



**Data Validation Report
SDG 160-18591-2**

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

Revision 1

ALLIANT CORPORATION
320 N CEDAR BLUFF ROAD
KNOXVILLE, TN 37923

Validated by: *Dianne McNeill* Date: 10/14/2016
Dianne McNeill

SCOPE

This report contains Level 3 data validation results for analytical data for Sample Delivery Group (SDG) 160-18591-2 for nine composite soil samples collected at the Proposed Outfall 200 Mercury Treatment Facility located at the Y-12 National Security Complex, Oak Ridge, Tennessee. The evaluation covers analyses for the following radionuclide analyses: Americium 241, Neptunium-237, isotopic Plutonium, isotopic Thorium, isotopic Uranium, Carbon-14, and Radium-226 (Ra-226).

METHOD

The analytical data were validated using applicable portions of the following guidelines:

- *Characterization of Structures, Items, Solutions, and Soils at the Proposed Outfall 200 Treatment Systems Site Work Plan* (AC-4326-002-WP, July 2016)
- *Sampling and Analysis Plan/Quality Assurance Project Plan for Geotechnical and Waste Characterization of the Outfall 200 Mercury Treatment Facility Area at the National Security Complex, Oak Ridge, Tennessee* (DOE/OR-01-2657&D1, November 2015) (SAP/QAPP).
- *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
- *es/er/ms-5, Evaluation of Radiochemical Data Usability*, Oak Ridge National Laboratory, U.S. Department of Energy (April, 1997)
- *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 nine composite soil samples were evaluated. The Ra-226 analyses were performed by TestAmerica in Earth City, Missouri (TA-St. Louis). The analyses for Americium-241, Neptunium-237, isotopic Plutonium, isotopic Thorium, isotopic Uranium, and Carbon-14 were subcontracted to and performed by TestAmerica in Richland, Washington (TA-RL), Washington. The subcontract SDG is 51964. Total Characteristic Leaching Procure (TCLP) metals and polychlorinated biphenyls (PCBs) were also listed on the Chain of Custody (COC) for these nine samples; however, the TCLP metals and PCBs were reported in a separate SDG. There no effect on completeness for this evaluation. The table below lists analytical methods and sample numbers for reported results evaluated in this Data Validation Report (DVR). Subcontract work order (ID) numbers are shown in parenthesis.

Project Sample ID	Laboratory Sample ID	Analysis
YMTFA 44 SO 005	160-18591-1 (M84W9)	Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA 33 SO 002	160-18591-2 (M84XA)	Ra-226 Americium-241

Project Sample ID	Laboratory Sample ID	Analysis
		Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA 45 SO 005	160-18591-3 (M84XC)	Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA 46 SO 005	160-18591-4 (M84XD)	Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA 50 SO 005	160-18591-5 (M84XE)	Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA 47 SO 005	160-18591-6 (M84XF)	Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA 48 SO 005	160-18591-7 (M84XG)	Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA 63 SO 010	160-18591-8 (M84XJ)	Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14

Project Sample ID	Laboratory Sample ID	Analysis
YMTFA 64 SO 010	160-18591-9 (M84XL)	Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14

Holding times

Based on evaluation of the date of sample collection (08/10/16) and date of sample analyses, 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 COC. The COC was signed indicating the samples were appropriately relinquished by the field personnel and accepted by the analytical laboratory. Sample temperature at receipt was acceptable at 1.6 °C.

Analytical Methods, Reporting Units, and Detection Limits

All analytical methods specified (or equivalent to those specified) on the COC (COC No. 160-4422-2174.4) were utilized for the analyses, except TCLP-metals and PCBs which were reported in a separate SDG as noted above. All results were reported in appropriate units. Detection limits were appropriate for all methods.

Trip Blank

Not Applicable.

Equipment Blanks (EB)

Not applicable.

Field Blank (FB)

Not applicable.

Field Duplicates

Not applicable.

Laboratory Case Narratives

The following issues were noted in the case narratives:

Radionuclides

Ra-226

- The samples could not be thoroughly homogenized before sub-sampling was performed due to sample matrix. The samples were of varying colors and contained rocks.
- The sample duplicate precision (RER/RPD) was outside of the control limits: (relative error ratio [RER]: 3.83, relative percent difference [RPD]: 115%). (Validator note: the duplicate analysis was performed on a sample not evaluated for and not representative of the samples discussed in this DVR).

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
J	Result is estimated
U	Analyte is not detected at or above the stated reporting limit
R	Result is rejected
UJ	Analyte is not detected but there is uncertainty about the reporting limit

Radionuclides

Nine composite soil samples were analyzed for the following radionuclides (Environmental Measurements Laboratory [EML]/HASL method/methodology in parenthesis):

- Ra-226 (ST-RC-0301/Alpha Spectrometry),
- Americium-241 (RL-ALP-003/Alpha Spectroscopy),
- Neptunium-237 (RL-ALP-013/Alpha Spectroscopy),
- Isotopic Plutonium (RL-ALP-002/Alpha Spectroscopy),
- Isotopic Thorium (RL-ALP-001/Alpha Spectroscopy),
- Isotopic Uranium (RL-ALP-015/Alpha Spectroscopy), and
- Carbon-14 (RL-LSC-008/LSC).

Holding times, applicable instrument calibrations, and sample and batch QCs (blanks, LCSs, duplicates) were acceptable for all methods, except as noted below. Traceable standard certificates were acceptable. Tracer and chemical recoveries and yields were acceptable.

Alpha Spectrometry

Ra-226 was detected in the method blank at 0.2498 pCi/g, and the normalized difference was calculated by the validator for all samples, using the equation:

$$|S - B|/\sqrt{([TPU]_S)^2 + ([TPU]_B)^2}$$

Where

S = Sample result

B = Method blank result

TPU = Total Propagated Uncertainty

If the normalized absolute difference is > 2.58 no qualification is assigned, as at the 1% level of significance, the conclusion is reached that the method blank and sample differ significantly. If the normalized absolute difference is between 1.96 and 2.58, samples are qualified as estimated (J) and the sample and method blank differ at the 5% level of significance (sample results < MDC do not require qualification). If the normalized absolute difference is between 0 and 1.96, deficiencies in other quality-indicator samples are considered prior to qualifying the samples.

The normalized absolute difference was > 2.58 for all but the following samples: YMTFA 33 SO 002 and YMTFA 64 SO 010. The normalized absolute difference was also < 1.96 for samples YMTFA 33 SO 002 and YMTFA 64 SO 010, but there were no other quality issues affecting these samples, so results for these samples were qualified as estimated (J).

As noted previously, the Ra-226 sample duplicate precision (RER/RPD) was outside of the control limits, with an RER of 3.83 and RPD of 115%. This batch duplicate was performed using a project sample evaluated in a different DVR. The matrix for the duplicate sample was concrete and is not considered to be representative of the soil samples in this SDG.

No other quality issues were identified for any of the analyses.

Summary

- Ra-226 was detected in the method blank at 0.2498 pCi/g and the normalized difference was < 2.58 for samples YMTFA 33 SO 002 and YMTFA 64 SO 010. Therefore, the Ra-226 results were qualified as estimated (J) for these two samples. There were no other qualifications assigned to any samples evaluated for this DVR.

Summary of Result Qualifiers

Sample No.	Parameter	Laboratory Result	Qualified Result	Units	Laboratory Qualifier	Validation Qualifier
YMTFA 33 SO 002	Ra-226	1.15	1.15	pCi/g		J
YMTFA 64 SO 010	Ra-226	1.16	1.16	pCi/g		J

Appendix A
Verification Summary Tables

Data Verification	Y	N	N/A	Comment
Custody of Samples				
Are samples traceable through inspection of signature records on field and laboratory chains of custody (COCs)?	Y			COC No: 160-4422-2174.4
Has contractual turn-around time been met for all samples?	Y			Samples collected on 8/10/2016
Have all samples been preserved correctly and pertinent documentation included?	Y			Samples received at 1.6°C.
Is the laboratory log in sample receipt checklist present	Y			
Are any sample receipt non-conformances noted?	Y			
Standard Traceability				
Have certificate(s) been included for the LCS and MS?	Y			
Standards have not exceeded the certificate expiration date	Y			
Are chemical standards and reference materials traceable to a reliable source? (Reagent traceability summary)	Y			
Analytical Completeness				
Are all COC samples and associated analytical results reported in the laboratory data package?		N		PCBs and TCLP-Metals were reported in a different SDG.
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.	Y			
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)	Y			
Sample Data				
Is the Sample Data included for each COC requested analytical method?		N		PCBs and TCLP-Metals were reported in a different SDG.

Data Verification	Y	N	N/A	Comment
Is the calibration data included for each method? (ICAL, ICV, CCAL as required for each method)	Y			
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)	Y			
Are the method run logs and/or bench sheets included for each method?	Y			
Are the method preparation/extraction logs included for each applicable method?	Y			
Is the sample and QC raw data included for each method?	Y			
Is the internal Laboratory Review documented by checklists and included in the data package?	Y			

Appendix B
Validation Summary Tables

Radionuclide Analyses: Alpha Spectrometry Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
Preservation and Holding Times					
Were samples preserved correctly?	Y				
Were samples analyzed within holding times?	Y				
Standard Traceability					
Were all certificates included for the LCS and MS samples?	Y				
Were all standards and reference materials traceable to reliable source material?	Y				
Calibration Verification					
Are efficiencies within tolerance limits?	Y				
Are energies within tolerance limits?	Y				
Are background performance check count rates within tolerance limits?	Y				
Are appropriate peak resolution within control criteria?	Y				
LCS					
Has at least one LCS been prepared for up to 20 samples?	Y				
Is the LCS the same matrix as the samples in the reporting batch?	Y				
Are LCS %D (or %R) within QC acceptance limit?	Y				
Laboratory Duplicate					
Has at least one laboratory duplicate been prepared for up to 20 samples?	Y				
Are RPD and DER within QC acceptance limit?					YMTFA 44 SO 005 used for duplicate for Pu, isotopic U, Am, Th, C-14, Np. Results acceptable. Ra-226; RER > 1 at 3.83 on sample in different SDG. Duplicate sample was from same project, but the matrix of duplicate samples identified as concrete. Matrix issues affecting the Ra-226 are not

Radionuclide Analyses: Alpha Spectrometry Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
					representative of the samples in this SDG.
Matrix Spike					
Has at least one MS been prepared for up to 20 samples?			N/A		
Is MS %D (or %R) within QC acceptance limit?			N/A		
Method Blank					
Has at least one method blank been prepared for up to 20 samples?	Y				
Is the method blank the same matrix as the samples in the reporting batch?	Y				
Are the results less than 1.65 * CSU or within control limits?					<p>All blank results ND except Ra-226. Ra-226 blank only; Ra-226 result = 0.2498 pCi/g -</p> <p>The normalized difference was calculated for all samples and determined to be < 2.58 in two samples, which were also < 1.96. Because there were no other applicable quality issues, the results for samples YMTFA 33 SO 002 and YMTFA 64 SO 010 were qualified J. See table inserted after checklist for values.</p>
Chemical Yield - Tracers and Carriers					
Is yield reported for all samples and QC samples in the reporting batch?	Y				
Are percent recovery criteria satisfied for all yield results?	Y				

Text from es/er/ms-5, Evaluation of Radiochemical Data Usability, 1997.

The normalized absolute difference between the method blank and a sample result, given by the relationship below, is used in testing the null hypothesis that the sample and the method blank do not differ significantly when compared to their respective TPU. This test may be used as long as the method blank is reported in terms of activity per unit weight or volume consistent with the sample results.

$$\frac{|S - B|}{\sqrt{[TPU]_S^2 + [TPU]_B^2}}$$

S = Sample result

B = Method blank result

TPU = Total Propagated Uncertainty

If the normalized absolute difference is > 2.58 no qualification is necessary, as at the 1% level of significance, the conclusion is reached that the method blank and sample differ significantly. If the normalized absolute difference is between 1.96 and 2.58, qualify sample results \$ MDC "J," the sample and method blank differ at the 5% level of significance (sample results < MDC do not require qualification). If the normalized absolute difference is between 0 and 1.96 consider the effects of deficiencies in other quality-indicator samples prior to qualifying sample results "R", the conclusion is reached that the method blank and sample results differ at the 1% level of significance. If multiple quality deficiencies are encountered, qualify using the guidance provided in Appendix B.

Sample No.	Units	Lab Result	Total Uncertainty	Normalized Absolute Difference Calculation	Normalized Absolute Difference Final Result	Validation Qualifier
Blank	pCi/g	0.2498	0.0827			
YMTFA 44 SO 005	pCi/g	1.41	0.211	$\frac{ 1.41 - 0.2498 }{\sqrt{0.0445 + 0.0068}}$	5.1	None needed
YMTFA 33 SO 002	pCi/g	1.15	0.185	$\frac{ 1.15 - 0.2498 }{\sqrt{1.3225 + 0.0068}}$	0.78	J
YMTFA 45 SO 005	pCi/g	1.35	0.201	$\frac{ 1.35 - 0.2498 }{\sqrt{0.040 + 0.0068}}$	5.5	None needed
YMTFA 46 SO 005	pCi/g	1.17	0.188	$\frac{ 1.17 - 0.2498 }{\sqrt{0.035344 + 0.0068}}$	4.6	None needed
YMTFA 50 SO 005	pCi/g	1.83	0.247	$\frac{ 1.83 - 0.2498 }{\sqrt{0.061009 + 0.0068}}$	6.1	None needed
YMTFA 47 SO 005	pCi/g	1.55	0.227	$\frac{ 1.55 - 0.2498 }{\sqrt{0.0515 + 0.0068}}$	5.4	None needed
YMTFA 48 SO 005	pCi/g	1.21	0.191	$\frac{ 1.21 - 0.2498 }{\sqrt{0.0365 + 0.0068}}$	4.6	None needed
YMTFA 63 SO 010	pCi/g	0.913	0.166	$\frac{ 0.913 - 0.2498 }{\sqrt{0.0276 + 0.0068}}$	3.5	None needed
YMTFA 64 SO 010	pCi/g	1.16	0.186	$\frac{ 1.16 - 0.2498 }{\sqrt{1.3456 + 0.0068}}$	0.78	J

Analytical Data Review Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	JD Milloway	Date Verified:	9/7/16
SDG No(s).	18591-1; 18591-2		

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				
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		
	-SVOAs to Extraction ≤7 days (liquids),			X		

Analytical Data Review Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	JD Milloway	Date Verified:	9/7/16
SDG No(s).	18591-1; 18591-2		

Item No.	Criteria	Acceptable?				Comments
		Yes	No	NA	NR	
	≤14 days (solids)					
	-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		
	-Hexane Extractable Material, Oil and Grease ≤28 days			X		

Analytical Data Review Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	JD Milloway	Date Verified:	9/7/16
SDG No(s).	18591-1; 18591-2		

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