



**Data Validation Report
SDG 160-18633-2**

**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. 160-18633-2 for three soil samples collected at the Y-12 Outfall 200 (Project ORNL Y-12 Outfall 200 Characterization). The evaluation covers analyses for radionuclides (radium-226, isotopic neptunium-237, isotopic americium-241, isotopic plutonium, isotopic uranium, isotopic thorium, and carbon-14).

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 three soil samples (SDG No. 160-18633-2) were evaluated. The radium-226 analyses were performed by TestAmerica in Earth City, Missouri (TA-St. Louis). The remaining radionuclides analyses (isotopic neptunium-237, isotopic americium-241, isotopic plutonium, isotopic uranium, isotopic thorium, and carbon-14) were subcontracted and performed by TestAmerica in Richland, Washington (TA-RL), Washington. The following lists analytical methods and sample numbers for reported results.

Analysis	Project Sample ID Numbers	Laboratory Sample ID Numbers
Radionuclides	YMTFA54 SO 010 (Sub J6H240419-1)	160-18633-1
Radionuclides	YMTFA54 SO 010D (Sub J6H240419-2)	160-18633-2
Radionuclides	YMTFA58 SO 010 (Sub J6H240419-3)	160-18633-3

Holding times

The date of sample collection (08/12/16) 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 and TA-RL intact and in good condition under valid chain of custody (COC). The COC was signed indicating the samples were appropriately relinquished by the field personnel and accepted by the analytical laboratory.

All samples were received at TA-St. Louis outside temperature criteria at 12°C. Per the laboratory SDG narrative, the client was contacted regarding the temperature exceedance and the lab was instructed to proceed with analysis. The radiochemistry sample results do not require qualification for temperature at receipt. Cooler receipt temperature is not noted for TA-RL.

Sample receipt checklists are included for both facilities and no further preservation or sample receipt issues are noted.

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

One field duplicate sample was collected and reported. The field duplicate pair is: Sample YMTFA54 SO 010 and field duplicate YMTFA54 SO 010D. Field duplicate analytical results met QC acceptance criteria.

Laboratory Case Narratives

The following issues were noted in the case narratives:

Radionuclides:

- Radium-226 by Alpha Spectrometry:
Samples YMTFA54 SO 010, YMTFA54 SO 010D and YMTFA58 SO 010 were analyzed for Radium-226 by Alpha Spectrometry in accordance with ST-RC-0301. The samples were dried

on 8/16/2016, prepared on 9/15/2016 and analyzed on 9/24/2016. The three samples in the SDG could not be thoroughly homogenized before sub-sampling was performed due to sample matrix. The samples contained small rocks and were of varying colors.

- No further analytical or quality issues were noted in the laboratory SDG narratives.

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

Radionuclides

Three samples were analyzed for the following radionuclides: radium-226, isotopic neptunium-237, isotopic americium-241, isotopic plutonium, isotopic uranium, isotopic thorium, and carbon-14. Holding times, applicable instrument calibrations, and sample and batch QCs were acceptable for all methods, with the exceptions listed below. Traceable standard certificates were acceptable.

Alpha Spectroscopy

Radium-226, isotopic americium (Am-241), isotopic neptunium (Np-237), 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, and U-238) analyses were performed by Alpha Spectroscopy. The Laboratory Control Sample (LCS) 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. Method blank results were less than the MDAs with the following exceptions: Rd-226 was detected in the method blank at 0.4325 pCi/g. The Rd-226 detections in the samples at less than 10x the method blank contamination are qualified as estimated, J. Thorium-230 was detected in the method blank at 0.0537 pCi/g. The thorium-230 detections in the samples at less than 10x the method blank contamination are qualified as estimated, J. No other qualification of data was required.

Liquid Scintillation Counter

Carbon-14 was analyzed by liquid Scintillation counter. The Laboratory Control Sample (LCS) 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. Method blank results were less than the MDA.

Summary

- Rd-226 and thorium-230 were detected in the associated method blank. Therefore, Rd-226 and thorium-230 detects for the samples are qualified as estimated, J.

Summary of Result Qualifiers

Sample No.	Parameter	Laboratory Result	Qualified Result	Units	Laboratory Qualifier	Validation Qualifier
YMTFA54 SO 010	Rd-226	1.74	1.74 J	pCi/g	None	J
YMTFA54 SO 010D	Rd-226	1.64	1.64 J	pCi/g	None	J
YMTFA58 SO 010	Rd-226	0.972	0.972 J	pCi/g	None	J
YMTFA54 SO 010	Thorium-230	0.334	0.334 J	pCi/g	None	J
YMTFA54 SO 010D	Thorium-230	0.536	0.536 J	pCi/g	None	J
YMTFA58 SO 010	Thorium-230	0.179	0.179 J	pCi/g	None	J

Appendix A
Verification Summary Table

Data Verification SDG 160-18633-2	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			All samples were received at TA-St. Louis outside temperature criteria at 12°C. Per the laboratory SDG narrative, the client was contacted regarding the temperature exceedance and the lab was instructed to proceed with analysis. radiochemistry sample results do not require qualification for temperature at receipt.
Is the laboratory log in sample receipt checklist present	x			TA-St. Louis and TA-RL sample receipt checklists are included
Are any sample receipt non-conformances noted?		x		
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 Verification SDG 160-18633-2	Y	N	N/A	Comment
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, internal standards, serial dilution as required and applicable for each method)	x			
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 TA-RL are included.

Appendix B
Validation Summary Tables

Radiological Data Validation Alpha Spectrometry 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				The lab duplicate batch QC was performed on a sample not in this SDG.
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 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			The radium-226 and thorium-230 method blanks were detected above the MDC/MDL. The associated sample results are detected at less than 10x the method blank detects and 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:	JD Milloway	Date Verified:	9/8/16
SDG No(s).	18633-1; 18633-2		

Item No.	Criteria	Acceptable?				Comments
		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				
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			Cooler received at 12 deg C. FedEx failed to deliver on Saturday as listed on Air Bill
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				
	-VOAs to Extraction/Analysis ≤14 days			X		

Analytical Data Review Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	JD Milloway	Date Verified:	9/8/16
SDG No(s).	18633-1; 18633-2		

Item No.	Criteria	Acceptable?				Comments
		Yes	No	NA	NR	
	-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		
	TOC ≤28 days			X		

Analytical Data Review Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	JD Milloway	Date Verified:	9/8/16
SDG No(s).	18633-1; 18633-2		

Item No.	Criteria	Acceptable?				Comments
		Yes	No	NA	NR	
	-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				