ALLIANT

Data Validation Report
SDG 160-18639-1

Characterization of Structures, Items, Solutions, and Soil at the Proposed Outfall 200 Treatment System Sites Y-12 National Security Complex

Revision 0

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SCOPE

This report contains Level 3 data validation results for analytical data for SDG No. 160-18639-1 for two sludge samples collected at the Y-12 Outfall 200 (Project ORNL Y-12 Outfall 200 Characterization). The evaluation covers analyses for total metals, polychlorinated biphenyls (PCBs), and radionuclides (isotopic neptunium, isotopic americium 241, isotopic plutonium, isotopic uranium, isotopic thorium, radium-226, carbon-14, tritium, total beta strontium, and technetium-99).

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METHOD

The analytical data were validated using the following guidelines:

- Sampling and Analysis Plan / Quality Assurance Project Plan for Geotechnical and Waste Characterization of the Outfall 200 Mercury Treatment Facility Area at the Y-12 National Security Complex, Oak Ridge, Tennessee (November, 2015)
- *Guidance on Environmental Data Verification and Data Validation - EPA QA/G-8*, EP A/240/R-02/004, United States Environmental Protection Agency, Washington D.C
- National Functional Guidelines for Superfund Organic Methods Data Review (August 2014)
- National Functional Guidelines for Inorganic Superfund Data Review (August 2014)
- Verification and Validation of Radiological Data for Use in Waste Management and Environmental Remediation. ANSI/ANS-41.5-2012. (February, 2012)
- Multi-Agency Radiological Laboratory Analytical Protocols Manual (July, 2004)

VERIFICATION AND VALIDATION RESULTS

Completeness

Results for two sludge samples (SDG No. 160-18639-1) were evaluated. Analyses were performed by TestAmerica in Earth City, Missouri (TA-St. Louis). The following lists analytical methods and sample numbers for reported results.

Analysis	Project Sample ID Numbers	Laboratory Sample ID Numbers
Total metals, PCBs and radionuclides	YMTFA81 9404 G	160-18639-1
Total metals, PCBs and radionuclides	YMTFA93 9404-8G	160-18639-2

Holding times

The date of sample collection (08/15/16) and dates of sample analyses were evaluated. Based on these, all recommended holding times per the analytical methods were met.

Preservation and Laboratory Sample Receipt

All samples arrived at TA-St. Louis intact and in good condition under valid chain of custody (COC). The COC was signed indicating the samples were appropriately relinquished by the sampler and accepted by the analytical laboratory. Per the lab case narrative, the temperature of the cooler at receipt was

3.0° C. The laboratory Log-in Sample Receipt Checklist is included in the data package. The COC lists TCLP metals analysis. Per the lab case narrative, the client requested that total metals and mercury (Hg) be logged in place of TCLP metals. The case narrative notes that limited volume was received for sample YMTFA93 9404-8G. No further log-in issues or discrepancies are noted in the narrative and checklist.

Analytical Methods, Reporting Units, and Detection Limits

All analytical methods specified (or equivalent to those specified) on the COC (COC No.160-4416-2171.2) were utilized for the analyses. All results were reported in appropriate units. The detection limits were appropriate for all methods.

Transcription (COC and Lab Data)

There were no transcription errors in sample numbers or other information listed on COCs and in data reports that would impact the results.

Trip Blank

Not Applicable.

Equipment Blanks (EB)

Not applicable.

Field Blank (FB)

Not applicable.

Field Duplicates

A field duplicate sample was not collected for the field samples reported in this SDG.

Laboratory Case Narratives

The following issues were noted in the case narratives:

PCBs:

Analytical Batch 160-266473

- Elevated reporting limits are provided for sample YMTFA93 9404-8G due to limited sample volume available for preparation.
- The CCV recoveries for aroclor 1260 and were outside the lower QC limits on the secondary column, but within acceptable QC limits on the primary column. The sample detects reported for aroclor 1260 are confirmed on the second column within acceptance criteria, and the sample surrogate recoveries were within acceptable QC limits on the primary and secondary column. Therefore, sample detect qualifications are not required.
- The IS eluted outside the retention time window on the column for CCV 160-266473/3. The retention time shift was taken into account when reviewing the samples for target compounds.

Total Metals (ICP) and Mercury:

- Samples YMTFA81 9404 G and YMTFA93 9404-8G were diluted due to high salts in the sample matrix. Elevated reporting limits (RLs) are provided.

- The case narrative lists matrix spike/matrix spike duplicate (MS/MSD) recoveries and RPDs outside control limits for barium and lead. Review of the MS/MSD reporting forms (Form 5A) found that the lab had incorrectly calculated % recoveries. The MS/MSD results are within acceptance criteria, and no sample qualification is required due to MS/MSD results.
- Mercury: Sample YMTFA93 9404-8G was diluted to bring the sample mercury concentration within the instrument calibration range. The sample mercury RL has been raised accordingly. No sample qualification is required.

Radionuclides:

- For isotopic americium 241, isotopic neptunium, isotopic plutonium, isotopic thorium, isotopic uranium and radium-226 (alpha spectrometry analyses), the case narrative notes that samples YMTFA81 9404 G and YMTFA93 9404-8G could not be thoroughly homogenized before sub-sampling due to sample matrix. The samples contained rocks.
- The carbon-14 MS/MSD analyses failed recovery and precision criteria. The MS/MSD batch QC samples were performed on a sample not in this SDG, and data qualification is not required.
- No further analytical or quality issues were noted in the laboratory SDG narrative.

Verification/Validation Checklists, Data Qualifiers, and Qualifier Definitions

Verification and validation checklists are presented in Appendix A and Appendix B. Applicable validation qualifier codes are defined in the table below.

Qualifier	Definition
U	analyte is not detected at or above the stated reporting limit
UJ	analyte is not detected but there is uncertainty about the reporting limits.
J	result is estimated
R	result is rejected

TCLP Extractions

Per the lab case narrative, the TCLP metals analysis requested on the COC was changed to total metals and Hg due to client request. TCLP extractions were therefore not performed.

Total Metals (ICP) and Mercury

Two sludge samples were prepared and analyzed for ICP metals by SW-846 Method 6010C and mercury by SW-846 Method7470A. Holding times, initial and continual calibrations, batch QC (blank, LCS, MS/MSD) were acceptable. No qualification of metals data was required.

Polychlorinated Biphenyl by GC

Two sludge samples were extracted and analyzed for PCBs by SW-846 Method 8082A. Holding times, initial and continual calibrations, batch QCs (blank, LCS, MS/MSD) and sample specific QCs (internal standards, surrogates) were acceptable. No qualification of data was required.

Radionuclides

Two sludge samples were analyzed for the following radionuclides: isotopic neptunium, isotopic americium 241, isotopic plutonium, isotopic uranium, isotopic thorium, tritium, total beta strontium, radium-226, carbon-14, and technetium-99. Holding times, applicable instrument calibrations, and sample and batch QCs were acceptable for all methods excepted as listed below. Traceable standard certificates were acceptable.

Alpha Spectroscopy

Radium-226, isotopic americium (Am-241), isotopic neptunium (Np-237 and Np-239), isotopic plutonium (Pu-238 and Pu-239/240), isotopic thorium (Th-228, Th-230, Th-232) and isotopic uranium (U-233/234, U-235/236, U-238) analyses were performed by Alpha Spectroscopy. The Laboratory Control Sample (LCS) and matrix spike (MS) had acceptable percent recoveries. The laboratory duplicate analyses had acceptable relative percent difference (RPD) and duplicate error ratio (DER) results. Chemical recoveries and yields were within acceptable limits with the following exception: Thorium-229 tracer recovery was below the QC limit in sample UMTFA73SE001. All thorium isotopes were qualified as estimated (J) in this sample. Method blank results were less than the MDAs, with the following exceptions: Thorium-230 was detected in the method blank at 0.1419 pCi/g. Radium-226 was detected in the method blank at 0.4325 pCi/g. Thorium-230 and radium-226 detects in the samples at less than 10x the method blank result are qualified as estimated, J. No other qualification of data was required.

Gas Flow Proportional Counter

Total beta strontium analysis was performed by gas flow proportional counter. The Laboratory Control Sample (LCS) had acceptable percent recoveries. The laboratory batch QC duplicate analysis had acceptable relative percent difference (RPD) and duplicate error ratio (DER) results. Chemical recoveries and yields were within acceptable limits. Method blank results were less than the MDAs. No qualification of data was required.

Liquid Scintillation Counter

Tritium, technetium (Tc-99) and carbon-14 were analyzed by liquid Scintillation counter. The Laboratory Control Sample (LCS) and matrix spike (MS) had acceptable percent recoveries except for the following: MSD recovery for carbon-14 was below the QC limit and the MS/MSD RPD was outside acceptance criteria. The carbon-14 MS/MSD analyses were batch QC performed on a sample not in this SDG. Per the case narrative, non-homogeneity and matrix interference is suspected. Since the batch QC was performed on another client's sample, the sample carbon-14 sample results in this SDG are not qualified for the MS/MSD recovery and precision. The laboratory duplicate analyses had acceptable relative percent difference (RPD) and duplicate error ratio (DER) results. Chemical recoveries and yields were within acceptable limits. Method blank results were less than the MDAs.

Summary

- Thorium-230 and radium-226 detects in the samples at less than 10x the method blank result are qualified as estimated, J.

Summary of Result Qualifiers

Sample No.	Parameter	Laboratory Result	Qualified Result	Units	Laboratory Qualifier	Validation Qualifier
YMTFA81 9404 G	Thorium-230	1.17	1.17 J	pCi/g	None	J
YMTFA93 9404-8G	Thorium-230	0.313	0.313 J	pCi/g	None	J
YMTFA81 9404 G	Radium-226	1.68	1.68 J	pCi/g	None	J
YMTFA93 9404-8G	Radium-226	1.83	1.83 J	pCi/g	None	J

Appendix A

Verification Summary Table

Data Verification SDG 160-18639-1	Y	N	N/A	Comment
Custody of Samples				
Are samples traceable through inspection of signature records on field and laboratory chains of custody (COCs)?	x			
Has contractual turn-around time been met for all samples?			x	
Have all samples been preserved correctly and pertinent documentation included?	x			
Is the laboratory log in sample receipt checklist present	x			
Are any sample receipt non-conformances noted?	x			Limited sample volume was received for sample YMTFA93 9404-8G
Standard Traceability				
Have certificate(s) been included for the LCS and MS?	x			
Standards have not exceeded the certificate expiration date	x			
Are chemical standards and reference materials traceable to a reliable source? (Reagent traceability summary)	x			
Analytical Completeness				
Are all COC samples and associated analytical results reported in the laboratory data package?	x			
Data Summaries				
The case narrative is present and summarizes the sample receipt and analysis information including any analytical anomalies for all methods reported in the data package.	x			
Other data summary forms are present as applicable (detection, sample results, surrogate, tracer/carrier, QC results and association, prep and analysis chronicle, method and sample summaries)	x			
Sample Data				
Is the Sample Data included for each COC requested analytical method?	x			
Is the calibration data included for each method? (ICAL, ICV, CCAL as required for each method)	x			
Are the QC summary forms included for each method? (MB, ICS/CCB, LCS/LCSD, MS/MSD, surrogates,	x			

Data Verification SDG 160-18639-1	Y	N	N/A	Comment
internal standards, serial dilution as required and applicable for each method)				
Are the method run logs and/or bench sheets included for each method?	x			
Are the method preparation/extraction logs included for each applicable method?	x			
Is the sample and QC raw data included for each method?	x			
Is the internal Laboratory Review documented by checklists and included in the data package?		x		Lab internal review checklists for the sample analyses are not included for the TA-St. Louis lab.

Appendix B

Validation Summary Tables

Polychlorinated Biphenyl (Method 8082A)	Y	N	N/A	Qualifier	Comment or Reason Code
Preservation and Holding Times					
Were samples properly preserved?	x				
Have the samples been analyzed within holding times?	x				
Detection Limits and Preservation					
Are all laboratory RLs <= recommended RLs in the SAP?	x				Elevated reporting limits are provided for sample YMTFA93 9404-8G due to limited sample volume available for preparation.
Initial Calibration					
Are minimum calibration curve with minimum 5 points analyzed prior to sample analysis?	x				
Are %RSDs within method criteria?	x				
Calibration Verification					
Are calibration verification standard analyzed at the appropriate frequency?	x				
RT within RT windows established by initial calibration?		x			The IS eluted outside the retention time window on the column for CCV 160-266473/3. The retention time shift was taken into account when reviewing the samples for target compounds.

Polychlorinated Biphenyl (Method 8082A)	Y	N	N/A	Qualifier	Comment or Reason Code
Are %D (difference or drift) within 20% of the average initial calibration factors?		x			The CCV recoveries for aroclor 1260 and were outside the lower QC limits on the secondary column, but within acceptable QC limits on the primary column.
Method Blank					
Is the Method Blank extracted and analyzed for each analytical batch of up to 20 samples?	x				
Is the Method Blank Summary form present?	x				
Is the method blank the same matrix as the samples in the reporting batch?	x				
Is the blank at similar (low, medium, or trace) concentration level?	x				
Does the blank have any detects above MDL?		x			
Surrogate Recovery					
Are all samples and QCs spiked with surrogate compounds?	x				
Are percent recoveries within the method criteria results?	x				
LCS/LCSD					
Has at least one LCS been prepared for each preparation batch containing up to 20 samples?	x				
Is the LCS the same matrix as the samples in the reporting batch?	x				
Is the LCS spiked with all target analytes listed in the SAP?			x		The lab QC samples ae spiked with aroclor 1016 and 1260
Are the LCS %RECs within the applicable QC criteria?	x				
Are the LCS/LCSD RPDs within the applicable QC criteria?			x		

Polychlorinated Biphenyl (Method 8082A)	Y	N	N/A	Qualifier	Comment or Reason Code
Matrix Spike/Matrix Spike Duplicate					
Has at least one MS/MSD pair been prepared for a batch with sample counts up to 20 samples?	x				
Are the MS/MSD spiked with target analyte specified in the SAP?			x		The lab QC samples are spiked with aroclor 1016 and 1260
MS and MSD %RECs within the applicable QC limits?	x				
MS/MSD RPDs within the applicable QC limits?	x				
Internal Standards					
Were internal standards added to all samples and QC samples?	x				
Are internal standard retention times within method criteria?		x		None	
Are internal standard areas within method criteria?		x			
Target Analyte Identification					
Do the positively identified compounds meet the identification criteria?	x				Reported PCB-1254 and PCB-1260 detects in the samples were confirmed on the second column.
Are the RTs of the positively identified target analytes within RT windows established by initial calibration standards?	x				
Target Analyte Quantitation and Reported Quantitation Limit					
Are the results for all positively identified analytes calculated correctly?	x				
Are the reporting limits calculated for the non-detects and reported correctly	x				

TCLP Metals by ICP (SW6010) **TCLP Mercury by CVAA (SW7470A)**	Y	N	N/A	Qualifier	Comment or Reason Code
Preservation and Holding Times					
Were samples properly preserved?	x				
Are sample preparation sheets present and account for all extractions and digestions for reported samples?	x				Total metals are analyzed and reported. The COC lists TCLP metals analysis, and the case narrative notes that the client requested total metals to be performed instead of TCLP.
Have the samples been prepared and analyzed within holding times?	x				
Detection Limits and Target Analytes					
Do all samples show RLs <= the SAP Recommended Reporting Limits?	x				
Are all the SAP target analytes reported?	x				
Initial Calibration					
Was the Calibration within acceptance criteria?	x				
Calibration Verification					
Was a second source ICV analyzed after calibration with recoveries within acceptance criteria?	x				
Were CCVs analyzed at the required frequency with recoveries within acceptance criteria? For ICP, CCVs and low level CCVs (CCVL) as applicable.	x				
Are the ICV and CCV/CCVL Summary forms present?	x				
Was the ICP CRQL Check Standard analyzed with recoveries within acceptance criteria?	x				
Method Blank and ICB/CCBs					
Has at least one method blank been prepared For each batch of up to 20 samples?	x				
Is the method blank the same matrix as the samples in the reporting batch?	x				
Were target analytes detected in the method blank above the MDL?		x			
Were the ICB and CCBs analyzed at the required frequency with results within acceptance criteria?	x				
Are the Method Blank and ICB/CCB Summary forms present?	x				

TCLP Metals by ICP (SW6010) **TCLP Mercury by CVAA (SW7470A)**	Y	N	N/A	Qualifier	Comment or Reason Code
ICP Interference Check Samples					
Were the ICP ICSA/ICSAB interference check standards analyzed as required with results within acceptance criteria?	x				
LCS/LCSD					
Has at least one LCS been prepared for each preparation batch containing up to 20 samples?	x				
Is the LCS the same matrix as the samples in the reporting batch?	x				
Is the LCS spiked with all target analytes listed in the SAP?	x				
Are the LCS %RECs within the applicable QC criteria?	x				
Are the LCS/LCSD RPDs within the applicable QC criteria?			x		
Matrix Spike/Matrix Spike Duplicate					
Has at least one MS/MSD pair been prepared for a batch containing up to 20 samples?	x				
Are the MS/MSD spiked with all target analytes listed in the SAP?	x				
Are MS and MSD %RECs within the applicable QC limits?	x				The case narrative lists matrix spike/matrix spike duplicate (MS/MSD) recoveries and RPDs outside control limits for barium and lead. Review of the MS/MSD reporting forms (Form 5A) found that the lab had incorrectly calculated % recoveries. The MS/MSD results are within acceptance criteria, and no sample qualification is required due to MS/MSD results
Are MS/MSD RPDs within the applicable QC limits?	x				
Duplicates					

TCLP Metals by ICP (SW6010) **TCLP Mercury by CVAA (SW7470A)**	Y	N	N/A	Qualifier	Comment or Reason Code
Has a laboratory duplicate been prepared for a batch containing up to 20 samples? (If an MS/MSD pair has been prepared, the laboratory duplicate is not required.)			x		
If a laboratory duplicate was analyzed, were the RPDs within acceptance criteria?			x		
Was a field duplicate collected?		x			
If a field duplicate was analyzed, were the RPDs within the 50% acceptance criteria?			x		
Serial Dilution					
Was the Serial Dilution within acceptance limits?	x				
Sample Quantitation and Documentation					
Are reported sample concentrations within the instrument linear range?	x				
Have sample reporting limits and reported concentrations been adjusted for analytical dilutions?	x				5x analytical dilutions were performed due to high salts in the sample matrix.
Are instrument runlogs present and account for all reported sample results?	x				
Have all Laboratory Case Narrative comments and findings been addressed in the data validation process?	x				

Radiological Data Validation **Alpha Spectrometry** **Gas Flow Proportional Counter** **Liquid Scintillation Counting**	Y	N	N/A	Qualifier	Comment or Reason Code
Sample Handling and Preservation					
Were samples preserved correctly?	x				
Holding Times					
Were samples analyzed within holding times?	x				
Standard Traceability					
Were all certificates included for the LCS and MS samples?	x				
Were all standards and reference materials traceable to reliable source material?	x				
Calibration Verification					
Are efficiencies within tolerance limits?	x				
Are energies within tolerance limits?	x				
Are background performance check count rates within tolerance limits?	x				
Are appropriate peak resolution within appropriate control criteria?	x				
LCS					
Has at least one LCS been prepared for up to 20 samples?	x				
Is the LCS the same matrix as the samples in the reporting batch?	x				
Are LCS %D (or %R) within QC acceptance limits?	x				
Laboratory Duplicate					
Has at least one laboratory duplicate been prepared for up to 20 samples?	x				
ARE RPD and DER within QC acceptance limits?	x				
Matrix Spike					
Has at least one MS been prepared for up to 20 samples?	x				
Is MS %D (or %R) within QC acceptance limits?	x				
Method Blank					
Has at least one method blank been prepared for up to 20 samples?	x				
Is the method blank the same matrix as the samples in the reporting batch?	x				

Radiological Data Validation **Alpha Spectrometry** **Gas Flow Proportional Counter** **Liquid Scintillation Counting**	Y	N	N/A	Qualifier	Comment or Reason Code
Are the results less than 1.65 * CSU or within control limits?		x		J	The radium-226 and thorium-230 method blanks were detected above the MDC/MDL. The associated sample results detected at less than 10x the method blank detects are therefore qualified as estimated, J. No further sample qualifications were required due to the method blanks.
Chemical Yield - Tracers and Carriers					
Is yield reported for all samples and QC samples in the reporting batch?	x				
Are percent recovery criteria satisfied for all yield results?	x				

Analytical Data Review
Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	Brandy Gilliam	Date Verified:	10/14/2016
SDG No(s).	18639-1		

		Acceptable?				Comments
Item No.	Criteria	Yes	No	NA	NR	
1.	Case Narrative Present	X				
2.	Lab Qualifiers Present	X				
3.	Methods Specified in SAP or Equivalent Methods were Used	X				
4.	Data is Complete for All Requested Analytes with All Samples	X				Insufficient volumes for analyses requested; no tritium analysis. Total metals analyzed instead of TCLP metals.
5.	Units are as Specified in SOW/Contract or Otherwise are Appropriate	X				
6.	Detection Limits Meet Contract Required Detection Limits or Other Project Defined Limits (e.g., regulatory limits)	X				
7,	Samples IDs and Analytes Agree with those on COCs	X				
8.	Samples IDs Agree Throughout Report	X				
9.	Raw Data Results Agree with Data Reports and Electronic Data	X				
10.	COCs – Samples Traceable	X				
11.	All Samples Preserved Correctly	X				
12.	Samples Arrived Intact	X				
13.	Custody Seals on Samples			X		COC seals on coolers only
14.	Holding Times Met	X				
	-Metals other than Mercury ≤ 180 days	X				
	-Mercury ≤28 days	X				
	-TCLP Metals other than Mercury to TCLP Extraction ≤180 days			X		
	-TCLP Metals other than Mercury TCLP Extraction to Analysis ≤180 days			X		
	-TCLP Mercury to TCLP Extraction ≤28 days			X		
	-TCLP Mercury TCLP Extraction to Analysis ≤28 days			X		

Analytical Data Review
Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	Brandy Gilliam	Date Verified:	10/14/2016
SDG No(s).	18639-1		

		Acceptable?				Comments
Item No.	Criteria	Yes	No	NA	NR	
	-VOAs to Extraction/Analysis ≤14 days			X		
	-SVOAs to Extraction ≤7 days (liquids), ≤14 days (solids)			X		
	-SVOAs Extraction to Analysis ≤40 days			X		
	-Pesticides to Extraction ≤7 days (liquids), ≤14 days (solids)			X		
	-Pesticides Extraction to Analysis ≤40 days			X		
	-Herbicides to Extraction ≤7 days (liquids), ≤14 days (solids)			X		
	-Herbicides Extraction to Analysis ≤40 days			X		
	PCBs - none	X				
	-TCLP VOAs to TCLP Extraction ≤14 days			X		
	-TCLP VOAs TCLP Extraction to Analysis ≤14 days			X		
	-TCLP SVOAs to TCLP Extraction ≤14 days			X		
	-TCLP SVOAs TCLP Extraction to Prep Extraction ≤7 days			X		
	-TCLP SVOAs Prep Extraction to Analysis ≤40 days			X		
	-TCLP Pesticides to TCLP Extraction ≤14 days			X		
	-TCLP Pesticides TCLP Extraction to Prep Extraction ≤7 days			X		
	-TCLP Pesticides Prep Extraction to Analysis ≤40 days			X		
	-TCLP Herbicides to TCLP Extraction ≤14 days			X		
	-TCLP Herbicides TCLP Extraction to Prep Extraction ≤7 days			X		
	-TCLP Herbicides Prep Extraction to Analysis ≤40 days			X		

Analytical Data Review
Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	Brandy Gilliam	Date Verified:	10/14/2016
SDG No(s).	18639-1		

		Acceptable?				Comments
Item No.	Criteria	Yes	No	NA	NR	
	TOC ≤28 days			X		
	-Hexane Extractable Material, Oil and Grease ≤28 days			X		
	-Chloride, Fluoride, Nitrate, Sulfate ≤28 days			X		
	-Cyanide ≤14 days			X		
	-Sulfide ≤7 days			X		
	-pH – immediately			X		
	-Specific Conductance - immediately			X		
	-Radionuclides 180 days (best practice)	X				