



**Data Validation Report  
SDG 160-18632-1**

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

Revision 0

ALLIANT CORPORATION  
320 N CEDAR BLUFF ROAD  
KNOXVILLE, TN 37923

*Dianne McNeill*

Validated by: \_\_\_\_\_  
Dianne McNeill

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## SCOPE

This report contains Level 3 data validation results for analytical data for sample delivery group (SDG) 160-18632-1 (J18632-1) for twelve 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 asbestos content, Total Characteristic Leaching Procedure (TCLP) Metals and Mercury (Hg), Polychlorinated Biphenyls (PCBs) and the following radionuclide analyses: Americium 241, Neptunium-237, isotopic Plutonium (Pu), isotopic Thorium (Th), isotopic Uranium (U), Carbon-14 (C-14), Total Beta Strontium (Total  $\beta$  Sr), Technetium-99 (Tc-99), Tritium, 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
- National Functional Guidelines for Superfund Organic Methods Data Review (September 2016)
- National Functional Guidelines for Inorganic Superfund Data Review (September 2016)
- 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 12 samples were evaluated. The TCLP Metals (with Hg), PCB, Total  $\beta$  Sr, Tc-99, Tritium, and Ra-226 analyses were performed by TestAmerica in Earth City, Missouri (TA-St. Louis). The analyses for asbestos content, Americium-241, Neptunium-237, isotopic Pu, isotopic Thorium, isotopic Uranium, and Carbon-14 were subcontracted to and performed by TestAmerica in Richland, Washington (TA-RL), Washington.

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
YMTFA69C	160-18632-1 (M841L)	PCBs TCLP Metals/Mercury Tritium Total $\beta$ Sr Tc-99 Ra-226 Americium-241

Project Sample ID	Laboratory Sample ID	Analysis
		Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA68C	160-18632-2 (M8408)	PCBs TCLP Metals/Mercury Tritium Total $\beta$ Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA67C	160-18632-3 (M841A)	PCBs TCLP Metals/Mercury Tritium Total $\beta$ Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA51C	160-18632-4 (M841C)	PCBs TCLP Metals/Mercury Tritium Total $\beta$ Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA70C	160-18632-5 (M841D)	PCBs TCLP Metals/Mercury Tritium Total $\beta$ Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14

Project Sample ID	Laboratory Sample ID	Analysis
YMTFA71C	160-18632-6 (M841E)	PCBs TCLP Metals/Mercury Tritium Total $\beta$ Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA71C	160-18632-7 (M841F)	PCBs TCLP Metals/Mercury Tritium Total $\beta$ Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA85 IN	160-18632-8 (M85R3)	PCBs TCLP Metals/Mercury Tritium Total $\beta$ Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA86 IN	160-18632-9 (M85R4)	Asbestos Content
YMTFA87 IN	160-18632-10 (M85R5)	Asbestos Content
YMTFA92 IN	160-18632-11 (M85R6)	Asbestos Content
YMTFA89 IN	160-18632-12 (M85R7)	Asbestos Content

### Holding times

Based on evaluation of the date of sample collection (08/12/16) and date of sample preparation and analyses, all recommended holding times per the analytical methods were met.

## **Preservation and Laboratory Sample Receipt**

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 recorded by the laboratory as 4.4° C and 19.0° C for the two coolers containing samples evaluated in this DVR, which is acceptable for the requested analyses. Custody seals were present at receipt on the cooler(s) received from the field and the cooler(s) used for lab-to-lab transfer.

## **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; COC 160-91468.1 for lab to lab sample transportation) were utilized for the analyses. 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:

### General

- These concrete core samples were disaggregated, dried, then puck milled and split for a variety of analyses. The possible heat generation may have compromised the Tritium, C-14 and Tc-99 native to these samples.

### Organics

#### PCBs:

- EPA Method 8082/8082A requires a minimum of 3 peaks to be used for PCB quantitation. Due to the presence of multiple Aroclors in sample YMTFA68C, less than 5 peaks were used for quantitation. (Validator note: chromatography review was performed for this sample; chromatograms were not markedly different from other samples in the SDG)
- CCV recoveries for Aroclor 1260 and the surrogate were outside the lower quality control (QC) limits on the secondary column, but within acceptable QC limits on the primary column for CCV 160-266473/27 and CCV 160-266473/39. The laboratory case narrative noted that there were no

hits above the reporting limit (RL) for Aroclor 1260 and the surrogate recoveries in the samples were within acceptable QC limits on the primary column, so confirmation was not needed.

- The internal standard (IS) eluted outside the retention time window for CCV 160-266473/3, CCV 160-267639/49, CCV 160-267639/50 and ICV 160-267639/14. The laboratory case narrative noted that this retention time shift was taken into account when reviewing the sample(s) for target compounds.
- It was noted that that laboratory control sample (LCS) and method blank required a copper clean-up to reduce matrix interferences caused by sulfur. (Validator note: No sample was listed, only lab QC).

Inorganics

TCLP Metals (ICP) and Mercury:

- Hg was detected in the method blank at a concentration above the method detection limit (MDL) but below the RL.

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.

**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.

<b>Qualifier</b>	<b>Definition</b>
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

### **TCLP Extractions**

Seven composite concrete samples were extracted by SW-846 Method 1311 with appropriate batch QCs. There were no problems noted during the extraction.

### **Polychlorinated Biphenyls by Gas Chromatography (GC)**

Seven composite concrete samples were extracted and analyzed for PCBs by SW-846 Method 8082A.

For the initial calibration verifications (ICVs), the % difference (%D) were slightly > 20% for multiple peaks in multiple ICVs; however, the criterion for the minimum number of acceptable peaks (3) was met in all cases. For the continuing calibration verifications (CCVs), peaks were slightly > 20% for multiple peaks. Generally, the minimum # of acceptable peaks (3) was available. However, fewer than 3 peaks were available for PCB-1221 and PCB-1260 in CCVs associated with the samples evaluated in this DVR. PCB-1221 was not detected in any of the samples so no qualifications were applied to PCB-1221 results. PCB-1260 detects were qualified as estimated (J) in YMTFA68C, YMTFA67C, YMTFA70C.

The intercolumn relative percent differences (RPD) were > 40% for the PCB-1260 detections in samples YMTFA68C, YMTFA67C, and YMTFA70C. The PCB-1260 and total PCB results were qualified as estimated (J) in these samples.

Batch QC (method blank, LCS, MS/MSD) were acceptable except as noted above. Sample QCs (surrogates, internal standards) were acceptable except as noted above.

### **TCLP Metals by Inductively Coupled Plasma (ICP) and Mercury by Cold Vapor Atomic Absorption (CVAA)**

TCLP extracts of seven composite concrete samples were extracted and analyzed for Metals and Mercury by SW-846 Method 6010C and 7470A. Initial calibration, ICVs, CCVs, LCS, MS/MSD and serial dilution (SD) were acceptable. Hg was detected in the method blank at a level below the RL. The low concentrations of Hg detected in samples YMTFA51C, YMTFA70C, YMTFA71C, and YMTFA84 UNK3 C were therefore qualified as nondetects (U) and the RLs were raised to the levels reported.

### **Asbestos Content by Polarized Light Microscopy (PLM)**

Five samples were analyzed for asbestos content National Institute for Occupational Safety and Health (NIOSH) Method 9002. All data were acceptable.

### **Radionuclides**

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

- Tritium (H3-04-RC/liquid scintillation counting [LSC]),
- Total  $\beta$  Sr (Method SR-03-RC/gas flow proportional counter [GFPC]),
- Tc-99 (Method TC-02-RC/LSC),
- Ra-226 (ST-RC-0301/Alpha Spectrometry),
- Americium-241 (RL-ALP-001/Alpha Spectroscopy),
- Neptunium-237 (RL-ALP-013/Alpha Spectroscopy),
- Isotopic Pu (RL-ALP-002/Alpha Spectroscopy),
- Isotopic Th (RL-ALP-001/Alpha Spectroscopy),
- Isotopic U (RL-ALP-009/Alpha Spectroscopy), and
- C-14 (RL-LSC-008/LSC).

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

These concrete core samples were disaggregated, dried, then puck milled and split for a variety of analyses. The possible heat generation may have compromised the Tritium, C-14 and Tc-99 native to these samples. The results for these parameters were therefore qualified as estimated (J/UJ) in all seven samples.

#### Alpha Spectrometry

##### Ra-226

Ra-226 was detected in the method blank and the normalized difference was calculated by the validator to be < 2.58 for all samples using the equation shown below. The Ra-226 results were therefore qualified as estimated (J) for all seven samples.

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

Where

S = Sample result

B = Method blank result

TPU = Total Propagated Uncertainty

##### Isotopic Th

The relative error ratio (RER) was > 1 for Th-230 in the laboratory duplicate. The sample was considered to be indicative of matrix for all samples, so the Th-230 detects in all seven samples were qualified as estimated (J).

##### Isotopic Uranium

The RER was > 1 for U-233/234 in the laboratory duplicate. The sample was considered to be indicative of matrix for all samples, so the U-233/234 detects in all seven samples were qualified as estimated (J).

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

### **Summary**

For the seven samples analyzed for PCBs, TCLP Metals plus Hg, and Radionuclides, the qualifications summarized below were assigned. No qualifications were needed for the five samples analyzed for asbestos content only.

- Tritium, Tc-99, and C-14 results for all seven samples were qualified as estimated (J/UJ) due to possible heat generation during sample preparation.
- Ra-226 results for all seven samples were qualified as estimated (J) because Ra-226 was detected in the method blank and the normalized difference for each sample was < 2.58.
- Th-230 was also qualified as estimated (J) in all seven samples because the Th-230 duplicate RER < 1.

- U-233/234 results for all seven samples were qualified estimated (J) because the duplicate RER <1.
- PCB-1260 and Total PCBs were qualified estimated (J) for samples YMTFA68C, YMTFA67C, and YMTFA70C because the intercolumn RPD >40%. PCB-1260 was also qualified estimated (J) in the listed samples for CCV outlier %Ds.
- Low concentration Hg detections were qualified as nondetect (U) and the RLs raised to the level reported in samples YMTFA51C, YMTFA70C, YMTFA71C, and YMTFA84 UNK3 C due to blank contamination.

There were no other qualifications assigned to any samples evaluated for this DVR.

### Summary of Result Qualifiers

Sample ID	Parameter	Lab Result	Qualified Result	Units	Lab Qualifier	Validation Qualifier
YMTFA69C	Ra-226	<b>0.671</b>	<b>0.671</b>	pCi/g		J
YMTFA69C	Tritium	0.0102	0.0102	pCi/g	U	UJ
YMTFA69C	Tc-99	0.119	0.119	pCi/g	U	UJ
YMTFA69C	Th-230	<b>0.136</b>	<b>0.136</b>	pCi/g		J
YMTFA69C	U-233/234	<b>0.267</b>	<b>0.267</b>	pCi/g		J
YMTFA69C	C-14	-0.0876	-0.0876	pCi/g	U	UJ
YMTFA68C	PCB-1260	<b>0.014</b>	<b>0.014</b>	mg/Kg	J	J
YMTFA68C	Total PCBs	<b>0.030</b>	<b>0.030</b>	mg/Kg	J	J
YMTFA68C	Ra-226	<b>0.29</b>	<b>0.29</b>	pCi/g		J
YMTFA68C	Tritium	-0.011	-0.011	pCi/g	U	UJ
YMTFA68C	Tc-99	-0.0324	-0.0324	pCi/g	U	UJ
YMTFA68C	Th-230	<b>0.144</b>	<b>0.144</b>	pCi/g		J
YMTFA68C	U-233/234	<b>0.173</b>	<b>0.173</b>	pCi/g		J
YMTFA68C	C-14	0.0368	0.0368	pCi/g	U	UJ
YMTFA67C	PCB-1260	<b>0.0081</b>	<b>0.0081</b>	mg/Kg	J	J
YMTFA67C	Total PCBs	<b>0.0081</b>	<b>0.0081</b>	mg/Kg	J	J
YMTFA67C	Ra-226	<b>1.02</b>	<b>1.02</b>	pCi/g		J
YMTFA67C	Tritium	-0.0516	-0.0516	pCi/g	U	UJ
YMTFA67C	Tc-99	0.177	0.177	pCi/g	U	UJ
YMTFA67C	Th-230	<b>0.137</b>	<b>0.137</b>	pCi/g		J
YMTFA67C	U-233/234	<b>0.233</b>	<b>0.233</b>	pCi/g		J
YMTFA67C	C-14	0.1	0.1	pCi/g	U	UJ
YMTFA51C	Ra-226	<b>0.617</b>	<b>0.617</b>	pCi/g		J
YMTFA51C	Tritium	<b>0.4</b>	<b>0.4</b>	pCi/g		J
YMTFA51C	Tc-99	0.116	0.116	pCi/g	U	UJ
YMTFA51C	Th-230	<b>0.11</b>	<b>0.11</b>	pCi/g		J
YMTFA51C	U-233/234	<b>0.152</b>	<b>0.152</b>	pCi/g		J
YMTFA51C	C-14	0.101	0.101	pCi/g	U	UJ
YMTFA51C	Hg	<b>0.0001</b>	0.0001	mg/L	JB	U

Sample ID	Parameter	Lab Result	Qualified Result	Units	Lab Qualifier	Validation Qualifier
YMTFA70C	PCB-1260	<b>0.009</b>	<b>0.009</b>	mg/Kg	J	J
YMTFA70C	Total PCBs	<b>0.009</b>	<b>0.009</b>	mg/Kg	J	J
YMTFA70C	Ra-226	<b>0.943</b>	<b>0.943</b>	pCi/g		J
YMTFA70C	Tritium	0.0366	0.0366	pCi/g	U	UJ
YMTFA70C	Tc-99	0.104	0.104	pCi/g	U	UJ
YMTFA70C	Th-230	<b>0.106</b>	<b>0.106</b>	pCi/g		J
YMTFA70C	U-233/234	<b>0.21</b>	<b>0.21</b>	pCi/g		J
YMTFA70C	C-14	-0.0665	-0.0665	pCi/g	U	UJ
YMTFA70C	Hg	<b>0.00014</b>	0.00014	mg/L	JB	U
YMTFA71C	Ra-226	<b>0.606</b>	<b>0.606</b>	pCi/g		J
YMTFA71C	Tritium	-0.0546	-0.0546	pCi/g	U	UJ
YMTFA71C	Tc-99	-0.0221	-0.0221	pCi/g	U	UJ
YMTFA71C	Th-230	<b>0.171</b>	<b>0.171</b>	pCi/g		J
YMTFA71C	U-233/234	<b>0.376</b>	<b>0.376</b>	pCi/g		J
YMTFA71C	C-14	0.0301	0.0301	pCi/g	U	UJ
YMTFA71C	Hg	<b>0.00013</b>	0.00013	mg/L	JB	U
YMTFA84 UNK3 C	Ra-226	<b>0.738</b>	<b>0.738</b>	pCi/g		J
YMTFA84 UNK3 C	Tritium	-0.0828	-0.0828	pCi/g	U	UJ
YMTFA84 UNK3 C	Tc-99	0.118	0.118	pCi/g	U	UJ
YMTFA84 UNK3 C	Th-230	<b>0.339</b>	<b>0.339</b>	pCi/g		J
YMTFA84 UNK3 C	U-233/234	<b>0.205</b>	<b>0.205</b>	pCi/g		J
YMTFA84 UNK3 C	C-14	-0.0662	-0.0662	pCi/g	U	UJ
YMTFA84 UNK3 C	Hg	<b>0.00014</b>	0.00014	mg/L	JB	U

**Appendix A**  
**Verification Summary Tables**

<b>Data Verification</b>	<b>Y</b>	<b>N</b>	<b>N/A</b>	<b>Comment</b>
<b>Custody of Samples</b>				
Are samples traceable through inspection of signature records on field and laboratory chains of custody (COCs)?	Y			COC No. 160-4416-2171.2; COC 160--91468.1 for lab to lab sample transportation. Custody seals were present at receipt on both. Temps were 4.4° C and 19.0° C. Asbestos samples were noted as received in bags, rather than bottles per the method and the approved QAPP but no qualifications are needed.
Has contractual turn-around time been met for all samples?			N/A	Samples rec'd by lab on 8/13/16 and reported on 9/30/2016. Contractual TAT for lab was not available to the validator.
Have all samples been preserved correctly and pertinent documentation included?	Y			
Is the laboratory log in sample receipt checklist present	Y			
Are any sample receipt non-conformances noted?			N/A	Asbestos samples logged in after other samples, per lab notations (not contracted). All samples logged in and run. No nonconformance.
<b>Standard Traceability</b>				
Have certificate(s) been included for the LCS and MS?	Y			

<b>Data Verification</b>	<b>Y</b>	<b>N</b>	<b>N/A</b>	<b>Comment</b>
Standards have not exceeded the certificate expiration date	Y			
Are chemical standards and reference materials traceable to a reliable source? (Reagent traceability summary)	Y			
<b>Analytical Completeness</b>				
Are all COC samples and associated analytical results reported in the laboratory data package?	Y			
<b>Data Summaries</b>				
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			Case narrative does not identify all issues. See validation checklists and DVR for details.
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			
<b>Sample Data</b>				
Is the Sample Data included for each COC requested analytical method?	Y			
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**

<b>TCLP Extraction</b>	Y	N	N/A	Qualifier	Comment or Reason Code
Was a ZHE vessel used for VOAs?			N/A		
Was ZHE checked for leaks after extraction?			N/A		
Did the lab use proper bottles?	Y				
Was the %solid determined correctly?			N/A		Concrete samples, reported on an as-received basis.
If appropriate, did the lab reduce particle size?	Y				
Was the correct extraction fluid used?	Y				
Was the pH of the extraction fluid correct?	Y				
Was the correct weight of extraction fluid used?	Y				
For VOAs, was the sample weight 25 grams or less?			N/A		
Were the TCLP extracts properly preserved?	Y				
Is there a TCLP blank with the TCLP fluid for a batch of up to 20 samples?	Y				

<b>Metals by ICP (SW6010) Mercury by CVAA (SW7470A)</b>	Y	N	N/A	Qualifier	Comment or Reason Code
<b>Preservation and Holding Times</b>					
Were samples properly preserved?	Y				
Are sample preparation sheets present and account for all extractions and digestions for reported samples?	Y				
Have the samples been prepared and analyzed within holding times?	Y				
<b>Detection Limits and Target Analytes</b>					
Do all samples show RLs <= the SAP Recommended Reporting Limits?	Y				
Are all the SAP target analytes reported?	Y				
<b>Initial Calibration</b>					
Was the Calibration within acceptance criteria?	Y				Yes, for all target analytes.
<b>Calibration Verification</b>					
Was a second source ICV analyzed after calibration with recoveries within acceptance criteria?	Y				
Were CCVs analyzed at the required frequency with recoveries within acceptance criteria? For ICP, CCVs and low level CCVs (CCVL) as applicable.	Y				
Are the ICV and CCV/CCVL Summary forms present?	Y				
Was the ICP CRQL Check Standard analyzed with recoveries within acceptance criteria?	Y				
<b>Method Blank and ICB/CCBs</b>					
Has at least one method blank been prepared For each batch of up to 20 samples?	Y				
Is the method blank the same matrix as the samples in the reporting batch?	Y				
Were target analytes detected in the method blank above the MDL?	Y				Low level detection of Hg in MB. Low Hg detects (at approx. same concentration as blank) in samples YMTFA51C, YMTFA70C, YMTFA71C, YMTFA84 UNK3 C were qualified as nondetects (U) and the RLS raised to the concentration

<b>Metals by ICP (SW6010)</b>	Y	N	N/A	Qualifier	Comment or Reason Code
<b>Mercury by CVAA (SW7470A)</b>					reported.
Were the ICB and CCBs analyzed at the required frequency with results within acceptance criteria?	Y				
Are the Method Blank and ICB/CCB Summary forms present?	Y				
<b>ICP Interference Check Samples</b>					
Were the ICP ICSA/ICSAB interference check standards analyzed as required with results within acceptance criteria?	Y				
<b>LCS/LCSD</b>					
Has at least one LCS been prepared for each preparation batch containing up to 20 samples?	Y				
Is the LCS the same matrix as the samples in the reporting batch?	Y				
Is the LCS spiked with all target analytes listed in the SAP?	Y				
Are the LCS %RECs within the applicable QC criteria?	Y				
Are the LCS/LCSD RPDs within the applicable QC criteria?			N/A		LCS ONLY
<b>Matrix Spike/Matrix Spike Duplicate</b>					
Has at least one MS/MSD pair been prepared for a batch containing up to 20 samples?	Y				
Are the MS/MSD spiked with all target analytes listed in the SAP?	Y				
Are MS and MSD %RECs within the applicable QC limits?	Y				
Are MS/MSD RPDs within the applicable QC limits?	Y				
<b>Duplicates</b>					
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.)		N			
If a laboratory duplicate was analyzed, were the RPDs within acceptance criteria?			N/A		
Was a field duplicate analyzed?		N			
If a field duplicate was analyzed, were the RPDs within the 50% acceptance criteria?			N/A		
<b>Serial Dilution</b>					
Was the Serial Dilution within acceptance limits?			N/A		SD on project sample was NC due to low levels and nondetects. SD was

<b>Metals by ICP (SW6010) Mercury by CVAA (SW7470A)</b>	Y	N	N/A	Qualifier	Comment or Reason Code
					qualitatively acceptable.
<b>Sample Quantitation and Documentation</b>					
Are reported sample concentrations within the instrument linear range?	Y				
Have sample reporting limits and reported concentrations been adjusted for analytical dilutions?			N/A		
Are instrument runlogs present and account for all reported sample results?	Y				
Have all Laboratory Case Narrative comments and findings been addressed in the data validation process?	Y				

<b>Polychlorinated Biphenyl</b>	Y	N	N/A	Qualifier	Comment or Reason Code
<b>Preservation and Holding Times</b>					
Were samples properly preserved?	Y				
Have the samples been analyzed within holding times?	Y				
<b>Detection Limits and Preservation</b>					
Do all laboratory RLs <= recommended reporting limits in the SAP?	Y				
<b>Initial Calibration</b>					
Are minimum calibration curve with minimum 5 points analyzed prior to sample analysis?	Y				
Are %RSDs within method criteria?		N			%D on multiple ICV peaks were slightly > 20% for multiple peaks. In all cases, minimum # acceptable peaks (3) were available.
<b>Calibration Verification</b>					
Are calibration verification standard analyzed at the appropriate frequency?	Y				
RT within RT windows established by initial calibration?	Y				IS outside window in two CCVs. No issues were identified with sample IS RTs; however, this is indicative of instrument issues and/or need to reestablish initial cal.
Are %D (difference or drift) within 20% of the average initial calibration factors?			N		%D on multiple CCV peaks were slightly > 20% for multiple peaks. Minimum # acceptable peaks (3) were available for all peaks except for PCB-1221 and PCB-1260. No identification issues are suspected. PCB-1221 was not detected so no qualifications were applied to PCB-1221 results. PCB-1260 detects were qualified J in YMTFA68C, YMTFA67C, YMTFA70C.
<b>Method Blank</b>					
Is the Method Blank extracted and analyzed for each analytical batch of up to 20 samples?	Y				
Is the Method Blank Summary form present?	Y				
Is the method blank the same matrix as the samples in the reporting batch?		N			Blank is solid matrix. Samples are crushed concrete. No qualifications assigned.
Is the blank at similar (low, medium, or trace)	Y				

<b>Polychlorinated Biphenyl</b>	Y	N	N/A	Qualifier	Comment or Reason Code
concentration level?					
Does the blank have any detects above MDL?		N			
<b>Surrogate Recovery</b>					
Are all samples and QCs spiked with surrogate compounds?	Y				
Are percent recoveries within the method criteria results?	Y				Surrogate out high in one CCV, but okay in all samples.
<b>LCS/LCSD</b>					
Has at least one LCS been prepared for each preparation batch containing up to 20 samples?	Y				
Is the LCS the same matrix as the samples in the reporting batch?	Y				
Is the LCS spiked with all target analytes listed in the SAP?	Y				
Are the LCS %RECs within the applicable QC criteria?	Y				
Are the LCS/LCSD RPDs within the applicable QC criteria?			N/A		LCS only.
<b>Matrix Spike/Matrix Spike Duplicate</b>					
Has at least one MS/MSD pair been prepared for a batch with sample counts up to 20 samples?	Y				
Are the MS/MSD spiked with target analyte specified in the SAP?	Y				
MS and MSD %RECs within the applicable QC limits?	Y				
MS/MSD RPDs within the applicable QC limits?	Y				
<b>Target Analyte Identification</b>					
Do the positively identified compound meet the identification criteria?		N			PCB-1260 and Total PCBs qualified J in YMTFA68C, YMTFA67C, YMTFA70C for intercolumn RPD >40%.
Are the RTs of the positively identified target analytes within RT window established by initial calibration standards?	Y				
<b>Target Analyte Quantitation and Reported Quantitation Limit</b>					
Are the results for all positively identified analytes are calculated correctly?			N/A		Recalculations not performed for Level 3.
Are the reporting limits calculated for the non-detects and reported correctly?			N/A		See above.

<b>Radionuclide Analyses:</b> Alpha Spectrometry Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
<b>Preservation and Holding Times</b>					
Were samples preserved correctly?	Y				
Were samples analyzed within holding times?	Y				Narrative notes that the samples could not be thoroughly homogenized before sub-sampling was performed due to sample matrix and that the samples had small rocks and were of varying colors.
<b>Standard Traceability</b>					
Were all certificates included for the LCS and MS samples?	Y				
Were all standards and reference materials traceable to reliable source material?	Y				
<b>Calibration Verification</b>					
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				
<b>LCS</b>					
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				
<b>Laboratory Duplicate</b>					
Has at least one laboratory duplicate been prepared for up to 20 samples?	Y				
Are RPD and DER within QC acceptance limit?		N			RER >1 for Th-230 at 2.5 and U-233/234. J qualifiers assigned to all samples for Th-230 and U-233/234 results.

<b>Radionuclide Analyses:</b> <b>Alpha Spectrometry</b> <b>Gas Flow Proportional Counting</b> <b>Liquid Scintillation Counting</b>	Y	N	N/A	Qualifier	Comment or Reason Code
<b>Matrix Spike</b>					
Has at least one MS been prepared for up to 20 samples?		N			MS for Tritium only; Tritium MS acceptable.
Is MS %D (or %R) within QC acceptance limit?			N/A		
<b>Method Blank</b>					
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?		N			All blank results ND except Ra-226.  The normalized difference was calculated for all samples and determined to be < 2.58 in all samples. Ra-226 qualified J in all samples.  See table inserted after checklist for values.
<b>Chemical Yield - Tracers and Carriers</b>					
Is yield reported for all samples and QC samples in the reporting batch?	Y				
Are percent recovery criteria satisfied for all yield results?	Y				

Note: Tritium, Carbon-14 (C-14) and Technetium-99 (Tc-99) qualified J/UJ in all samples because possible heat generated during prep may have compromised this parameter.

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.

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

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.	Analyte	Units	Lab Result	Total Uncertainty	Normalized Absolute Difference Final Result	Validation Qualifier
Blank	Ra-226	pCi/g	0.4325	0.125		
YMTFA69C	Ra-226	pCi/g	0.671	0.147	1.236	J
YMTFA68C	Ra-226	pCi/g	0.29	0.186	-0.636	J
YMTFA67C	Ra-226	pCi/g	1.02	0.211	2.396	J
YMTFA51C	Ra-226	pCi/g	0.617	0.134	1.007	J
YMTFA70C	Ra-226	pCi/g	0.943	0.199	2.172	J
YMTFA71C	Ra-226	pCi/g	0.606	0.136	0.939	J
YMTFA84 UNK3 C	Ra-226	pCi/g	0.738	0.149	1.571	J

<b>Asbestos by Polarized Light Microscopy</b>	Y	N	N/A	Qualifier	Comment or Reason Code
<b>Preservation and Holding Times</b>					
Were samples properly preserved?			N/A	None	Samples were noted as received in bags, rather than bottles per the method and the approved QAPP; no qualifications are needed.
Have the samples been analyzed within holding times?			N/A	None	Asbestos has a holding time of "indefinite".
<b>Detection Limits</b>					
Do all laboratory RLs <= recommended reporting limits in the SAP?		Y			
<b>Asbestos Content Evaluation</b>					
Were each of the listed parameters evaluated? Actinolite Amosite Anthophyllite Chrysotile Crocidolite Tremolite		Y			
<b>Laboratory Duplicate</b>					
Has at least one laboratory duplicate been prepared for up to 20 samples?		N			No duplicate was prepared or evaluated in association with the two samples in this SDG. No qualifications are needed.
<b>Overall Evaluation</b>					
Were any issues or anomalies noted?		N			

## Analytical Data Review Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	JD Milloway	Date Verified:	10/19/16
SDG No(s).	18632-1		

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 of insulation samples was not required to be delivered on ice
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:	10/19/16
SDG No(s).	18632-1		

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:	10/19/16
SDG No(s).	18632-1		

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				