



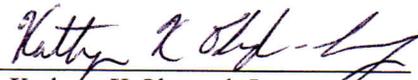
**Data Validation Report
SDG 160-18646-2**

**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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Validated by:


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Date: 10/14/2016

SCOPE

This report contains Level 3 data validation results for analytical data for SDG No. 160-18646-2 for two solid 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 (total strontium and technetium-99).

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 solid samples (SDG No. 160-18646-2) 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, radionuclides (total strontium and technetium-99)	YMTFA53EJ	160-16846-7
Total metals, PCBs, radionuclides (total strontium and technetium-99)	YMTFA52EJ	160-16846-8

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 TAL 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 0.4° C. The laboratory Log-in Sample Receipt Checklist is included in the data package. No log-in issues or discrepancies are noted in the checklist.

Analytical Methods, Reporting Units, and Detection Limits

The COC requested TCLP metals analysis was not performed. Total metals analysis was performed and reported for the two solid samples in this SDG. All other 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)

The COC requested TCLP metals analysis was not performed. Total metals analysis was performed and reported for the two solid samples in this SDG. 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

Not applicable.

Laboratory Case Narratives

The following issues were noted in the case narratives:

PCBs:

- Samples YMTFA53EJ, YMTFA52EJ, LCS 160-266347/2-A and MB 160-266347/1-A required a copper clean-up to reduce matrix interferences caused by sulfur.
- Surrogate recovery was outside control limits for samples YMTFA53EJ and YMTFA52EJ. Evidence of matrix interference is present; therefore, re-extraction and/or re-analysis was not performed.
- Internal standard (ISTD) responses exceeded the control limit on the primary column for samples YMTFA53EJ and YMTFA52EJ. The sample results associated with this ISTD were reported from the other column, which met ISTD acceptance criteria.
- The IS eluted outside the retention time window for samples YMTFA53EJ, YMTFA52EJ, and CCV 160-267639/49, CCV 160-267639/50 and ICV 160-265639/14. The retention time shift was taken into account when reviewing the samples for target compounds.

Total Metals (ICP):

- Samples YMTFA53EJ and YMTFA52EJ were diluted due to sample matrix. The samples were high in salts. Elevated reporting limits are provided.

- The matrix spike/matrix spike duplicate (MS/MSD) recovery and/or precision for barium, cadmium, and lead was outside control limits, indicating a matrix interference. The sample was a non-homogeneous mixture of soil. The LCS was within acceptable limits.

Radionuclides (Total Strontium):

- The samples became tarry/sticky on heating. They could not be ground or pulverized per the standard procedure. The samples were processed “as received”.

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
NJ	The analyte has been “tentatively identified“ or “presumptively” as present and the associated numerical value is the estimated concentration in the sample.
R	result is rejected

TCLP Extractions

TCLP extractions were not performed for the samples in this SDG. The COC lists TCLP metals analysis, but total metals were analyzed and reported for the samples in this SDG.

Metals (ICP) and Mercury

Two samples were prepared and analyzed for total ICP metals by SW-846 Method 6010C and mercury by SW-846 Method 7470A. Holding times, initial and continual calibrations, batch QC (blank, LCS, MS/MSD) were acceptable except for the following: The sample YMTFA53EJ matrix spike duplicate (MSD) recoveries were outside control limits for barium, cadmium and lead. The MS/MSD RPD was outside control limits for barium. Sample YMTFA53EJ and YMTFA52EJ results for barium, cadmium and lead are qualified as estimated, J. No further qualification of metals data was required.

Polychlorinated Biphenyl by GC

Two 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, with the following exceptions: Internal standards eluted outside the retention time window for the samples and for associated calibration checks (ICV and CCVs). Internal standard responses for the samples exceeded the control limit on the primary column, and the sample results were therefore reported from the second column, which met internal standard response acceptance criteria. The RPD between columns exceeds criteria for the aroclor 1260 detects reported for the two samples. The sample surrogate recoveries are high for the reported detects. The PCB-1260 detections for both samples are qualified as tentatively identified, NJ.

Radionuclides

Two samples were analyzed for the following radionuclides: total strontium, and technetium-99. Holding times, applicable instrument calibrations, and sample and batch QCs were acceptable for all methods. Traceable standard certificates were acceptable.

Gas Flow Proportional Counter

Total beta strontium analysis was performed by gas flow proportional counter. The Laboratory Control Sample (LCS) had acceptable percent recovery. 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. No qualification of data was required.

Liquid Scintillation Counter

Technetium (Tc-99) was analyzed by liquid Scintillation counter. The Laboratory Control Sample (LCS) had acceptable percent recovery. 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

- The PCB-1260 detects for samples YMTFA52EJ and YMTFA53EJ are qualified as tentatively identified and estimated due to compound identification criteria exceedances (retention time shifts affecting the calibration verification standards [ICV and CCVs] and samples) and sample internal standard and surrogate recoveries and column confirmation precisions outside control limits.
- The barium, cadmium and lead detects for samples YMTFA52EJ and YMTFA53EJ are qualified as estimated due to matrix spike duplicate results outside of control limits.

Summary of Result Qualifiers

Sample No.	Parameter	Laboratory Result	Qualified Result	Units	Laboratory Qualifier	Validation Qualifier
YMTFA52EJ	PCB-1260	0.61 J	0.61 NJ	mg/Kg	J	NJ
YMTFA53EJ	PCB-1260	0.053 J	0.053 NJ	mg/Kg	J	NJ
YMTFA52EJ	Barium	84	84 J	mg/Kg	None	J
YMTFA53EJ	Barium	24	24 J	mg/Kg	None	J
YMTFA52EJ	Cadmium	16	16 J	mg/Kg	None	J
YMTFA53EJ	Cadmium	13	13 J	mg/Kg	None	J
YMTFA52EJ	Lead	180	180 J	mg/Kg	None	J
YMTFA53EJ	Lead	41	41 J	mg/Kg	None	J

Appendix A

Verification Summary Table

Data Verification SDG 160-18646-2	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			
Is the laboratory log in sample receipt checklist present	x			
Are any sample receipt non-conformances noted?	x			The COC lists TCLP metals analysis. Total metals analysis was performed and reported for the samples.
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			

Data Verification SDG 160-18646-2	Y	N	N/A	Comment
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 the sample analyses are not included.

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				
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			There were retention time shifts for the CCV bracketing the sample analyses for both columns.
Are %D (difference or drift) within 20% of the average initial calibration factors?		x			The ICV and CCV %Ds for the second column were within criteria. The sample results are reported from the second 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				

Polychlorinated Biphenyl (Method 8082A)	Y	N	N/A	Qualifier	Comment or Reason Code
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			The sample YMTF53EJ and YMTFA52EJ surrogate recoveries are high for the sample results reported from the second column. The recoveries are 193% and 167%, respectively.
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 are 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		
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				

Polychlorinated Biphenyl (Method 8082A)	Y	N	N/A	Qualifier	Comment or Reason Code
Internal Standards					
Were internal standards added to all samples and QC samples?	x				
Are internal standard retention times within method criteria?		x		None	The internal standard (ISTD) eluted outside the retention time window for samples YMTFA53EJ, YMTFA52EJ and associated calibration checks CCV 160-26639/49, CCV 160-267639/50 and ICV 160-267639/14. This retention time shift was taken into account when reviewing the samples for target compounds.
Are internal standard areas within method criteria?		x			The ISTD response exceeded control limits on the primary column for samples YMTFA53EJ and YMTFA52EJ. Therefore, the sample results were reported from the second column, which met ISTD acceptance criteria.
Target Analyte Identification					

Polychlorinated Biphenyl (Method 8082A)	Y	N	N/A	Qualifier	Comment or Reason Code
Do the positively identified compounds meet the identification criteria?		x			Due to high IS % recovery for the first column, The Aroclor 1260 detect %RPD between the first and second column results are high outside criteria: Sample YMTFA53EJ: 196.8% RPD between first and second column detects. YMTFA52EJ: 196.61 % RPD between first and second column detects.
Are the RTs of the positively identified target analytes within RT windows established by initial calibration standards?		x			The RT shift for the internal standard was taken into account when reviewing the samples for target compounds.
Target Analyte Quantitation and Reported Quantitation Limit					
Are the results for all positively identified analytes calculated correctly?		x			The Aroclor 1260 detections for the samples are qualified as tentatively identified "NJ".
Are the reporting limits calculated for the non-detects and reported correctly	x				

Total Metals by ICP (SW6010) Total 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.
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				

Total Metals by ICP (SW6010) Total 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		J	MSD %RECs are outside QC limits for the following analytes: Ba MSD: 134%REC Cd MSD: 126 %REC Pb MSD: 130 %REC
Are MS/MSD RPDs within the applicable QC limits?		x		J	The MS/MSD RPDs are outside QC limits as follows: Ba: 35%RPD
Duplicates					
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			

Total Metals by ICP (SW6010) Total Mercury by CVAA (SW7470A)	Y	N	N/A	Qualifier	Comment or Reason Code
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 Gamma Spectrometry Gas Flow Proportional Counting 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				
Are the results less than 1.65 * CSU or within control limits?	x				
Chemical Yield - Tracers and Carriers					
Is yield reported for all samples and QC samples in the reporting batch?	x				

Radiological Data Validation Gamma Spectrometry Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
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).	18646-1; 18646-2; 18646-3		

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				Expansion Joint Samples- Insufficient amount for all analysis required. Tritium not analyzed, 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	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).	18646-1; 18646-2; 18646-3		

Item No.	Criteria	Acceptable?				Comments
		Yes	No	NA	NR	
	Analysis ≤28 days					
	-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).	18646-1; 18646-2; 18646-3		

Item No.	Criteria	Acceptable?				Comments
		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				