



Tetra Tech

INTERNAL CORRESPONDENCE

TO: J. ORIENT **DATE:** February 5, 2013
FROM: M. CARSON **COPIES:** DV FILE
SUBJECT: ORGANIC DATA VALIDATION – PFOA/PFOS
NAS BRUNSWICK, CTO 432
SDG 280-36516-1

SAMPLES: 3/Soil/
NASB-B611-SB01-0304 NASB-B611-SB02-0304
NASB-B611-SBDUP01

Overview

The sample set for NAS Brunswick, CTO 432, SDG 280-36516-1 consisted of three (3) soil environmental samples. All of the samples were analyzed for perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). This SDG contains one field duplicate pair: NASB-B611-SB01-0304/ NASB-B611-SBDUP01.

The samples were collected by Tetra Tech on November 28th and 29th, 2012 and analyzed by TestAmerica. All analyses were conducted in accordance with TestAmerica method DV-LC-0012. The data was evaluated based on the following parameters:

- * • Data Completeness
- * • Holding Times
- * • GC/MS Instrument Tuning and System Performance
- * • Blank Results
- * • Initial and Continuing Calibration
- * • Blank Spike Results
- * • Surrogate Spike Recoveries
- * • Internal Standard Recoveries
- * • Matrix Spike/Matrix Spike Duplicate Results
- * • Field Duplicate Precision
- * • Detection Limits

The asterisk (*) indicates that all quality control criteria were met for this parameter. Qualified (if applicable) analytical results are summarized in Appendix A. Results as reported by the laboratory are presented in Appendix B. Appendix C contains documentation to support the findings as discussed in this data validation report. A Tier II validation was performed on the data in this SDG. The text of this report has been formulated to address only those problem areas affecting data quality.

TO: J. Orient
SDG: 280-36516-1

PAGE 2

PFOA/PFOS

All sample results were acceptable for validation purposes.

Notes:

Non-detected results were reported to the Limit of Detection (LOD).

Positive results less than the Limit of Quantitation (LOQ) and less than the Method Detection limit (MDL) were reported as estimated (J).

EXECUTIVE SUMMARY

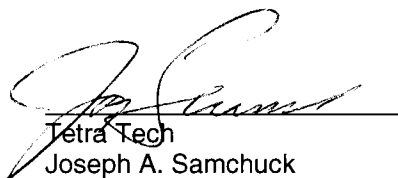
Laboratory Performance: None.

Other Factors Affecting Data Quality: None.

The data for these analyses were reviewed with reference to the criteria listed in the project specific SAP, the "USEPA Region 1 Laboratory Data Validation Functional Guidelines – Part II" (12/96), and the Department of Defense (DOD) document entitled "Quality Systems Manual (QSM) for Environmental Laboratories" (October 2010).



Tetra Tech
Megan Carson
Chemist/Data Validator



Tetra Tech
Joseph A. Samchuck
Quality Assurance Officer

Attachments:

1. Appendix A - Qualified Analytical Results
2. Appendix B - Results as reported by the Laboratory
3. Appendix C - Support Documentation

APPENDIX A

QUALIFIED ANALYTICAL RESULTS

Qualifier Codes:

A	=	Lab Blank Contamination
B	=	Field Blank Contamination
C	=	Calibration Noncompliance (i.e., % RSDs, %Ds, ICVs, CCVs, RRFs, etc.)
C01	=	GC/MS Tuning Noncompliance
D	=	MS/MSD Recovery Noncompliance
E	=	LCS/LCSD Recovery Noncompliance
F	=	Lab Duplicate Imprecision
G	=	Field Duplicate Imprecision
H	=	Holding Time Exceedance
I	=	ICP Serial Dilution Noncompliance
J	=	ICP PDS Recovery Noncompliance; MSA's $r < 0.995$
K	=	ICP Interference - includes ICS % R Noncompliance
L	=	Instrument Calibration Range Exceedance
M	=	Sample Preservation Noncompliance
N	=	Internal Standard Noncompliance
N01	=	Internal Standard Recovery Noncompliance Dioxins
N02	=	Recovery Standard Noncompliance Dioxins
N03	=	Clean-up Standard Noncompliance Dioxins
O	=	Poor Instrument Performance (i.e., base-time drifting)
P	=	Uncertainty near detection limit ($< 2 \times \text{IDL}$ for inorganics and $< \text{CRQL}$ for organics) Other problems (can encompass a number of issues; i.e. chromatography, interferences, etc.)
Q	=	etc.)
R	=	Surrogates Recovery Noncompliance
S	=	Pesticide/PCB Resolution
T	=	% Breakdown Noncompliance for DDT and Endrin
U	=	RPD between columns/detectors $> 40\%$ for positive results determined via GC/HPLC
V	=	Non-linear calibrations; correlation coefficient $r < 0.995$
W	=	EMPC result
X	=	Signal to noise response drop
Y	=	Percent solids $< 30\%$
Z	=	Uncertainty at 2 sigma deviation is less than sample activity
Z1	=	Tentatively Identified Compound considered presumptively present
Z2	=	Tentatively Identified Compound column bleed

PROJ_NO: 00958 SDG: 280-36516-1 FRACTION: MISC MEDIA: SOIL	NSAMPLE	NASB-B611-SB01-0304			NASB-B611-SB02-0304			NASB-B611-SBDUP01		
	LAB_ID	280-36516-2			280-36516-1			280-36516-3		
	SAMP_DATE	11/28/2012			11/29/2012			11/28/2012		
	QC_TYPE	NM			NM			NM		
	UNITS	UG/KG			UG/KG			UG/KG		
	PCT_SOLIDS	94.1			95.1			94.0		
	DUP_OF									
PARAMETER		RESULT	VQL	QLCD	RESULT	VQL	QLCD	RESULT	VQL	QLCD
PENTADECAFLUOROOCTANOIC ACID		0.3	J	P	0.62	U		0.25	J	P
PERFLUOROOCTANE SULFONIC ACID		0.6	U		0.62	U		0.62	U	

APPENDIX B

RESULTS AS REPORTED BY THE LABORATORY

Analytical Data

Client: Tetra Tech, Inc

Job Number: 280-36516-1

Client Sample ID: NASB-B611-SB01-0304

Lab Sample ID: 280-36516-2

Date Sampled: 11/28/2012 0830

Client Matrix: Solid

% Moisture: 5.9

Date Received: 12/01/2012 0900

PFOA/PFOS PFOA/PFOS LC/MS/MS

Analysis Method:	PFOA/PFOS	Analysis Batch:	280-151009	Instrument ID:	LC_LCMS5
Prep Method:	PFC leach	Prep Batch:	280-150302	Lab File ID:	PC512I06026.d
Dilution:	1.0			Initial Weight/Volume:	10.58 g
Analysis Date:	12/06/2012 1708			Final Weight/Volume:	20 mL
Prep Date:	12/04/2012 1050			Injection Volume:	20 uL

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	DL	LOQ
Perfluorooctanoic acid (PFOA)		0.30	J	0.21	0.80
Perfluorooctane Sulfonate (PFOS)		0.60	U	0.19	0.80

Surrogate	%Rec	Qualifier	Acceptance Limits
13C8 PFOA	106		57 - 153
13C8 PFOS	101		70 - 130

Analytical Data

Client: Tetra Tech, Inc

Job Number: 280-36516-1

Client Sample ID: NASB-B611-SB02-0304

Lab Sample ID: 280-36516-1

Date Sampled: 11/29/2012 0920

Client Matrix: Solid

% Moisture: 4.9

Date Received: 12/01/2012 0900

PFOA/PFOS PFOA/PFOS LC/MS/MS

Analysis Method:	PFOA/PFOS	Analysis Batch:	280-151009	Instrument ID:	LC_LCMS5
Prep Method:	PFC leach	Prep Batch:	280-150302	Lab File ID:	PC512I06023.d
Dilution:	1.0			Initial Weight/Volume:	10.12 g
Analysis Date:	12/06/2012 1645			Final Weight/Volume:	20 mL
Prep Date:	12/04/2012 1050			Injection Volume:	20 uL

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	DL	LOQ
Perfluorooctanoic acid (PFOA)		0.62	U	0.22	0.83
Perfluorooctane Sulfonate (PFOS)		0.62	U	0.20	0.83

Surrogate	%Rec	Qualifier	Acceptance Limits
13C8 PFOA	109		57 - 153
13C8 PFOS	104		70 - 130

Analytical Data

Client: Tetra Tech, Inc

Job Number: 280-36516-1

Client Sample ID: NASB-B611-SBDUP01

Lab Sample ID: 280-36516-3

Date Sampled: 11/28/2012 0000

Client Matrix: Solid

% Moisture: 6.0

Date Received: 12/01/2012 0900

PFOA/PFOS PFOA/PFOS LC/MS/MS

Analysis Method:	PFOA/PFOS	Analysis Batch:	280-151009	Instrument ID:	LC_LCMS5
Prep Method:	PFC leach	Prep Batch:	280-150302	Lab File ID:	PC512106027.d
Dilution:	1.0			Initial Weight/Volume:	10.24 g
Analysis Date:	12/06/2012 1716			Final Weight/Volume:	20 mL
Prep Date:	12/04/2012 1050			Injection Volume:	20 uL

Analyte	DryWt Corrected: Y	Result (ug/Kg)	Qualifier	DL	LOQ
Perfluorooctanoic acid (PFOA)		0.25	J	0.22	0.83
Perfluorooctane Sulfonate (PFOS)		0.62	U	0.20	0.83

Surrogate	%Rec	Qualifier	Acceptance Limits
13C8 PFOA	102		57 - 153
13C8 PFOS	105		70 - 130

Analytical Data

Client: Tetra Tech, Inc

Job Number: 280-36516-1

General Chemistry

Client Sample ID: NASB-B611-SB02-0304

Lab Sample ID: 280-36516-1

Date Sampled: 11/29/2012 0920

Client Matrix: Solid

Date Received: 12/01/2012 0900

Analyte	Result	Qual	Units	DL	LOQ	Dil	Method
Percent Moisture	4.9		%	0.10	0.10	1.0	Moisture
Analysis Batch: 280-150423		Analysis Date: 12/04/2012 1506				DryWt Corrected: N	

Analytical Data

Client: Tetra Tech, Inc

Job Number: 280-36516-1

General Chemistry

Client Sample ID: NASB-B611-SB01-0304

Lab Sample ID: 280-36516-2

Date Sampled: 11/28/2012 0830

Client Matrix: Solid

Date Received: 12/01/2012 0900

Analyte	Result	Qual	Units	DL	LOQ	Dil	Method
Percent Moisture	5.9		%	0.10	0.10	1.0	Moisture
Analysis Batch: 280-150423		Analysis Date: 12/04/2012 1506				DryWt Corrected: N	

Analytical Data

Client: Tetra Tech, Inc

Job Number: 280-36516-1

General Chemistry**Client Sample ID:** NASB-B611-SBDUP01**Lab Sample ID:** 280-36516-3**Date Sampled:** 11/28/2012 0000**Client Matrix:** Solid**Date Received:** 12/01/2012 0900

Analyte	Result	Qual	Units	DL	LOQ	Dil	Method
Percent Moisture	6.0		%	0.10	0.10	1.0	Moisture

Analysis Batch: 280-150423 Analysis Date: 12/04/2012 1506 DryWt Corrected: N

APPENDIX C

REGIONAL WORKSHEETS

APPENDIX D

SUPPORT DOCUMENTATION

Page 139 of 140

CASE NARRATIVE
Client: Tetra Tech
Project: NAS Brunswick
Contract Task Order: WE49 / N62470-08-D-1001
Project Manager: Jeff Orient
Report Number: 280-36516-1

With the exceptions noted as flags or footnotes, standard analytical protocols were followed in the analysis of the samples and no problems were encountered or anomalies observed. In addition all laboratory quality control samples were within established control limits, with any exceptions noted below. Each sample was analyzed to achieve the lowest possible reporting limit within the constraints of the method. In some cases, due to interference or analytes present at high concentrations, samples were diluted. For diluted samples, the reporting limits are adjusted relative to the dilution required.

Calculations are performed before rounding to avoid round-off errors in calculated results.

All holding times were met and proper preservation noted for the methods performed on these samples, unless otherwise detailed in the individual sections below.

The PFC method DV-LC-0012 is an isotope dilution method; therefore, the internal standards are added prior to the extraction process. This technique inherently corrects for variability in the extraction efficiency due to sample matrix. Dilution of samples beyond the ability of the instrument to detect the internal standards is not recommended. Analyses performed at a dilution level requiring additional internal standard to be added after the extraction step in order to quantitate results has been shown to yield results with a significant low bias. As a result, data have been reported that exceed the calibration range and are qualified as estimated.

The PFC method is an isotope dilution method where the internal standards are added prior to extraction and used to quantitate results; therefore, the use of dilution factors is inappropriate. Application of dilution factors would yield results that are artificially high. Reporting limits and method detection limits are not adjusted for dilutions unless samples are fortified with additional internal standard, which is not recommended.

Internal standard abundances may vary depending upon both recovery and the dilution at which the analysis is performed. This is an inherent feature of the isotope dilution technique and is not indicative of bias to the reported results.

RECEIPT

The following report contains the analytical results for three samples received at TestAmerica Denver on December 1, 2012, according to documented sample acceptance procedures. The samples were received in good condition at a temperature of 4.4°C. No anomalies were encountered during sample receipt.

PFOA & PFOS

Samples NASB-B611-SB02-0304 (280-36516-1), NASB-B611-SB01-0304 (280-36516-2) and NASB-B611-SBDUP01 (280-36516-3) were analyzed for PFOA/PFOS LC/MS/MS in accordance with LCMS PFOA. The samples were prepared on 12/04/2012 and analyzed on 12/06/2012.

No difficulties were encountered during the LCMS analyses.

All quality control parameters were within the acceptance limits.

PERCENT SOLIDS

Samples NASB-B611-SB02-0304 (280-36516-1), NASB-B611-SB01-0304 (280-36516-2) and NASB-B611-SBDUP01 (280-36516-3) were analyzed for percent solids in accordance with EPA SW846 3550C. The samples were analyzed on 12/04/2012.

No difficulties were encountered during the % solids analyses.

All quality control parameters were within the acceptance limits.

Login Sample Receipt Checklist

Client: Tetra Tech, Inc

Job Number: 280-36516-1

Login Number: 36516

List Source: TestAmerica Denver

List Number: 1

Creator: Wheeler, Virginia L

Question	Answer	Comment
Radioactivity wasn't checked or is $\leq$ background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	True	
Sample custody seals, if present, are intact.	True	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	True	
Cooler Temperature is acceptable.	True	
Cooler Temperature is recorded.	True	
COC is present.	True	
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	True	
Is the Field Sampler's name present on COC?	True	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time.	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	N/A	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is $<6\text{mm}$ (1/4").	N/A	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	

HOLDTIME

SDG 280-36516-1

SORT	UNITS	NSAMPLE	LAB_ID	QC_TYPE	SAMP_DATE	EXTR_DATE	ANAL_DATE	SMP_EXTR	EXTR_ANL	SMP_ANL
ACID	UG/KG	NASB-B611-SBDUP01	280-36516-3	NM	11/28/2012	12/4/2012	12/6/2012	6	2	8
ACID	UG/KG	NASB-B611-SB06-0910	280-36837-3	NM	12/6/2012	12/11/2012	12/13/2012	5	2	7
ACID	UG/KG	NASB-B611-SB05-0708	280-36837-2	NM	12/5/2012	12/11/2012	12/13/2012	6	2	8
ACID	UG/KG	NASB-B611-SB04-0910	280-36837-1	NM	12/5/2012	12/11/2012	12/13/2012	6	2	8
ACID	UG/KG	NASB-B611-SB03-0304	280-36837-4	NM	12/6/2012	12/11/2012	12/13/2012	5	2	7
ACID	UG/KG	NASB-B611-SB02-0304	280-36516-1	NM	11/29/2012	12/4/2012	12/6/2012	5	2	7
ACID	UG/KG	NASB-B611-SB01-0304	280-36516-2	NM	11/28/2012	12/4/2012	12/6/2012	6	2	8
ACID	UG/L	NASB-B611-RB120712	280-36837-5	NM	12/7/2012	12/11/2012	12/13/2012	4	2	6

FORM IV
LCMS METHOD BLANK SUMMARY

Lab Name: TestAmerica Denver Job No.: 280-36516-1
 SDG No.: _____
 Lab File ID: PC512106021.d Lab Sample ID: MB 280-150302/1-A
 Matrix: Solid Date Extracted: 12/04/2012 10:50
 Instrument ID: LC LCMS5 Date Analyzed: 12/06/2012 16:29
 Level: (Low/Med) Low

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES:

CLIENT SAMPLE ID	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED
	LCS 280-150302/2-A	PC512106022 .d	12/06/2012 16:37
NASB-B611-SB02-0304	280-36516-1	PC512106023 .d	12/06/2012 16:45
NASB-B611-SB02-0304 MS	280-36516-1 MS	PC512106024 .d	12/06/2012 16:52
NASB-B611-SB02-0304 MSD	280-36516-1 MSD	PC512106025 .d	12/06/2012 17:00
NASB-B611-SB01-0304	280-36516-2	PC512106026 .d	12/06/2012 17:08
NASB-B611-SBDUP01	280-36516-3	PC512106027 .d	12/06/2012 17:16

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Denver Job No.: 280-36516-1
 SDG No.: _____
 Client Sample ID: _____ Lab Sample ID: MB 280-150302/1-A
 Matrix: Solid Lab File ID: PC512106021.d
 Analysis Method: PFOA/PFOS Date Collected: _____
 Extraction Method: PFC leach Date Extracted: 12/04/2012 10:50
 Sample wt/vol: 10.09(g) Date Analyzed: 12/06/2012 16:29
 Con. Extract Vol.: 20(mL) Dilution Factor: 1
 Injection Volume: 20(uL) GC Column: Gemini-NX ID: _____
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 151009 Units: ug/Kg

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	LOD	DL
335-67-1	Perfluorooctanoic acid (PFOA)	0.59	U	0.79	0.59	0.21
1763-23-1	Perfluorooctane Sulfonate (PFOS)	0.59	U	0.79	0.59	0.19

CAS NO.	SURROGATE	%REC	Q	LIMITS
STL01052	13C8 PFOA	110		57-153
STL01054	13C8 PFOS	106		70-130

FORM I
LCMS ORGANICS ANALYSIS DATA SHEET

Lab Name: TestAmerica Denver Job No.: 280-36516-1
 SDG No.: _____
 Client Sample ID: _____ Lab Sample ID: CCB 280-151009/12
 Matrix: Solid Lab File ID: PC512106017.d
 Analysis Method: PFOA/PFOS Date Collected: _____
 Extraction Method: _____ Date Extracted: _____
 Sample wt/vol: 1 (mL) Date Analyzed: 12/06/2012 15:58
 Con. Extract Vol.: 1.0 (mL) Dilution Factor: 1
 Injection Volume: 20 (uL) GC Column: Gemini-NX ID: _____
 % Moisture: _____ GPC Cleanup: (Y/N) N
 Analysis Batch No.: 151009 Units: ug/L

CAS NO.	COMPOUND NAME	RESULT	Q	LOQ	LOD	DL
335-67-1	Perfluorooctanoic acid (PFOA)	0.0080	U	0.55	0.0080	0.0045
1763-23-1	Perfluorooctane Sulfonate (PFOS)	0.020	U	0.15	0.020	0.0098

CAS NO.	SURROGATE	%REC	Q	LIMITS
STL01052	13C8 PFOA			
STL01054	13C8 PFOS			

FORM III
LCMS LAB CONTROL SAMPLE RECOVERY

Lab Name: TestAmerica Denver Job No.: 280-36516-1
SDG No.: _____
Matrix: Solid Level: Low Lab File ID: PC512106022.d
Lab ID: LCS 280-150302/2-A Client ID: _____

COMPOUND	SPIKE ADDED (ug/Kg)	LCS CONCENTRATION (ug/Kg)	LCS % REC	QC LIMITS REC	#
Perfluorooctanoic acid (PFOA)	19.9	22.0	110	70-130	
Perfluorooctane Sulfonate (PFOS)	19.0	20.3	107	74-115	

Column to be used to flag recovery and RPD values

FORM III PFOA/PFOS

FORM III
LCMS MATRIX SPIKE RECOVERY

Lab Name: TestAmerica Denver Job No.: 280-36516-1
SDG No.: _____
Matrix: Solid Level: Low Lab File ID: PC512106024.d
Lab ID: 280-36516-1 MS Client ID: NASB-B611-SB02-0304 MS

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC	QC LIMITS REC	#
Perfluorooctanoic acid (PFOA)	20.0	0.62 U	21.9	110	70-130	
Perfluorooctane Sulfonate (PFOS)	19.1	0.62 U	20.0	105	74-115	

Column to be used to flag recovery and RPD values

FORM III PFOA/PFOS

FORM III
LCMS MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: TestAmerica Denver Job No.: 280-36516-1
 SDG No.: _____
 Matrix: Solid Level: Low Lab File ID: PC512106025.d
 Lab ID: 280-36516-1 MSD Client ID: NASB-B611-SB02-0304 MSD

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC	% RPD	QC LIMITS		#
					RPD	REC	
Perfluorooctanoic acid (PFOA)	19.6	21.5	110	2	20	70-130	
Perfluorooctane Sulfonate (PFOS)	18.7	20.7	111	4	20	74-115	

Column to be used to flag recovery and RPD values

FORM III PFOA/PFOS

FORM III
LCMS DETECTION LIMIT CHECK STANDARD RECOVERY

Lab Name: TestAmerica Denver Job No.: 280-36516-1
SDG No.: _____
Matrix: Solid Level: Low Lab File ID: PC512106019.d
Lab ID: DLCK 280-151009/14 Client ID: _____

COMPOUND	SPIKE ADDED (ug/L)	DLCK CONCENTRATION (ug/L)	DLCK % REC	QC LIMITS REC	#
Perfluorooctanoic acid (PFOA)	0.200	0.227 J	114	70-130	
Perfluorooctane Sulfonate (PFOS)	0.191	0.211	111	70-130	

Column to be used to flag recovery and RPD values

FORM III PFOA/PFOS

FORM II
LCMS SURROGATE RECOVERY

Lab Name: TestAmerica Denver

Job No.: 280-36516-1

SDG No.: _____

Matrix: Solid

Level: Low

GC Column (1): Gemini-NX ID: _____

Client Sample ID	Lab Sample ID	PFOA #	PFOS #
NASB-B611-SB02-030 4	280-36516-1	109	104
NASB-B611-SB01-030 4	280-36516-2	106	101
NASB-B611-SBDUP01	280-36516-3	102	105
	MB 280-150302/1-A	110	106
	LCS 280-150302/2-A	109	106
NASB-B611-SB02-030 4 MS	280-36516-1 MS	106	104
NASB-B611-SB02-030 4 MSD	280-36516-1 MSD	106	103
	DLCK 280-151009/14	112	98

PFOA = 13C8 PFOA
PFOS = 13C8 PFOS

QC LIMITS
57-153
70-130

Column to be used to flag recovery values

FORM II PFOA/PFOS

FORM VI
LCMS INITIAL CALIBRATION DATA
INTERNAL STANDARD CURVE EVALUATION

Lab Name: TestAmerica Denver Job No.: 280-36516-1 Analy Batch No.: 151009

SDG No.: _____

Instrument ID: LC_LCMS5 GC Column: Gemini-NX ID: _____ Heated Purge: (Y/N) N

Calibration Start Date: 12/06/2012 14:49 Calibration End Date: 12/06/2012 15:51 Calibration ID: 11959

Calibration Files:

LEVEL:	LAB SAMPLE ID:	LAB FILE ID:
Level 1	STD0002 280-151009/3	PC512106008.d
Level 2	STD0005 280-151009/4	PC512106009.d
Level 3	STD0010 280-151009/5	PC512106010.d
Level 4	STD0020 280-151009/6	PC512106011.d
Level 5	STD0050 280-151009/7	PC512106012.d
Level 6	STD0100 280-151009/8	PC512106013.d
Level 7	STD0200 280-151009/9	PC512106014.d
Level 8	STD0500 280-151009/10	PC512106015.d
Level 9	STD1250 280-151009/11	PC512106016.d

ANALYTE	RRF					CURVE TYPE	COEFFICIENT			#	MIN RRF	%RSD	#	MAX %RSD	R^2 OR COD	#	MIN R^2 OR COD
	LVL 1 LVL 6	LVL 2 LVL 7	LVL 3 LVL 8	LVL 4 LVL 9	LVL 5		B	M1	M2								
Ammonium Perfluorooctanoate (APFO)	1.0297 0.9146	0.8464 0.8437	1.0209 0.7627	0.9170 0.6949	0.9131	Lin2	0.0397	0.8449							0.9860		0.9800
Perfluorooctanoic acid (PFOA)	1.0532 0.9517	0.8808 0.8780	1.0624 0.7937	0.9542 0.7232	0.9501	Lin2	0.0363	0.8805							0.9860		0.9800
Perfluorooctane Sulfonate (PFOS)	1.5408 1.1841	1.3624 1.0843	1.1008 0.9835	1.1310 0.9371	1.1853	Lin2	0.0961	1.0686							0.9920		0.9800
13C8 PFOA	0.9680 0.8644	0.7938 0.8644	0.9440 0.7616	0.8723 0.6910	0.8826	Lin2	0.0277	0.8218							0.9900		0.9800
13C8 PFOS	0.9833 0.9402	0.8099 0.8532	0.8035 0.7944	0.8561 0.7321	0.9131	Lin2	0.0234	0.8298							0.9920		0.9800

Note: The m1 coefficient is the same as Ave RRF for an Ave curve type.

FORM VI
LCMS INITIAL CALIBRATION DATA
INTERNAL STANDARD RESPONSE AND CONCENTRATION

Lab Name: TestAmerica Denver Job No.: 280-36516-1 Analy Batch No.: 151009

SDG No.: _____

Instrument ID: LC_LCMS5 GC Column: Gemini-NX ID: _____ Heated Purge: (Y/N) N

Calibration Start Date: 12/06/2012 14:49 Calibration End Date: 12/06/2012 15:51 Calibration ID: 11959

Calibration Files:

LEVEL:	LAB SAMPLE ID:	LAB FILE ID:
Level 1	STD0002 280-151009/3	PC512106008.d
Level 2	STD0005 280-151009/4	PC512106009.d
Level 3	STD0010 280-151009/5	PC512106010.d
Level 4	STD0020 280-151009/6	PC512106011.d
Level 5	STD0050 280-151009/7	PC512106012.d
Level 6	STD0100 280-151009/8	PC512106013.d
Level 7	STD0200 280-151009/9	PC512106014.d
Level 8	STD0500 280-151009/10	PC512106015.d
Level 9	STD1250 280-151009/11	PC512106016.d

ANALYTE	IS REF	CURVE TYPE	RESPONSE					CONCENTRATION (UG/L)				
			LVL 1 LVL 6	LVL 2 LVL 7	LVL 3 LVL 8	LVL 4 LVL 9	LVL 5	LVL 1 LVL 6	LVL 2 LVL 7	LVL 3 LVL 8	LVL 4 LVL 9	LVL 5
Ammonium Perfluorooctanoate (APFO)	OA	Lin2	200824 6715104	433666 15055053	1046692 32081782	1584027 71651826	4218103	0.208 10.4	0.520 20.8	1.04 52.0	2.08 130	5.20
Perfluorooctanoic acid (PFOA)	OA	Lin2	197395 6715104	433666 15055053	1046692 32081782	1584027 71651826	4218103	0.200 10.0	0.500 20.0	1.00 50.0	2.00 125	5.00
Perfluorooctane Sulfonate (PFOS)	PFOS	Lin2	86835 2655290	199344 5914707	340310 12380979	582150 27878692	1654329	0.191 9.56	0.478 19.1	0.956 47.8	1.91 120	4.78
13C8 PFOA	OA	Lin2	181429 6099370	390824 14821511	930043 30783598	1448122 68462190	3918216	0.200 10.0	0.500 20.0	1.00 50.0	2.00 125	5.00
13C8 PFOS	PFOS	Lin2	55415 2108280	118506 4653837	248395 10000726	440636 21779667	1274455	0.191 9.56	0.478 19.1	0.956 47.8	1.91 120	4.78

Curve Type Legend:

Lin2 = Linear 1/conc^2 ISTD

FORM VII
LCMS CONTINUING CALIBRATION DATA

Lab Name: TestAmerica Denver Job No.: 280-36516-1
 SDG No.: _____
 Lab Sample ID: CCV 280-151009/23 Calibration Date: 12/06/2012 17:23
 Instrument ID: LC_LCMS5 Calib Start Date: 12/06/2012 14:49
 GC Column: Gemini-NX ID: _____ Calib End Date: 12/06/2012 15:51
 Lab File ID: PC512106028.d Conc. Units: ug/L

ANALYTE	CURVE TYPE	AVE RRF	RRF	MIN RRF	CALC AMOUNT	SPIKE AMOUNT	%D	MAX %D
Ammonium Perfluorooctanoate (APFO)	Lin2		0.9025		5.51	5.20	5.9	30.0
Perfluorooctanoic acid (PFOA)	Lin2		0.9392		5.29	5.00	5.8	30.0
Perfluorooctane Sulfonate (PFOS)	Lin2		1.107		4.86	4.78	1.8	30.0
13C8 PFOA	Lin2		0.8452		5.11	5.00	2.2	30.0
13C8 PFOS	Lin2		0.8512		4.87	4.78	2.0	30.0

FORM VIII
LCMS INTERNAL STANDARD AREA AND RETENTION TIME SUMMARY

Lab Name: TestAmerica Denver Job No.: 280-36516-1
 SDG No.: _____
 Instrument ID: LC_LCMS5 Calibration Start Date: 12/06/2012 14:49
 GC Column: Gemini-NX ID: _____ Calibration End Date: 12/06/2012 15:51
 Calibration ID: 11959

		OA		PFOS			
		AREA #	RT #	AREA #	RT #	AREA #	RT #
INITIAL CALIBRATION MEAN AREA AND MEAN RT		8654517	4.42	2674226	4.61		
UPPER LIMIT		13414501	4.92	3476494	5.11		
LOWER LIMIT		5192710	3.92	1203402	4.11		
LAB SAMPLE ID	CLIENT SAMPLE ID						
CCB 280-151009/12		10170231	4.41	3157336	4.60		
ICV 280-151009/13		10503513	4.41	3163736	4.60		
DLCK 280-151009/14		10105813	4.41	2855335	4.59		
MB 280-150302/1-A		10260601	4.41	3173003	4.60		
LCS 280-150302/2-A		10120723	4.41	3290541	4.60		
280-36516-1	NASB-B611-SB02-0304	11422061	4.41	3465110	4.59		
280-36516-1 MS	NASB-B611-SB02-0304 MS	11093244	4.41	3335362	4.58		
280-36516-1 MSD	NASB-B611-SB02-0304 MSD	9533709	4.40	2784541	4.58		
280-36516-2	NASB-B611-SB01-0304	9417546	4.41	2679302	4.58		
280-36516-3	NASB-B611-SBDUP01	8825635	4.40	2797991	4.58		
CCV 280-151009/23		9412472	4.40	3016777	4.58		

OA = 13C4 PFOA (IS)
 PFOS = 13C4 PFOS (IS)

Area Limit = 60%-155% of internal standard area
 RT Limit = $\pm$ 0.5 minutes of internal standard RT

Column used to flag values outside QC limits

FORM VIII PFOA/PFOS

LCMS ANALYSIS RUN LOG

Lab Name: TestAmerica DenverJob No.: 280-36516-1

SDG No.: _____

Instrument ID: LC_LCMS5Start Date: 12/06/2012 14:26Analysis Batch Number: 151009End Date: 12/06/2012 17:23

LAB SAMPLE ID	CLIENT SAMPLE ID	DATE ANALYZED	DILUTION FACTOR	LAB FILE ID	COLUMN ID
ZZZZZ		12/06/2012 14:26	1		Gemini-NX
ICB 280-151009/2		12/06/2012 14:41	1		Gemini-NX
STD0002 280-151009/3 IC		12/06/2012 14:49	1	PC512106008.d	Gemini-NX
STD0005 280-151009/4 IC		12/06/2012 14:56	1	PC512106009.d	Gemini-NX
STD0010 280-151009/5 IC		12/06/2012 15:04	1	PC512106010.d	Gemini-NX
STD0020 280-151009/6 ICISAV		12/06/2012 15:12	1	PC512106011.d	Gemini-NX
STD0050 280-151009/7 IC		12/06/2012 15:20	1	PC512106012.d	Gemini-NX
STD0100 280-151009/8 IC		12/06/2012 15:27	1	PC512106013.d	Gemini-NX
STD0200 280-151009/9 IC		12/06/2012 15:35	1	PC512106014.d	Gemini-NX
STD0500 280-151009/10 IC		12/06/2012 15:43	1	PC512106015.d	Gemini-NX
STD1250 280-151009/11 IC		12/06/2012 15:51	1	PC512106016.d	Gemini-NX
CCB 280-151009/12		12/06/2012 15:58	1	PC512106017.d	Gemini-NX
ICV 280-151009/13		12/06/2012 16:06	1	PC512106018.d	Gemini-NX
DLCK 280-151009/14		12/06/2012 16:14	1	PC512106019.d	Gemini-NX
ZZZZZ		12/06/2012 16:22	1		Gemini-NX
MB 280-150302/1-A		12/06/2012 16:29	1	PC512106021.d	Gemini-NX
LCS 280-150302/2-A		12/06/2012 16:37	1	PC512106022.d	Gemini-NX
280-36516-1	NASB-B611-SB02-0304	12/06/2012 16:45	1	PC512106023.d	Gemini-NX
280-36516-1 MS	NASB-B611-SB02-0304 MS	12/06/2012 16:52	1	PC512106024.d	Gemini-NX
280-36516-1 MSD	NASB-B611-SB02-0304 MSD	12/06/2012 17:00	1	PC512106025.d	Gemini-NX
280-36516-2	NASB-B611-SB01-0304	12/06/2012 17:08	1	PC512106026.d	Gemini-NX
280-36516-3	NASB-B611-SBDUP01	12/06/2012 17:16	1	PC512106027.d	Gemini-NX
CCV 280-151009/23		12/06/2012 17:23	1	PC512106028.d	Gemini-NX

13-IN
ANALYSIS RUN LOG
GENERAL CHEMISTRY

Lab Name: TestAmerica Denver Job No.: 280-36516-1

SDG No.: _____

Instrument ID: NoEquip Method: Moisture

Start Date: 12/04/2012 15:06 End Date: 12/04/2012 15:06

Lab Sample ID	D / F	T y p e	Time	Analytes															
				M o i s t															
280-36516-1	1	T	15:06	X															
280-36516-1 DU	1	T	15:06	X															
280-36516-2	1	T	15:06	X															
280-36516-3	1	T	15:06	X															
ZZZZZZ			15:06																

Prep Types

T = Total/NA

Wet Chemistry Data Review Checklist For Gravimetric Methods

Test Name/Method #: Moisture Analysis Date: 12/4/12
 SOP #: W-WC-0023 Analyst: Alex Benson Instrument: BAL

Lot / Sample Number	Matrix	Prep Batch	Batch	Method	Special Inst
<u>36265</u>	<u>12-13</u>	<u>solid</u>	<u>150308</u>	<u>—</u>	<u>—</u>
<u>36516</u>	<u>12-7</u>	<u>solid</u>	<u>150423</u>	<u>—</u>	<u>—</u>
<u>36547</u>	<u>12-6</u>	<u>solid</u>	<u>↓</u>	<u>—</u>	<u>—</u>

A. Balance, Oven, and DI Water QC Checks	Yes	No	N/A	2 nd Level
1. Was the balance calibration verified before and after processing samples and noted in the "Balance Calibration Log" for the date(s) the samples were processed?	☒	☐	☐	☒
2. Was the oven temperature within method requirements and recorded in the "Oven Temperature" logbook for the date(s) the samples were processed?	☒	☐	☐	☒
3. Was the daily conductivity check of the deionized water recorded in the "Conductivity Logbook"?	☐	☐	☒	☒
B. Method Requirements				
1. If sample is visibly oily, was this noted on the benchsheet?	☐	☒	☐	☒
2. Was final residue weight within minimum/maximum requirements?	☒	☐	☐	☒
3. Were the initial and final drying dates and times recorded on the benchsheet and were all samples dried for at least one hour?	☒	☐	☐	☒
C. Sample Results				
1. TDS/Conductivity ratio or historical data checked?	☐	☐	☒	☒
2. For % Moisture, was the Final Dried Weight < the Initial Pan Weight or is the result greater than 100%?	☐	☒	☐	☒
3. Were sample analyses done within holding time?	☒	☐	☐	☒
4. Were special client requirements met?	☒	☐	☐	☒
5. Were data that were manually transcribed from instrument printouts into TALS verified 100% including significant figures and units?	☒	☐	☐	☒
6. Do the prep and analysis dates in TALS reflect the actual dates? Lots/Dates report checked?	☒	☐	☐	☒
7. STD/True Value information is updated and included?	☐	☐	☒	☒
8. Are raw data copies prepared, scanned, and uploaded?	☐	☐	☒	☒
D. Preparation/Matrix QC				
1. Method blank < RL or all reported samples > 10 X RL?	☐	☐	☒	☒
2. Method blank < 1/2 RL or NCM provided?	☐	☐	☒	☒
3. LCS/LCSD run for batch and within QC limits?	☐	☐	☒	☒
4. DUP run for batch and RPD < 20% for samples > 5 X RL?	☒	☐	☐	☒

Analyst: Alexander Benson Date: 12/5/12

Comments:

2nd Level Reviewer: BS Date: 12/5/12

Comments:

GENERAL CHEMISTRY BATCH WORKSHEET

Lab Name: TestAmerica DenverJob No.: 280-36516-1

SDG No.: _____

Batch Number: 150423Batch Start Date: 12/04/12 15:06Batch Analyst: Benson, Alex FBatch Method: Moisture

Batch End Date: _____

Lab Sample ID	Client Sample ID	Method Chain	Basis	DISH#	DishWeight	SampleMassWet	SampleMassDry		
280-36516-A-1	NASB-B611-SB02-0304	Moisture	T	1	1.33 g	17.56 g	16.76 g		
280-36516-A-1 DU	NASB-B611-SB02-0304	Moisture	T	2	1.33 g	17.58 g	16.71 g		
280-36516-A-2	NASB-B611-SB01-0304	Moisture	T	3	1.33 g	17.35 g	16.40 g		
280-36516-A-3	NASB-B611-SBDUP01	Moisture	T	4	1.34 g	17.59 g	16.62 g		

Batch Notes

Balance ID	H31422 No Unit
Date samples were placed in the oven	12/4/2012
Oven Temp when samples are put in oven	103 Degrees C
Time samples were place in the oven	15:14
Date samples were removed from oven	12/5/2012
Oven Temp when samples removed from oven	103 Degrees C
Time Samples were removed from oven	8:10
Oven ID	moisture oven
ID number of the thermometer	E
Uncorrected In Temperature	103 Celsius
Uncorrected Out Temperature	103 Celsius

Basis	Basis Description
T	Total/NA

Moisture

Page 1 of 1

DODCMD_ID	INSTALLATION_ID	SDG	SITE_NAME	NORM_SITE_NAME	LOCATION_NAME	LOCATION_TYPE_DESC	COORD_X	COORD_Y	CONTRACT_ID	DO_CTO_NUMBER	CONTR_NAME	SAMPLE_NAME	SAMPLE_MATRIX_DESC	SAMPLE_TYPE_DESC	COLLECT_DATE	ANALYTICAL_METHOD	ANALYTICAL_METHOD_GRP_DESC
MID_ATLANTIC	BRUNSWICK_NAS	280-36516-1	RCRA CLOSURE	SITE 00011	B611-MW01S	Monitoring well	3015059.27	384392.01	N6246704D0055	432	TETRA TECH NUS, INC.	NASB-B611-SB01-0304	Soil	Normal (Regular)	28-Nov-12	TA_WS-LC-0025	Perfluoroalkyl Compounds
MID_ATLANTIC	BRUNSWICK_NAS	280-36516-1	RCRA CLOSURE	SITE 00011	B611-MW02	Monitoring well	3015020.33	384370.14	N6246704D0055	432	TETRA TECH NUS, INC.	NASB-B611-SB02-0304	Soil	Normal (Regular)	29-Nov-12	TA_WS-LC-0025	Perfluoroalkyl Compounds
MID_ATLANTIC	BRUNSWICK_NAS	280-36516-1	RCRA CLOSURE	SITE 00011	B611-MW01S	Monitoring well	3015059.27	384392.01	N6246704D0055	432	TETRA TECH NUS, INC.	NASB-B611-SB01-0304-D	Soil	Field duplicate	28-Nov-12	TA_WS-LC-0025	Perfluoroalkyl Compounds