SAND2018-8441C

Sample Management and Quality Assurance / Quality Control

PRESENTED BY

Corey White

ENVIRONMENT SAFETY & HEALTH

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia LLC, a wholly owned subsidiary of Honeywell International Inc. for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

Outline

- Sample Management Office
- Sample Custody
- Sample Packaging
- Quality Control Samples
- Laboratory Oversight
- Data Verification and Validation
- Laboratory Communication

Sample Management Office

Provide centralized management of samples and analyses performed by contract laboratories

- Ensure that analytical data is of adequate technical quality and content to meet customer data quality objectives (DQO's) and project requirements

- Provide customers an option for analytical services integrated with on-site capabilities

Sample Custody

A Sample is considered to be in ones custody if

- It is physically in one's possession
- It is in the person's view after being in their physical possession
- It has been secured to prevent tampering, after it was physically in one's possession
- It is placed in a designated secure area

Slide 4

WCR1 get reference on this

White, Corey Robert, 6/14/2018

Sample Custody

Chain of Custody

Items to include on COC per the EPA National Functional Guidelines

- Sample matrix
- Field blanks and trip blanks (if applicable)
- Field duplicates (if applicable)
- Field spikes (if applicable)
- PE samples (if applicable)
- Sampling dates
- Sampling times
- Shipping dates
- Preservatives
- Types of analysis
- Contractor laboratory
- Transfer of custody names, dates, times

Chain of Custody

SMO 2012-ARCOC (4-2012)

CONTRACT LABORATORY
ANALYSIS REQUEST AND CHAIN OF CUSTODY

AOP 95-16

Internal Lab

Page 1 of __

Batch No.

SMO Use

AR/COC

Project Name:	Date Samples Shipped:	SMO Authorization:	☐ Waste Characterization
Project/Task Manager:	Carrier/Waybill No.	SMO Contact Phone:	☐ RMMA
Project/Task Number:	Lab Contact:		☐ Released by COC No.
Service Order:	Lab Destination:	Send Report to SMO:	☐ 4° Celsius
	Contract No.:	Stephanie Montano /505.284.2553	Bill to: Sandia National Laboratories (Accounts Payable), P.O. Box 5800, MS-0154 Albuquerque, NM 87185-0154
Tech Area:			
Building: Room:	Operational Site:		

Sample No.	Fraction	Sample Location Detail	Depth (ft)	Date/Time Collected	Sample Matrix	Container Type	Container Volume	Preserv-ative	Collection Method	Sample Type	Parameter & Method Requested	Lab Sample ID

Last Chain: ☐ Yes	Sample Tracking SMO Use	Special Instructions/QC Requirements:	Conditions on Receipt
Validation Req'd: ☐ Yes	Date Entered:	EDD ☐ Yes ☐ No	
Background: ☐ Yes	Entered by:	Turnaround Time ☐ 7 Day* ☐ 15 Day* ☐ 30 Day	
Confirmatory: ☐ Yes	QC inits.:	Negotiated TAT ☐	
Sample Team Members: Name / Signature / Init. / Company/Organization/Phone/Cell		Sample Disposal ☐ Return to Client ☐ Disposal by Lab	
		Return Samples By:	
		Comments:	Lab Use

1.Relinquished by	Org.	Date	Time	3.Relinquished by	Org.	Date	Time
1. Received by	Org.	Date	Time	3. Received by	Org.	Date	Time
2.Relinquished by	Org.	Date	Time	4.Relinquished by	Org.	Date	Time
2. Received by	Org.	Date	Time	4. Received by	Org.	Date	Time

*Prior confirmation with SMO required for 7 and 15 day TAT

Sample Traceability

Chain of Custody	Legally defensible
	Contains all pertinent sample details
	Signature and date of all personnel who maintained custody of the sample at any time from sampling to the lab
	Timeline should be without gaps
Sample Identification	Must be a unique identifier
	ID must match between the sample bottle and the Chain-of-Custody

Sample Packaging

Considerations		
	Hazardous Materials	Required training
		Safety measures in place
	Hold times	
	Temperature preservation	
	Secure Packaging	
	Container types	Breakable
		Volumes (small volumes may freeze)
	Custody Maintained	

Slide 9

WCR2 Adda picture of a loaded cooler

White, Corey Robert, 6/14/2018

Sample Packaging

Slide 10

WCR2 Adda picture of a loaded cooler

White, Corey Robert, 6/14/2018

Laboratory Receipt Confirmation

- Prompt notification from the lab after samples are received
- Anomalies should be noted and communicated
  - Decisions based on quality objects

GEL Laboratories LLC

SAMPLE RECEIPT & REVIEW FORM

Client: [redacted]	**SDG/AR/COC/Work Order:** [redacted]
Received By: [redacted]	**Date Received:** 06-05-2018 EK
Carrier and Tracking Number	Circle Applicable: FedEx Express FedEx Ground UPS Field Services Courier Other 7346 9017 3412

Suspected Hazard Information	Yes	No	*If Net Counts > 100cpm on samples not marked "radioactive", contact the Radiation Safety Group for further investigation.
Shipped as a DOT Hazardous?		✓	Hazard Class Shipped: UN#:
COC/Samples marked or classified as radioactive?		✓	Maximum Net Counts Observed* (Observed Counts - Area Background Counts): (CPS) / mR/Hr **Classified as: Rad 1 Rad 2 Rad 3**
Is package, COC, and/or Samples marked HAZ?		✓	If yes, select Hazards below, and contact the GEL Safety Group. PCB's Flammable Foreign Soil RCRA Asbestos Beryllium Other:

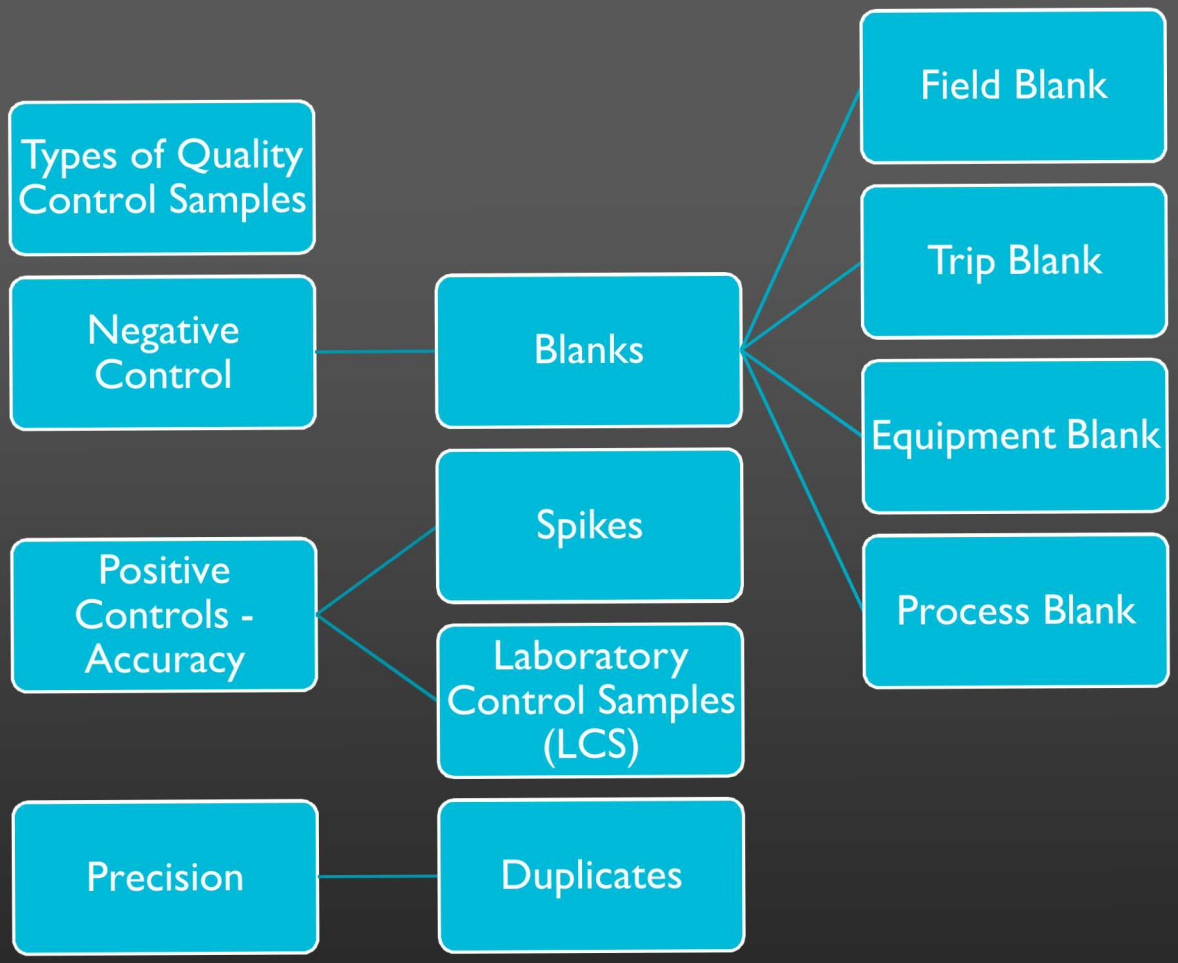
	Sample Receipt Criteria	Yes	NA	No	**Comments/Qualifiers (Required for Non-Conforming Items)**
1	Shipping containers received intact and sealed?	✓			Circle Applicable: Seals broken Damaged container Leaking container Other (describe)
2	Chain of custody documents included with shipment?	✓			
3	Samples requiring cold preservation within (0 ≤ 6 deg. C)?*	✓			Preservation Method: (Wet Ice) (Ice Packs) Dry ice None Other: *all temperatures are recorded in Celsius TEMP: 3c
4	Daily check performed and passed on IR temperature gun?	✓			**Temperature Device Serial #:** IR4-17 Secondary Temperature Device Serial # (If Applicable):
5	Sample containers intact and sealed?	✓			Circle Applicable: Seals broken Damaged container Leaking container Other (describe)
6	Samples requiring chemical preservation at proper pH?	✓			Sample ID's and Containers Affected: If Preservation added, Lot#:
7	Do any samples require Volatile Analysis?	✓			If Yes, Are Encores or Soil Kits present? Yes___ No X (If yes, take to VOA Freezer) Do VOA vials contain acid preservation? Yes X No___ N/A___ (If unknown, select No) VOA vials free of headspace? Yes___ No X N/A___ Sample ID's and containers affected: 605347-001 8260
8	Samples received within holding time?	✓			ID's and tests affected:
9	Sample ID's on COC match ID's on bottles?	✓			Sample ID's and containers affected:
10	Date & time on COC match date & time on bottles?	✓			Sample ID's affected:
11	Number of containers received match number indicated on COC?	✓			Sample ID's affected:
12	Are sample containers identifiable as GEL provided?			✓	
13	COC form is properly signed in relinquished/received sections?	✓			

Comments (Use Continuation Form if needed):

PM (or PMA) review: Initials EMR Date 6/7/18 Page 1 of 1

GL-CHL-SR-001 Rev 5

Quality Control Samples



Negative Controls

Field Blanks

Collected using DI source water during sampling.

Collected directly from the DI water container by simply transferring the water into the appropriate sampling containers with the correct chemical preservatives.

Used to capture contamination from field conditions which could include the source water.

Negative Controls

Trip Blanks

Originate at the commercial laboratory, and travel with the samples.

The trip blank is sealed and not opened until it arrives back at the lab for analysis.

Used for volatile organic analyses only.

Used to pick-up any volatile compound that might contaminate the sample during the entire process.

Negative Controls

Equipment Blanks	Blanks that are collected after decontamination of the equipment.
	The source water is drawn through the sampling equipment in several steps and collected after the final rinse.
	The rinse water is transferred to the appropriate sampling containers with the correct chemical preservatives.
	This type of blank is used to determine the efficacy of the decontamination process.

Negative Controls

Process Blanks	Blanks that are prepared and analyzed side by side with the samples in the laboratory.
	This type of blank is used to determine contamination from laboratory processes.

Blank Contamination

Common Laboratory Contaminants	Substances used at the lab that may contribute to positive results in blanks or samples
	Process blank is the best indicator of this
	Trending over time can help determine where the issue is originating
	Examples include Acetone, Toluene, Hexanes, etc..

Precision Measurement

Duplicate Samples	Field Duplicates	Provides information on the homogeneity of the sample
	Laboratory Duplicates	Provides information on the laboratory techniques
	Matrix-free duplicates	Provides information on the instrument stability

Positive Controls

Sample Spikes	Customer sample that is spiked in the lab with a known amount of analyte prior to processing
	Provides information about the matrix effects of the sample
	Can indicate whether the matrix is biasing the result high or low

Positive Controls

Laboratory Control Samples (LCS)	Matrix free sample prepared at the laboratory
	Known amount of analyte added
	Follows same preparation process as samples
	Indicates high/low bias of the process and/or instrument

Laboratory Oversight

Oversight elements	
	Laboratory Accreditations
	Laboratory Audits
	SNL/SMO Statement of Work (SOW) for Analytical Laboratories
	SMO Contract Verification Review (CVR)
	Data Validation
	Performance Testing (PT) Samples

Laboratory Oversight

Laboratory Accreditations	ISO 17025 – General Requirements for the Competence of Testing and Calibration Laboratories	Applicable to all organizations performing test and/or calibrations, regardless of number of employees	https://www.iso.org/standard/39883.html
	NELAP – National Environmental Laboratory Accreditation Program	TNI standard provides requirements under which laboratories may become accredited through state Accrediting Bodies under 5 different EPA regulatory programs	Clean Air Act (CAA) Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) Clean Water Act (CWA) Resource Conservation and Recovery Act (RCRA) Safe Drinking Water Act (SDWA)
	DOECAP – Department of Energy Consolidated Audit Program	Outlines DOE specific criteria for laboratories on top of ISO 17025 and NELAP accreditations	DoD/DOE Quality Systems Manual

Laboratory Oversight

Performance Testing (PT) Samples	Shows that the lab can get the right answer for a given method/matrix
	Extra layer of confidence that the lab is capable
	Required for many certifications (ISO 17025, NELAP, DOECAP)

Statement of Work

Detailed description of Requirements	
	How work will be communicated to the lab
	Who covers what costs (e.g. shipping, reanalyses for different reasons, etc.)
	Deliverable levels
	EDD requirements
	QA/QC requirements
	Required Detection Limits (RDLs)
	Laboratory certification requirements
	Audit requirements
	Storage requirements
	Excess sample and process waste
	Project specific requirements

Data Package

Level B	Results summary and QC results summary
Level C	Case Narrative, Analysis results, QC results, signed COC, shipping documents
Level D	All of the elements of the Level C, plus analyst worksheets, run logs, raw data, standards prep logs, instrument run logs, digestion and extraction logs

A level D package should have enough information to allow for the result to be recreated by the end user.

*Levels as described in the SNL/SOW.

Data Package

Radiochemistry
Technical Case Narrative
[redacted])
SDG #: [redacted]

Product: **GFPC, Gross A/B, liquid**
Analytical Method: EPA 900.0/SW846 9310
Analytical Procedure: [redacted]-A-001 REV# 20
Analytical Batch: [redacted]330

The following samples were analyzed using the above methods and analytical procedure(s).

[redacted] Sample ID#	Client Sample Identification
[redacted]7001	[redacted]06-007
[redacted]7010	[redacted]71-007
[redacted]7019	[redacted]64-007
[redacted]068	Method Blank (MB)
[redacted]069	[redacted]019[redacted]64-007) Sample Duplicate (DUP)
[redacted]070	[redacted]019[redacted]64-007) Matrix Spike (MS)
[redacted]071	[redacted]019[redacted]64-007) Matrix Spike Duplicate (MSD)[redacted]072

Laboratory Control Sample (LCS)

The samples in this SDG were analyzed on an "as received" basis.

Data Summary:

All sample data provided in this report met the acceptance criteria specified in the analytical methods and procedures for initial calibration, continuing calibration, instrument controls and process controls where applicable, with the following exceptions.

Quality Control (QC) Information

Blank Information
Aliquots for samples [redacted]068 (MB) and [redacted]072 (LCS) were changed to 1.0 per client request.

Technical Information

Gross Alpha/Beta Preparation Information
High hygroscopic salt content in evaporated samples can cause the sample mass to fluctuate due to moisture absorption. To minimize this interference, the salts are converted to oxides by heating the sample under a flame until a dull red color is obtained. The conversion to oxides stabilizes the sample weight and ensures that proper alpha/beta efficiencies are assigned for each sample. Volatile radioisotopes of carbon, hydrogen, technetium, polonium and cesium may be lost during sample heating, especially to a dull red heat. For this sample set, the prepared planchet was counted for beta activity before being flamed. After flaming, the planchet was counted for alpha activity.

Miscellaneous Information

Additional Comments
The matrix spike and matrix spike duplicate, [redacted]070 ([redacted]64-007MS) and [redacted]071 ([redacted]64-007MSD), aliquots were reduced to conserve sample volume.

Certification Statement

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless otherwise noted in the analytical case narrative.

Data Package

Certificate of Analysis

Company : [redacted]
Address : [redacted]
[redacted]
[redacted]
Contact: [redacted]
Project: Level C Data Package, Stormwater/Wastewater

Report Date: June 15, 2018

Client Sample ID: [redacted]06-007
Sample ID: [redacted]7001
Matrix: AQUEOUS
Collect Date: 22-MAY-18
Receive Date: 24-MAY-18
Collector: Client

Project: [redacted]00115
Client ID: [redacted]004
Client Desc.: [redacted]-24

Parameter	Qualifier	Result	Lc	TPU	MDA	Units	PF	DF	Analyst	Date Time	Batch	Mtd.
Rad Gas Flow Proportional Counting												
GFPC, Gross A/B, liquid "As Received"												
Beta		20.7	0.836	+/-1.61	1.73	pCi/L			JXK3	06/07/18 1558	1770330	1
Alpha		12.1	0.608	+/-1.65	1.36	pCi/L			JXK3	06/09/18 1459	1770330	2

The following Analytical Methods were performed

Method	Description
1	EPA 900.0/SW846 9310
2	EPA 900.0/SW846 9310

Surrogate/Tracer Recovery	Test	Batch ID	Recovery%	Acceptable Limits

Notes:

The MDC is a sample specific MDC.
TPU is calculated at the 95% confidence level (1.96-sigma).

Column headers are defined as follows:

DF: Dilution Factor
DL: Detection Limit
Lc/LC: Critical Level
MDA: Minimum Detectable Activity
MDC: Minimum Detectable Concentration
Mtd.: Method
PF: Prep Factor
RL: Reporting Limit
TPU: Total Propagated Uncertainty

Data Package

QC Summary

Report Date: June 15, 2018
Page 1 of 2

Client : [redacted]

Contact: [redacted]

Workorder: [redacted]

Parmname	NOM	Sample	Qual	QC	Units	RER	REC%	Range	Anlst	Date Time
Rad Gas Flow										
Batch [redacted]30										
QC[redacted]069 [redacted]019 DUP										
Alpha		19.1		20.1	pCi/L	0.203		(0-1)	JXK3	06/09/1815:00
	TPU:	+/-2.40		+/-2.45						
Beta		23.6		22.0	pCi/L	0.45		(0-1)		06/07/1815:57
	TPU:	+/-1.80		+/-1.71						
QC[redacted]072 LCS										
Alpha	12.1			11.5	pCi/L		95.2	(80%-120%)	JXK3	06/09/1815:00
	TPU:			+/-1.54						
Beta	46.8			50.3	pCi/L		107	(80%-120%)		06/07/1815:57
	TPU:			+/-2.09						
QC[redacted]068 MB										
Alpha			U	-0.0341	pCi/L				JXK3	06/09/1814:59
	TPU:			+/-0.0946						
Beta			U	-0.0604	pCi/L					06/07/1815:57
	TPU:			+/-0.0899						
QC[redacted]070 [redacted]7019 MS										
Alpha	483	19.1		485	pCi/L		96.6	(75%-125%)	JXK3	06/09/1815:00
	TPU:	+/-2.40		+/-49.6						
Beta	1870	23.6		1990	pCi/L		105	(75%-125%)		06/07/1815:57
	TPU:	+/-1.80		+/-95.3						
QC[redacted]071 [redacted]7019 MSD										
Alpha	483	19.1		569	pCi/L	0.814	114	(0-1)	JXK3	06/09/1815:00
	TPU:	+/-2.40		+/-52.9						
Beta	1870	23.6		2010	pCi/L	0.149	106	(0-1)		06/07/1815:57
	TPU:	+/-1.80		+/-74.6						

Notes:

TPU is calculated at the 95% confidence level (1.96-sigma).

The Qualifiers in this report are defined as follows:

* Recovery or %RPD not within acceptance limits and/or spike amount not compatible with the sample or the duplicate RPD's are not applicable where the concentration falls below the effective PQL.

Data Package

QC Summary

Workorder: [redacted]

Parmname	NOM	Sample	Qual	QC	Units	RER	REC%	Range	Anlst	Date Time

**	Indicates analyte is a surrogate compound.
B	The analyte was found in the blank above the effective MDL.
H	Analytical holding time was exceeded
J	Estimated value, the analyte concentration fell above the effective MDL and below the effective PQL
L	Low Abundance Qualifier
N	Results associated with a spike analysis that was outside control limits.
U	The analyte was analyzed for but not detected below this concentration. For Organic and Inorganic analytes the result is less than the effective MDL. For radiochemical analytes the result is less than the MDA
V	Results are considered a false positive due to interference.
X	Results are considered a false positive due to low abundance.
X	Results are considered a false positive due to peak not meeting identification criteria.
X	Uncertain identification for gamma spectroscopy.
d	The 2:1 depletion requirement was not met for this sample
h	Prep holding time exceeded

N/A indicates that spike recovery limits do not apply when sample concentration exceeds spike conc. by a factor of 4 or more or %RPD not applicable.
** Indicates analyte is a surrogate/tracer compound.
^ The Relative Percent Difference (RPD) obtained from the sample duplicate (DUP) is evaluated against the acceptence criteria when the sample is greater than five times (5X) the contract required detection limit (RL). In cases where either the sample or duplicate value is less than 5X the RL, a control limit of +/- the RL is used to evaluate the DUP result.
For PS, PSD, and SDILT results, the values listed are the measured amounts, not final concentrations.

Where the analytical method has been performed under NELAP certification, the analysis has met all of the requirements of the NELAC standard unless qualified on the QC Summary.

Detection Limits

Method Detection Limit (MDL)	Point at which an analyte can be detected with 99% certainty that it is distinguishable from the blank - 40 CFR Appendix B to part 136
	However, the amount present cannot be determined with confidence
Practical Quantification Limit (PQL), Reporting Limit (RL)	Minimum amount at which an analyte can be quantified accurately - 40 CFR Appendix B to part 136
	Generally the lowest concentration level from the calibration curve, 5-10 X greater than the MDL
	This should be below your action levels

Data quantified between these two levels should be qualified as "estimated", generally "J"

Dilution Factors

Sample 1

Parameter	Qualifier	Result	DL	RL	Units	PF	DF	Analy
Metals Analysis-ICP-MS								
6020/3005 As, Ca, Fe, Mg, Mn, K, Na "As Received"								
Arsenic	U	ND	0.002	0.005	mg/L	1.00	1	PRB
Iron	U	ND	0.033	0.100	mg/L	1.00	1	
Magnesium		19.2	0.010	0.030	mg/L	1.00	1	
Manganese	U	ND	0.001	0.005	mg/L	1.00	1	
Potassium		3.85	0.080	0.300	mg/L	1.00	1	
Calcium		64.2	0.800	2.00	mg/L	1.00	10	PRB
Sodium		63.0	0.800	2.50	mg/L	1.00	10	

The following Prep Methods were performed:

Method	Description	Analyst	Date	Time	Pr

Sample 2

Parameter	Qualifier	Result	DL	RL	Units	PF	DF	Analy
Metals Analysis-ICP-MS								
6020/3005 As, Ca, Fe, Mg, Mn, K, Na "As Received"								
Arsenic		0.0613	0.002	0.005	mg/L	1.00	1	PRB
Iron		1.82	0.033	0.100	mg/L	1.00	1	
Magnesium		21.8	0.010	0.030	mg/L	1.00	1	
Potassium		33.8	0.080	0.300	mg/L	1.00	1	
Calcium		71.1	0.800	2.00	mg/L	1.00	10	PRB
Manganese		7.36	0.010	0.050	mg/L	1.00	10	
Sodium		72.6	0.800	2.50	mg/L	1.00	10	

The following Prep Methods were performed:

Method	Description	Analyst	Date	Time	Pr
SW846 3005A	ICP-MS 3005 PREP	JXM8	06/15/18	1932	17

The following Analytical Methods were performed:

Method	Description	Analyst Comment

Data Verification

Verification Definition:	The process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements.- EPA QA/G-8
SMO Contract Verification Review (CVR)	Identify any elements of the data that don't meet the contract requirements

SNL/SMO Contract Verification

SMO-2012-CVR (11-2013) SMO-05-03

Contract Verification Form (CVR)

Project Leader [redacted] **Project Name** [redacted] **Project/Task No.** [redacted]

ARCOC No. [redacted] **Analytical Lab** [redacted] **SDG No.** [redacted]

In the tables below, mark any information that is missing or incorrect and give an explanation.

1.0 Analysis Request and Chain of Custody Record and Log-In Information

Line No.	Item	Complete? Yes	Complete? No	If no, explain
1.1	All items on ARCOC complete - data entry clerk initialed and dated	X		
1.2	Container type(s) correct for analyses requested	X		
1.3	Sample volume adequate for # and types of analyses requested	X		
1.4	Preservative correct for analyses requested	X		
1.5	Custody records continuous and complete	X		
1.6	Lab sample number(s) provided and SNL sample number(s) cross referenced and correct	X		
1.7	Date samples received	X		
1.8	Condition upon receipt information provided	X		

2.0 Analytical Laboratory Report

1 of 7

SNL/SMO Contract Verification

SMO-2012-CVR (11-2013) SMO-05-03

Line No.	Item	Yes	No	If no, Sample ID No./Fraction(s) and Analysis
3.1	Are reporting units appropriate for the matrix and meet contract specified or project-specific requirements? Inorganics and metals reported as ppm (mg/liter or mg/Kg)? Tritium reported in picocuries per liter with percent moisture for soil samples? Units consistent between QC samples and sample data	X		
3.2	Quantitation limit met for all samples	X		
3.3	Accuracy a) Laboratory control sample accuracy reported and met for all samples	X		Semi-Vol: Caprolactam LCS recovery was 26%, acceptance criteria is 30-61%. Result is acceptable per SOW line 3.5.8 d and f.
	b) Surrogate data reported and met for all organic samples analyzed by a gas chromatography technique	X		
	c) Matrix spike recovery data reported and met	X		
3.4	Precision a) Replicate sample precision reported and met for all inorganic and radiochemistry samples	X		
	b) Matrix spike duplicate RPD data reported and met for all organic samples	X		
3.5	Blank data a) Method or reagent blank data reported and met for all samples	X		
	b) Sampling blank (e.g., field, trip, and equipment) data reported and met	X		

SNL/SMO Contract Verification

Top issues identified during contract verification	Inaccurate or incomplete narratives
	Inaccurate sample ID numbers (ours)
	Positive "hits" in method or continuing calibration blanks
	Surrogate compound recoveries exceeding acceptance limits (low or high)
	Failure to meet minimum detectable activity concentration (MDA/MDC) – radiochemistry
	Lab qualifiers / flags incorrectly assigned
	Missed Holding time for initial and/or reanalysis

Data Validation

Validation Definition:	Analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set - EPA QA/G-8
In House Validation	Validation completed by the end user Pros – fast, inexpensive, user may no where to look for the problems Cons – Possible conflicts of interest
Third Party Data Validation	Validation performed by a 3rd party that has no vested interest in the outcome of the data Pros – Objective result Cons - $$$

Data Validation

Summary

Six samples were prepared and analyzed with accepted procedures using method EPA 8260B (VOCs). All compounds were successfully analyzed. Problems were identified with the data package that resulted in the qualification of data.

1. The initial calibration intercepts were negative with absolute values > the MDL but ≤3X the MDL for methylene chloride, acetone and dichlorodifluoromethane. The associated sample results were non-detect and will be **qualified UJ,I5**.

Data are acceptable and reported QC measures appear to be adequate. The following sections discuss the data review and validation.

Holding Times

The samples were analyzed within the prescribed holding time and were properly preserved.

Instrument Tune

All instrument tune requirements were met.

Calibration

The initial calibration and continuing calibration data met QC acceptance criteria except as noted above in the Summary section and as follows. The initial calibration RSDs were >15% but ≤40% for chloromethane and 2-hexanone. The associated sample results were non-detect and since no other calibration infractions occurred, will not be qualified.

The CCV %Ds were > 20% with positive bias for acetone and dichlorodifluoromethane. The associated sample results were non- detect and will not be qualified.

Blanks

No target analytes were detected in any of the blanks.

Surrogates

Data Validation

Analytical Method	Sample ID	Analyte Name (CAS#)	Qualifier, RC
EPA 350.1			
	[redacted]68-002/[redacted]	Nitrogen, Ammonia (7664-41-7)	J+, B,RP2
	[redacted]70-002/[redacted]	Nitrogen, Ammonia (7664-41-7)	J, I5,B,RP2
	[redacted]72-002/[redacted]	Nitrogen, Ammonia (7664-41-7)	J, RP2
EPA 353.2			
	[redacted]72-007/[redacted]	Nitrogen, Nitrate/Nitrite (NO3ASN)	UJ, MS1,RP1
SW846 3005/6020 DOE-AL			
	[redacted]72-010/T[redacted]	Manganese (7439-96-5)	J, MS1,RP1
SW846 8260B DOE-AL			
	[redacted]68-001[redacted]	Acetone (67-64-1)	UJ, I5
	[redacted]68-001[redacted]	Dichlorodifluoromethane (75-71-8)	UJ, I5
	[redacted]68-001[redacted]	Methylene chloride (75-09-2)	UJ, I5
	[redacted]69-001[redacted]-TB 1	Acetone (67-64-1)	UJ, I5
	[redacted]69-001[redacted]-TB 1	Dichlorodifluoromethane (75-71-8)	UJ, I5
	[redacted]69-001[redacted]-TB 1	Methylene chloride (75-09-2)	UJ, I5

Qualifiers Displayed
J – estimated
J+ - estimated high bias
UJ – not detected, but estimated

Reason Codes Displayed
B – MB contamination at concentration >MDL
RP2 - Replicate RPD failed
I5 - Intercept too large

Data Validation

Levels of Data Validation (Generalizations)	Level I: Contract Verification: Requested analytes reported, RDLs met, holding times met
	Level II: All components of Level I plus samples QC evaluations, receipt information
	Level III: All components of Levels I and II, plus all instrument related QC, calibration records, etc.
	Level IV: Calculation verifications, raw data reviews and spectral interpretations
	*Levels as described by Analytical Quality Associates Inc.

Data Validation

Focused Validation	Based on indicators from historical data
	Identify problem areas and focus validation efforts on a particular area of concern

Data Validation

Common issues identified during data validation	Calibration	Intercept too large
		Slope criteria not met (e.g. R^2 requirement)
	Replicate criteria not met	
	Holding time / Preservation issues	
	Blank contamination	
	Continuing calibration criteria not met	

Communicating Issues to the Lab

Know your Project Manager	Build a relationship through calls, emails, site visits
	Communicate issues early – don't run up against holding times if possible
	Keep detailed documentation (have communications included in data packages)
	Utilize lab web portals – all your data can be found there
	Get to know your methods and requirements

Anomalous Results

Reanalysis	Request that the laboratory reanalyze the sample from a logical starting point
	This may be based on QC results
Resample	Likely not practical in a storm water program
	Can a sample be taken from a fraction collected for a different analysis?
No Action with detailed narration	Depending on how the data are to be used, it may not be necessary to take any action, as long as the details around the anomalous results are captured

Questions