

SCOPE

This report contains Level 3 data validation results for analytical data for sample delivery group (SDG) 160-18571-1 for five concrete composite 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 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, isotopic Uranium, Carbon-14, Total Beta Strontium, 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 five composite concrete samples were evaluated. The TCLP Metals (with Hg), PCB, Total Beta Strontium, Tc-99, Tritium, and Ra-226 analyses were performed by TestAmerica in Earth City, Missouri (TA-St. Louis). The analyses for 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
YMTFA81 9404 C1	160-18571-1 (M84V7)	PCBs TCLP Metals/Mercury Tritium Total Beta Strontium Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA81 9404 C2	160-18571-2 (M84V9)	PCBs TCLP Metals/Mercury Tritium Total Beta Strontium Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA81 9404 C3	160-18571-3 (M84WC)	PCBs TCLP Metals/Mercury Tritium Total Beta Strontium Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA80 9418 C	160-18571-4 (M84WD)	PCBs TCLP Metals/Mercury Tritium Total Beta Strontium Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA82 UNK1 C	160-18571-5 (M84WE)	PCBs TCLP Metals/Mercury

Project Sample ID	Laboratory Sample ID	Analysis
		Tritium Total Beta Strontium Tc-99 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 (08/9/16) and date of sample preparation and 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 chain of custody (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 20 °C. Samples were stated to have been received “where required, on ice” in the laboratory case narrative prior to the statement in the narrative that samples were received with “no thermal preservation”. Based on the second statement and temperature at receipt, the first statement is presumed to be stock text that was inadvertently not deleted. Samples were not shipped on ice. Although this is a SAP/QAPP deviation, no impact upon target analyte recoveries is anticipated based on this temperature. No qualifications were assigned.

Samples were collected into bags. In Table 4.2.2 of the SAP/QAPP, containers listed for the analyses evaluated in this DVR differ from sample bags; however, elsewhere in the SAP/QAPP, it is stated that the concrete samples will be collected into sample bags. Bag material is not noted. The primary concern for samples containerized in bags is phthalate contamination. Phthalates are not target analytes for the samples evaluated in this DVR. No qualifications were assigned.

Analytical Methods, Reporting Units, and Detection Limits

All analytical methods specified (or equivalent to those specified) on the COC (COC No. 1160-4416-2171.2; 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 analyses. The possible heat generation may have compromised the Tritium, Carbon-14 and Tc-99 native to these samples.

Organics

PCBs:

- Sample YMTFA81 9404 C2 was diluted due to the abundance of PCB-1016. As such, the elevated reporting limits (RLs) were provided (Validator note: all other PCBs were reported from the undiluted analysis of the sample; therefore elevated RLs have no impact on data usability).
- The internal standard (IS) eluted outside the retention time window for the following continuing calibration verification (CCV): (CCVIS 160-266473/3). This retention time shift was taken into account when reviewing the sample(s) for target compounds.
- The CCV recoveries for Aroclor 1260 and the surrogate decachlorobiphenyl (DCB) were outside the lower QC limits on the secondary column, but within acceptable QC limits on the primary column. There were no hits above the RL for Aroclor 1260 and the surrogate recoveries in the samples were within acceptable quality control (QC) limits on the primary column; therefore confirmation is not needed. (CCV 160-266473/39) (Validator note: This case narrative statement is not accurate; Aroclor-1260 was detected above the RL in sample YMTFA81 9404 C2)
- The relative percent difference (RPD) between the primary and confirmation column exceeded 40% for PCB-1016 detected in sample YMTFA80 9418 C. The lower value has been reported and qualified in accordance with the laboratory's standard operating procedure (SOP).
- Surrogate recovery for the matrix spike duplicate (MSD) performed using sample YMTFA81 9404 C1 was outside control limits (160-18571-C-1-S MSD). The case narrative noted that evidence of matrix interference was present; therefore, re-extraction and/or re-analysis was not performed.
- EPA Method 8082/8082A requires a minimum of 3 peaks to be used for PCB quantitation. Due to the presence of matrix interferences in sample YMTFA81 9404 C1, less than 5 peaks were used for quantitation.
- The MSD recovery for PCB-1260 was outside control limits. It was noted in the case narrative that sample matrix interference and/or non-homogeneity were suspected because the associated

laboratory control sample (LCS) and matrix spike (MS) recoveries were within acceptance limits. (Validator note: the MS was performed using the same sample and should have exhibited similar matrix issues; see validation text).

- The MS/MSD precision for PCB-1016 and PCB-1260 was outside control limits. The laboratory noted that sample matrix interference and/or non-homogeneity were suspected because the associated LCS / laboratory control sample duplicate (LCSD) precision was within acceptance limits. (Validator note: This case narrative statement is not accurate; the LCS was acceptable, but no LCSD was reported).

Inorganics

TCLP Metals (ICP) and Mercury:

- The samples were diluted due to being high in salts. Elevated RLs were provided.
- The samples were re-digested/re-extracted due to a timer failure resulting in the samples digesting longer than permitted by the SOP. Therefore the MS/MSD was spiked after preservation.

Radionuclides

Ra-226

- Sample YMTFA81 9404 C1 produced white crystals during the co-precipitation process. The filter was rinsed several times to help break up the crystals. A few larger crystal chunks remained on the filter.
- The At-217 tracer recovery for the LCS was outside the QC limit of 30% at 28.2%. Quality Systems Manual for Environmental Laboratories (QSM Rev. 5.0) allows for reporting results as quantitative when tracer recoveries are below 30% if a) the relative uncertainty associated with the tracer recovery is less than 10% (2 sigma), b) spectral resolution requirements are met and there are no indications of spectral interferences, and c) detection limit requirements are met. All three of these criteria were met for the associated samples. (Validator note: all of the samples in this SDG were associated with this LCS).
- The At-217 tracer recovery for sample YMTFA81 9404 C1 was outside the QC limits of 30%: (25.3). QSM Rev. 5.0 allows for reporting results as quantitative when tracer recoveries are below 30% if a) the relative uncertainty associated with the tracer recovery is less than 10% (2 sigma), b) spectral resolution requirements are met and there are no indications of spectral interferences, and c) detection limit requirements are met. All of these criteria were met for this sample.

Tc-99

- Sample YMTFA81 9404 C1 and its duplicate had Tc-99 tracer recoveries below the 30% QC limit at 23.5%. The LCS had an acceptable spike recovery demonstrating acceptable sample preparation and instrument performance. Matrix interference is suspected. The data were qualified and reported.
- All 5 samples, the sample duplicate, the LCS, and method blank counted off the upper end of the quench curve parameter (transformed external standard spectrum [tSIE]/ automatic efficiency correction [AEC]). A small amount (10 uL) of quenching agent (nitromethane) was added to the affected vials and recounted. The recount results were within the quench curve parameter and were reported.
- Sample YMTFA81 9404 C1 and its duplicate did not meet the detection goal of 1.00 pCi/g due to a high bias attributed to low tracer recoveries. The data were qualified and reported.

Subcontracted Radionuclides

Isotopic Pu

- The Pu 239/240 activity for the blank was above the minimum detectable activity (MDA) and contract required detection limit (CRDL). The sample activity was below the MDA and CRDL and the samples act as their own blank (per the laboratory case narrative), and the data were accepted.

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

General

As noted in the laboratory case narrative, the samples were disaggregated, dried, then puck milled and split for a variety of analyses. The possible heat generation may have compromised the Tritium, Carbon-14 and Tc-99 native to these samples. The nondetect results for Tritium, Carbon-14 and Tc-99 were therefore qualified as estimated (UJ) in all samples.

TCLP Extractions

Five 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)

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

For the initial calibration and initial calibration verification (ICV) the % difference (%D) were slightly > 20% for PCB-1254, PCB-1221, and PCB-1260. The criterion for the minimum number of acceptable peaks (3) was met for PCB-1254; however, for PCB-1221, the criteria were consistently not met. Data review indicated that false positives were unlikely to be an issue; however, instrument conditions were indicated to not be optimal based on these results. However, because PCB-1221 was not detected in any of the samples, no qualifications were required based on the ICVs.

In the CCVs, the PCB-1260 peak was slightly >20% for multiple peaks, in multiple CCVs. The PCB-1260 detect in sample YMTFA81 9404 C2 was therefore qualified as estimated (J). The internal standard retention time (RT) shifted outside the RT window in CCV3.

The surrogate recovery was out high in one CCV. Surrogate recovery was also out high in the MSD run using sample YMTFA81 9404 C1. MSD recoveries were all approximately 2x above the upper QC limit and all MS/MSD RPDs exceeded the RPD limit. The laboratory case-narrative attributed the MSD surrogate and MSD spike recoveries to the sample matrix; however, similar issues were observed for the CCV, and not observed for the MS or parent sample. The validator noted that the MSD appears to have

been spiked 2x; however, without documentation of a laboratory error, no adjustments can be made. All results (all nondetects) were therefore qualified as estimated (UJ) in this sample.

The intercolumn RPD was > 40% for the PCB-1016 detection in sample YMTFA80 9418 C. The PCB-1016 detect was qualified as estimated (J) in this sample.

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 three composite concrete samples were extracted and analyzed for Metals and Mercury by SW-846 Method 6010C and 7470A. Initial calibration, ICVs, CCVs, batch QCs (blank, LCS, MS/MSD) were acceptable.

Radionuclides

Three 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 Strontium (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 Plutonium (RL-ALP-002/Alpha Spectroscopy),
- Isotopic Thorium (RL-ALP-001/Alpha Spectroscopy),
- Isotopic Uranium (RL-ALP-009/Alpha Spectroscopy), and
- Carbon-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, except as noted below.

Alpha Spectrometry

Ra-226

The Ra-226 Tracer At-217 was slightly low in sample YMTFA81 9404 C1 and in the LCS. The Ra-226 result was qualified as estimated (J) in sample YMTFA81 9404 C1. Because At217 was acceptable in other samples, the LCS issue was not considered representative of entire batch; therefore, no other samples were qualified for this issue.

Ra-226 was detected in the method blank at 0.1953 pCi/g, and the normalized difference was calculated by the validator to be < 2.58 for all samples. The Ra-226 results were therefore qualified as estimated (J) for all samples, using the calculation shown below.

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

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 $< \text{MDC}$ 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, with a minimum qualification of estimated (J).

Alpha Spectroscopy Isotopic Pu

Plutonium was detected in the method blank; however, because all Pu sample results were non-detect, no qualifications were needed.

Liquid Scintillation Counter Tc-99

The Tc-99 tracer recovery was less than the 30% limit for sample YMTFA81 9404 C1 and its duplicate. This sample duplicate pair was considered to be representative of the entire batch; therefore the Tc-99 results (all non-detects) were qualified as estimated (UJ) for all samples.

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

Summary

- Possible heat generated from puck-mill grinding may have compromised the Tritium, Carbon-14 and Tc-99 native to these samples. Results for Tritium, Carbon-14 and Tc-99 (all nondetects) were therefore qualified as estimated (UJ) in all samples.
- The PCB-1260 detect in sample YMTFA81 9404 C2 was qualified estimated (J) due multiple CCV %Ds $> 20\%$.
- All PCB results (all nondetects) were qualified estimated (UJ) in sample YMTFA81 9404 C1 for MSD recoveries and RPDs outside criteria for all spiked analytes.
- The PCB-1016 detect was qualified as estimated (J) in sample YMTFA80 9418 C for an intercolumn RPD $> 40\%$.
- Tc-99 results for all samples (all nondetects) were qualified estimated (UJ) for Tc-99 tracer recoveries below the 30% limit in sample YMTFA81 9404 C1 and its duplicate, which were considered to be representative of the batch.
- Ra-226 was qualified as estimated (J) in sample YMTFA81 9404 C1 because the At-217 tracer was recovered slightly below acceptable yield.
- Ra-226 was detected in the method blank and the normalized difference was < 2.58 for all samples. Therefore, the Ra-226 results (all detects) were qualified as estimated (J) for all 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
YMTFA81 9404 C1	PCB-1016	0.0096	0.0096	mg/Kg	U	UJ
YMTFA81 9404 C1	PCB-1221	0.0096	0.0096	mg/Kg	U	UJ
YMTFA81 9404 C1	PCB-1232	0.0096	0.0096	mg/Kg	U	UJ
YMTFA81 9404 C1	PCB-1242	0.0096	0.0096	mg/Kg	U	UJ
YMTFA81 9404 C1	PCB-1248	0.0096	0.0096	mg/Kg	U	UJ
YMTFA81 9404 C1	PCB-1254	0.0080	0.0080	mg/Kg	U	UJ
YMTFA81 9404 C1	PCB-1260	0.0080	0.0080	mg/Kg	U	UJ
YMTFA81 9404 C1	PCB-1262	0.0080	0.0080	mg/Kg	U	UJ
YMTFA81 9404 C1	PCB-1268	0.0080	0.0080	mg/Kg	U	UJ
YMTFA81 9404 C1	Tritium	0.178	0.178	pCi/g	U	UJ
YMTFA81 9404 C1	Ra-226	0.734	0.734	pCi/g		J
YMTFA81 9404 C1	Tc-99	0.0971	0.0971	pCi/g	U	UJ
YMTFA81 9404 C1	Carbon-14	5.09E-03±9.5E-02	5.09E-03±9.5E-02	pCi/g	U	UJ
YMTFA81 9404 C2	PCB-1260	0.069	0.069	mg/Kg		J
YMTFA81 9404 C2	Tritium	0.184	0.184	pCi/g	U	UJ
YMTFA81 9404 C2	Ra-226	0.566	0.566	pCi/g		J
YMTFA81 9404 C2	Tc-99	0.390	0.390	pCi/g	U	UJ
YMTFA81 9404 C2	Carbon-14	-9.56E-02 ± 9.3E-02	-9.56E-02 ± 9.3E-02	pCi/g	U	UJ
YMTFA81 9404 C3	Tritium	0.186	0.186	pCi/g	U	UJ
YMTFA81 9404 C3	Ra-226	0.736	0.736	pCi/g		J
YMTFA81 9404 C3	Tc-99	-0.0396	-0.0396	pCi/g	U	UJ
YMTFA81 9404 C3	Carbon-14	-1.09E-01 ± 9.1E-02	-1.09E-01 ± 9.1E-02	pCi/g	U	UJ
YMTFA80 9418 C	PCB-1016	0.013	0.013	mg/Kg	J	J
YMTFA80 9418 C	Tritium	0.132	0.132	pCi/g	U	UJ
YMTFA80 9418 C	Ra-226	0.354	0.354	pCi/g		J
YMTFA80 9418 C	Tc-99	0.0207	0.0207	pCi/g	U	UJ
YMTFA80 9418 C	Carbon-14	-4.36E-02 ± 9.0E-02	-4.36E-02 ± 9.0E-02	pCi/g	U	UJ
YMTFA82 UNK1 C	Tritium	0.213	0.213	pCi/g	U	UJ
YMTFA82 UNK1	Ra-226	0.632	0.632	pCi/g		J

Sample No.	Parameter	Laboratory Result	Qualified Result	Units	Laboratory Qualifier	Validation Qualifier
C						
YMTFA82 UNK1 C	Tc-99	0.127	0.127	pCi/g	U	UJ
YMTFA82 UNK1 C	Carbon-14	3.50E-02 ± 1.0E-01	3.50E-02 ± 1.0E- 01	pCi/g	U	UJ

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			
Has contractual turn-around time been met for all samples?	Y			
Have all samples been preserved correctly and pertinent documentation included?		N		Samples received at 20°C; temperature will not have an impact on target analytes. All other criteria were met. No qualifications were assigned.
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?	Y			
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			Case narrative is not accurate for all statements. 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			

Data Verification	Y	N	N/A	Comment
Sample Data				
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

TCLP Extraction	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				

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?		N			Samples were received at 20°C in bags; Analytes of interest are stable at this temp and metals analytes are not compromised by bagging. No qualifications were assigned.
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				
Detection Limits and Target Analytes					
Do all samples show RLs <= the SAP Recommended Reporting Limits?	Y				
Are all the SAP target analytes reported?	Y				
Initial Calibration					
Was the Calibration within acceptance criteria?	Y				Yes, for all target analytes.
Calibration Verification					
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				
Method Blank and ICB/CCBs					
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?		N			

Metals by ICP (SW6010) Mercury by CVAA (SW7470A)	Y	N	N/A	Qualifier	Comment or Reason Code
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				
ICP Interference Check Samples					
Were the ICP ICSA/ICSAB interference check standards analyzed as required with results within acceptance criteria?	Y				
LCS/LCSD					
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
Matrix Spike/Matrix Spike Duplicate					
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				
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.)		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		

Metals by ICP (SW6010) Mercury by CVAA (SW7470A)	Y	N	N/A	Qualifier	Comment or Reason Code
Serial Dilution					
Was the Serial Dilution within acceptance limits?			N/A		SD on non-project sample or project sample not evaluated for this DVR; results were NC
Sample Quantitation and Documentation					
Are reported sample concentrations within the instrument linear range?	Y				
Have sample reporting limits and reported concentrations been adjusted for analytical dilutions?	Y				
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				

Polychlorinated Biphenyl	Y	N	N/A	Qualifier	Comment or Reason Code
Preservation and Holding Times					
Were samples properly preserved?		N			Samples received at 20°C in sample bags. Temperature and sample containerization are not anticipated to have any impact on PCB recoveries.
Have the samples been analyzed within holding times?	Y				
Detection Limits and Preservation					
Do all laboratory RLs <= recommended reporting limits in the SAP?	Y				
Initial Calibration					
Are minimum calibration curve with minimum 5 points analyzed prior to sample analysis?	Y				
Are %RSDs within method criteria?		N			%D (not RSD) on multiple ICV peaks were slightly > 20% for PCB-1254, PCB-1221, PCB-1260. PCB-1254 met the minimum # acceptable peaks (3); however, PCB-1221 consistently did not. Data review indicated that false positives were unlikely to be an issue; however, instrument conditions are indicated to not be optimal based on these results. PCB-1221 was not detected in any samples so no qualifications were required; however, this should be noted in the DVR.
Calibration Verification					
Are calibration verification standard analyzed at the appropriate frequency?	Y				
RT within RT windows established by initial calibration?			N		Internal standard retention time shift outside RT window in CCV3.
Are %D (difference or drift) within 20% of the average initial calibration factors?			N		PCB-1260 peak slightly >20%, multiple peaks, multiple CCVs. The PCB-1260 detect in sample YMTFA81 9404 C2 was qualified as estimated, J.

Polychlorinated Biphenyl	Y	N	N/A	Qualifier	Comment or Reason Code
					Note also that the case narrative states that CCV recoveries for Aroclor 1260 and the surrogate were outside QC limits on the secondary column, but within acceptable QC limits on the primary column, then states that there were no hits above the RL for Aroclor 1260 and the surrogate recoveries in the samples were within acceptable quality control (QC) limits on the primary column; so confirmation is not needed. However, there is a sample detect for PCB-1260 > RL in sample YMTFA81 9404 C2. Qualification assigned as note above in this comment box for tis line item.
Method Blank					
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) concentration level?	Y				
Does the blank have any detects above MDL?		N			
Surrogate Recovery					
Are all samples and QCs spiked with surrogate compounds?	Y				
Are percent recoveries within the method criteria results?		N			Surrogate out high in one CCV. Surrogate also out high in MSD run using sample YMTFA81 9404 C1. Narrative attributed this to matrix; however, same issue not seen in MS or parent sample. MSD appears to have been spiked 2x; however, without

Polychlorinated Biphenyl	Y	N	N/A	Qualifier	Comment or Reason Code
					documentation and %R adjustment, surrogate & MSD results cannot be adjusted & require qualification. All results J/UJ in this sample.
LCS/LCSD					
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. Note that case narrative uses LCS/LCSD results to support matrix issues for cause of MS/MSD results and to indicate acceptable instrument reproducibility; however, no LCSD was reported. Review of run logs indicate no LCSD was run. LCSD analysis is not required and no qualifications are needed; however, this case narrative statement is not accurate.
Matrix Spike/Matrix Spike Duplicate					
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?		N			%RECs high for PCB-1016 and PCB-1260; MSD appears to have been spiked 2x; however, without documentation of this, no adjustment of %Rs can be made. All sample results qualified J/UJ
MS/MSD RPDs within the applicable QC limits?		N			Same as above.
Target Analyte Identification					
Do the positively identified compound meet the identification criteria?		N			PCB-1016 was qualified J in sample YMTFA80 9418 C for intercolumn RPD

Polychlorinated Biphenyl	Y	N	N/A	Qualifier	Comment or Reason Code
					>40%.
Are the RTs of the positively identified target analytes within RT window established by initial calibration standards?	Y				
Target Analyte Quantitation and Reported Quantitation Limit					
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.

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?		N			<p>Samples were received at 20°C in bags; ice is not required for rad & rad analytes are not compromised by bagging. No qualifications were assigned.</p> <p>Note: Samples were disaggregated, dried then puck milled and split for a variety of analyses. Per the case narrative, the possible heat generation may have compromised the Tritium, Carbon-14 and Technetium-99 native to these samples. These analytes were not detected & were qualified UJ in all samples.</p>
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				

Radionuclide Analyses: Alpha Spectrometry Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
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?	Y				
Matrix Spike					
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		
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?		N			<p>All blank results ND except Pu and Ra-226.</p> <p>Because Pu was not detected in any samples; no qualifications were needed for Pu in method blank.</p> <p>Ra-226 blank result = 0.1953 pCi/g -</p> <p>The normalized difference was calculated for all samples and determined to be < 2.58 in all 5 samples. RA results for all 5 samples were qualified J.</p> <p>See table inserted after checklist for values.</p>

Radionuclide Analyses: Alpha Spectrometry 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?	Y				
Are percent recovery criteria satisfied for all yield results?		N			<p>Ra-226 Tracer At-217 low at 25.3 (30-110) in sample YMTFA81 9404 C1 and LCS. Ra-226 result qualified in sample. Because At217 was acceptable in other samples, LCS issue was not considered representative of entire batch.</p> <p>Tc-99 parent sample YMTFA81 9404 C1 & dupe tracer slightly < 30% limit. Tc-99 qualified UJ in all samples (dupe considered representative of all project samples in this SDG).</p>

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	0.1953	0.0956			
YMTFA81 9404 C1	pCi/g	0.734	0.218	$\frac{ 0.734 - 0.1953 }{\sqrt{0.047 + 0.009}}$	2.27	J
YMTFA81 9404 C2	pCi/g	0.566	0.165	$\frac{ 0.566 - 0.1953 }{\sqrt{0.027 + 0.009}}$	1.95	J
YMTFA81 9404 C3	pCi/g	0.736	0.221	$\frac{ 0.736 - 0.1953 }{\sqrt{0.049 + 0.009}}$	2.24	J
YMTFA80 9418 C	pCi/g	0.354	0.115	$\frac{ 0.354 - 0.1953 }{\sqrt{0.013 + 0.009}}$	1.07	J
YMTFA82 UNK1 C	pCi/g	0.632	0.171	$\frac{ 0.632 - 0.1953 }{\sqrt{0.029 + 0.009}}$	2.241	J

Analytical Data Review Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	JD Milloway	Date Verified:	10/18/16
SDG No(s).	18571-1		

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				YMTFA81 9404 C1 and the associated lab duplicate sample did not meet the detection limit for Tc-99 due to low tracer recoveries.
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			Samples were not cooled to procedural prescribed temperature
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				

Analytical Data Review Verification Checklist

Laboratory:	TestAmerica	SOW or Contract No.:	Outfall 200
Verifier Name:	JD Milloway	Date Verified:	10/18/16
SDG No(s).	18571-1		

Item No.	Criteria	Acceptable?				Comments
		Yes	No	NA	NR	
	-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:	JD Milloway	Date Verified:	10/18/16
SDG No(s).	18571-1		

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				