Department Manager Approval: — Date: 29 Jan 2010

IV. Authorization to Resume Work

Where halting of work occurred, resumption of work must first be approved by Laboratory Management

Laboratory Department Manager Approval: NA — Date: NA

Deviation No. 2

(assigned by Study Director or Team Leader at the end of study or project)

3M Confidential

CONFIDENTIAL

RECORD OF DEVIATION/NONCONFORMANCE

I. Identification

Study / Project No.	Date(s) of Occurrence:	Document Number:
E07-0149 – Interim Report #2	Sequence g091211a and g100107a	ETS-8-110.1

Deviation type (Check one): ☐ SOP ☐ Equipment Procedure ☐ Method ☑ Protocol ☐ GPO ☐ Other:

II. Description (attach extra pages as needed)

Method Requirements:

1. Field matrix spikes (section 9.1 of Protocol).

Actual procedure/process:

(1) Location COIL-GW-MW-9-94 did not have a field matrix spike level appropriate for PFBA. Several other locations also did not have an appropriate field matrix spike level for PFBA. In those instances, a laboratory matrix spike was prepared at an appropriate level. This sample (COIL-GW-MW-9-94) was inadvertently missed when preparing laboratory matrix spikes.

III. Actions Taken

(such as amendment issued, SOP revision, etc.)

Corrective Action (☐ Yes ☑ No) Reference:

Acceptability of the nonconforming work:

1) Data will be flagged in the report as not having an appropriate matrix spike.

Actions: ☐ Halting of Work ☐ Client Notification ☐ Work Recall ☐ Withholding of Report
☑ Other: Deviation will be noted in final report.

Project Lead/PAI Approval: Susan Wolf — Date: 1/28/10

Study Director (if GLP): — Date: 2/12/10

Sponsor Approval (for GLP protocol deviations): Gary A. Hellerstein — Date: 2/5/10

Technical Reviewer (optional): — Date:

Laboratory Department Manager Approval: — Date: 29 Jan 2010

IV. Authorization to Resume Work

Where halting of work occurred, resumption of work must first be approved by Laboratory Management

Laboratory Department Manager Approval: NA — Date: NA

Deviation No. 3

(assigned by Study Director or Team Leader at the end of study or project)