



**Data Validation Report
SDG 160-18591-1**

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

Revision 0

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Validated by:  Date: 10/14/2016
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SCOPE

This report contains Level 3 data validation results for analytical data for SDG 160-18591 for nine 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 TCLP Metals, PCBs and radionuclides (tritium, total beta strontium, and technetium-99).

REFERENCES

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 (USEPA), (November 2002)
- *National Functional Guidelines for Superfund Organic Methods Data Review*. USEPA (August 2014)
- *National Functional Guidelines for Inorganic Superfund Data Review*. USEPA (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 nine soil samples were evaluated. All analyses were performed by TestAmerica in Earth City, Missouri (TA-St. Louis). The following lists analytical methods and sample numbers for reported results.

Project Sample ID	Laboratory Sample ID	Analysis
YMTFA44SO005	160-18591-1	PCBs TCLP Metals/mercury Radionuclides
YMTFA33SO002	160-18591-2	PCBs TCLP Metals/mercury Radionuclides
YMTFA45SO005	160-18591-3	PCBs TCLP Metals/mercury Radionuclides
YMTFA46SO005	160-18591-4	PCBs TCLP Metals/mercury Radionuclides
YMTFA50SO005	160-18591-5	PCBs TCLP Metals/mercury Radionuclides
YMTFA47SO005	160-18591-6	PCBs TCLP Metals/mercury Radionuclides
YMTFA48SO005	160-18591-7	PCBs TCLP Metals/mercury Radionuclides

Project Sample ID	Laboratory Sample ID	Analysis
YMTFA63SO010	160-18591-8	PCBs TCLP Metals/mercury Radionuclides
YMTFA64SO010	160-18591-9	PCBs TCLP Metals/mercury Radionuclides

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 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.

The samples arrived at TA-St. Louis facility at cooler temperature of 1.6 °C.

Analytical Methods, Reporting Units, and Detection Limits

All analytical methods specified (or equivalent to those specified) on the COC (COC No. 160-4422-2174.1) were utilized for the analyses. All results were reported in appropriate units. The 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:

PCBs:

- DCB (surrogate) and internal standard eluted outside the retention window for one of the CCBs. The retention time shift was taken into account when reviewing the samples for target compounds.
- RPDs between primary and confirmation column exceeded limit for surrogate and PCB-1260 in samples YMTFA64SO010 and YMTFA64SO010.
- Due to presence of multiple PCBs, less than 5 peaks were used for quantitation.

TCLP Metals (ICP) and Mercury:

- Sample YMTFA47SO005, YMTFA48SO00, YMTFA63SO010, YMTFA64SO010 were diluted due to sample matrix. Elevated RLs are provided.
- The ICSA was outside upper QC limit for arsenic.
- Mercury was detected in method blank at a level above the MDL but below the RL.

Radionuclides:

- Several samples counted off the upper end of the quench curve parameter. A small amount of quenching agent (nitromethane) was added to the affected vials and recounted. The recount results were within the quench curve parameter and are reported.

Verification/Validation Checklists, Data Qualifiers, and Qualifier Definitions

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

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

TCLP Extractions

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

Polychlorinated Biphenyl by GC

Nine soil 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 exception:

- RPDs between primary and confirmation column exceeded limit for surrogate and PCB-1260 in samples YMTFA64SO010 and YMTFA64SO010. PCB-1260 and total PCBs were qualified as estimated (J) in the two samples.

TCLP Metals(ICP) and Mercury

TCLP extracts of nine soil samples were prepared and analyzed for ICP metals and mercury by SW-846 Method 6010C and 7470A. Holding times, initial and continual calibrations, blanks, LCS, MS/MSD were acceptable with the following exception:

- All detected results for mercury were qualified as nondetects (U) at the RL.
- ICSA was recovered above the QC limit for arsenic. No qualifications were required since arsenic was not detected in the affected samples.

Radionuclides

Four sample was analyzed for the following radionuclides: tritium, total beta 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) and matrix spike (MS) had acceptable percent recoveries. The laboratory duplicate analyses had acceptable relative percent difference (RPD) and duplicate error ratio (DER) results. Chemical recoveries and yields were within acceptable limits. Method blank results were less than the MDAs. No qualification of data was required.

Liquid Scintillation Counter

Tritium, technetium (Tc-99) and carbon-14 were analyzed by liquid Scintillation counter. The Laboratory Control Sample (LCS) and matrix spike (MS) had acceptable percent recoveries. 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.

Summary

- RPDs for PCB-1260 between primary and confirmation results exceed the limit for samples YMTFA63SO010 and YMTFA64SO010. PCB-1260 and total PCBs in these samples were qualified as estimated (J) in these two samples.
- Mercury was detected in the method blank. All detected results for mercury were qualified as nondetects (U) at the RL.

Summary of Result Qualifiers

Sample No.	Parameter	Laboratory Result	Qualified Result	Units	Laboratory Qualifier	Validation Qualifier
YMTFA63SO010	PCB-1260	0.015	0.015	mg/kg	J	J
YMTFA63SO010	Total PCBs	0.015	0.015	mg/kg	J	J
YMTFA64SO010	PCB-1260	0.017	0.017	mg/kg	J	J
YMTFA64SO010	Total PCBs	0.030	0.017	mg/kg	J	J
YMTFA44SO005	Mercury	0.00013	0.0010	mg/L	JB	U
YMTFA33SO002	Mercury	0.00013	0.0010	mg/L	JB	U
YMTFA45SO005	Mercury	0.00014	0.0010	mg/L	JB	U
YMTFA46SO005	Mercury	0.00015	0.0010	mg/L	JB	U
YMTFA50SO005	Mercury	0.00015	0.0010	mg/L	JB	U
YMTFA47SO005	Mercury	0.00013	0.0010	mg/L	JB	U
YMTFA48SO005	Mercury	0.00014	0.0010	mg/L	JB	U
YMTFA63SO010	Mercury	0.00010	0.0010	mg/L	JB	U

Sample No.	Parameter	Laboratory Result	Qualified Result	Units	Laboratory Qualifier	Validation Qualifier
YMTFA64SO010	Mercury	0.00011	0.0010	mg/L	JB	U

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)?	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			
Standard Traceability				
Have certificate(s) been included for the LCS and MS?	x			
Standards have not exceeded the certificate expiration date	x			
Are chemical standards and reference materials traceable to a reliable source? (Reagent traceability summary)	x			
Analytical Completeness				
Are all COC samples and associated analytical results reported in the laboratory data package?	x			
Data Summaries				
The case narrative is present and summarizes the sample receipt and analysis information including any analytical anomalies for all methods reported in the data package.	x			
Other data summary forms are present as applicable (detection, sample results, surrogate, tracer/carrier, QC results and association, prep and analysis chronicle, method and sample summaries)	x			
Sample Data				
Is the Sample Data included for each COC requested analytical method?	x			
Is the calibration data included for each method? (ICAL, ICV, CCAL as required for each method)	x			
Are the QC summary forms included for each method? (MB, ICS/CCB, LCS/LCSD, MS/MSD, surrogates, 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			

Data Verification	Y	N	N/A	Comment
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			

Appendix B
Validation Summary Tables

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

Polychlorinated Biphenyls (SW8082A)	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				
Target Analytes and Detection Limits					
Are all the SAP target analytes reported?	x				
Do all laboratory RLs <= SAP recommended reporting limits?	x				
Initial Calibration					
Are minimum calibration curve with minimum 5 points analyzed prior to sample analysis?		x			6 point for PCB1016/1260 and PCB1221/1254 mixtures analyzed. Single point used for all other PCBs.
Are %RSDs within method criteria?	x				For PCB1221/1254. Linear regression r-square above 0.990 for PCB1016/1260
Calibration Verification					
Are calibration verification standard analyzed at the appropriate frequency?	x				
RT within RT windows established by initial calibration?	x				
Are %D (difference or drift) within 20% of the average initial calibration?	x				No closing CCV analyzed after samples.
Method Blank					
Is the Method Blank extracted and analyzed for each analytical batch of up to 20 samples?	x				
Is the Method Blank Summary form present?	x				
Is the method blank the same matrix as the samples in the reporting batch?	x				
Is the blank at similar (low, medium, or trace) concentration level?	x				
Does the blank have any detects above MDL?		x			

Polychlorinated Biphenyls (SW8082A)	Y	N	N/A	Qualifier	Comment or Reason Code
Surrogate Recovery					
Are all samples and QCs spiked with surrogate compounds?	x				
Are percent recoveries within the method criteria results?	x				
Internal Standard					
Were internal standards added to all samples and QCs?	x				
Are internal standard retention times within method criteria?	x				
Are internal standard area within method 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			PCB1016/1260 only.
Are the LCS %RECs within the applicable QC criteria?	x				
Are the LCS/LCSD RPDs within the applicable QC criteria?			x		No LCSD
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				The MS/MSD performed on sample not in this SDG.
Are the MS/MSD spiked with target analyte specified in the SAP?		x			PCB1016/1260 only.
MS and MSD %RECs within the applicable QC limits?	x				
MS/MSD RPDs within the applicable QC limits?	x				
Target Analyte Identification					
Do the positively identified compound meet the identification criteria?			x		
Are the RTs of the positively identified target analytes within RT window established by initial calibration standards?			x		
Target Analyte Quantitation and Reported Quantitation Limit					
Are the results for all positively identified analytes are calculated correctly?			x		

Polychlorinated Biphenyls (SW8082A)	Y	N	N/A	Qualifier	Comment or Reason Code
Are the reporting limits calculated for the non-detects and reported correctly?					
Are the RPD between primary and confirmation columns within criteria?		x		J	PCB-1260 RPDs 40% in samples YMTFA63SO0 10 and YMTFA64SO0 10.

Metals by ICP (SW6010) 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				
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			U	Hg was detected in the method blank above the MDL.
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				
ICP Interference Check Samples					
Were the ICP ICSA/ICSAB interference check standards analyzed as required with results within acceptance criteria?		x			ICSA was outside QC limit for arsenic.
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				

Metals by ICP (SW6010) Mercury by CVAA (SW7470A)	Y	N	N/A	Qualifier	Comment or Reason Code
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		No LCSD
Matrix Spike/Matrix Spike Duplicate					
Has at least one MS/MSD pair been prepared for a batch containing up to 20 samples?	x				MS/MSDs performed on samples not in this SDG.
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				
Are MS/MSD RPDs within the applicable QC limits?	x				
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 analyzed?		x			
If a field duplicate was analyzed, were the RPDs within the 50% acceptance criteria?			x		
Serial Dilution					
Was the Serial Dilution within acceptance limits?	x				
Sample Quantitation and Documentation					
Are reported sample concentrations within the instrument linear range?			x		
Have sample reporting limits and reported concentrations been adjusted for analytical dilutions?			x		
Are instrument runlogs present and account for all reported sample results?	x		x		
Have all Laboratory Case Narrative comments and findings been addressed in the data validation process?	x				

Radionuclide Analyses: Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
Preservation and Holding Times					
Were samples preserved correctly?	x				
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?					
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 limit?	x				
Laboratory Duplicate					
Has at least one laboratory duplicate been prepared for up to 20 samples?	x				
Are RPD and DER within QC acceptance limit?	x				
Matrix Spike					
Has at least one MS been prepared for up to 20 samples?	x				
Is MS %D (or %R) within QC acceptance limit?	x				MS performed on sample not in this SDG.
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				

Radionuclide Analyses: Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
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:	9/7/16
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				