



**Data Validation Report
SDG 160-18646-1**

**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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SCOPE

This report contains Level 3 data validation results for analytical data for sample delivery group (SDG) 160-18646-1 (J186646-1) for six 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 (Total β 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 six composite concrete samples were evaluated. The TCLP Metals (with Hg), PCB, Total β Sr, 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. Two expansion joint samples listed on the chain of custody (COC) were reported in a different SDG. There was no effect on completeness for the samples evaluated in this Data Validation Report (DVR).

The table below lists analytical methods and sample numbers for reported results evaluated in this DVR. Subcontract work order (ID) numbers are shown in parenthesis.

Project Sample ID	Laboratory Sample ID	Analysis
YMTFA72C	160-18646-1 (M85R9)	PCBs TCLP Metals/Mercury Tritium Total β Sr Tc-99

Project Sample ID	Laboratory Sample ID	Analysis
		Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA65C	160-18646-2 (M85TF)	PCBs TCLP Metals/Mercury Tritium Total β Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA58C	160-18646-3 (M85TJ)	PCBs TCLP Metals/Mercury Tritium Total β Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA57C	160-18646-4 (M85TK)	PCBs TCLP Metals/Mercury Tritium Total β Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium Isotopic Uranium Carbon-14
YMTFA56C	160-18646-5 (M85TL)	PCBs TCLP Metals/Mercury Tritium Total β Sr Tc-99 Ra-226 Americium-241 Neptunium-237 Isotopic Plutonium Isotopic Thorium

Project Sample ID	Laboratory Sample ID	Analysis
		Isotopic Uranium Carbon-14
YMTFA55C	160-18646-6 (M85TM)	PCBs TCLP Metals/Mercury Tritium Total β Sr 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 (08/15/16) 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 0.4 °C, which is acceptable for the requested analyses. Custody seals were present at receipt on the cooler received on the field but not the container 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 160-91913.2 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:

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 YMTFA65C, less than 5 peaks were used for quantitation. (Validators 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.
- Sample YMTFA72C required a copper clean-up to reduce matrix interferences caused by sulfur.

Inorganics

TCLP Metals (ICP) and Mercury:

- The samples were diluted due to being high in salts. Elevated RLs were provided.

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.

Total β Sr

Samples YMTFA72C and YMTFA65C had strontium carrier recoveries above the 110% QC limit due to matrix interferences. Per the case narrative, the laboratory control sample (LCS) had an acceptable spike recovery demonstrating acceptable sample preparation and instrument performance. The samples were truncated to 100% by the laboratory to reduce any potential bias a high carrier recovery may have. The data were qualified and reported by the laboratory. (Validator note: see validation text below).

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

TCLP Extractions

Six 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)

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

For the initial calibration verifications (ICVs), the % difference (%D) values 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. In all but one CCV, minimum # acceptable peaks (3) were available. However, PCB-1254 in two separate calibrations had %D at 98.1 and 96.9%. PCB-1254 was not detected in any samples so no qualifications were assigned. However, internal standard (IS) retention times (RTs) were outside the acceptable windows in two CCVs, and the surrogate in one CCV was recovered high. IS and surrogate recoveries and IS RTs were acceptable in all samples; however, these calibration outliers indicate that instrument maintenance and/or re-establishment of initial calibration parameters may be needed. Select sample chromatograms were evaluated to ensure these issues were not affecting sample results. Baseline rise was observed; however, this is attributable to sample matrix. No data were qualified.

The intercolumn relative percent difference (RPD) was > 40% for the PCB-1260 detection in sample YMTFA56C. The PCB-1260 detect and total PCB result were 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 six 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

Six 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 β 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 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

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 samples.

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

Where

S = Sample result

B = Method blank result

TPU = Total Propagated Uncertainty

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

Isotopic Th

Th-230 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 above. Th-230 results were therefore qualified as estimated (J) for all samples.

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

Isotopic Uranium

The RER was > 1 for U-238 in the laboratory duplicate which was considered to be indicative of matrix for all samples, so the U-238 detects in all samples were qualified as estimated (J).

GFPC

Total β Sr

Carrier recoveries were > the 100% QC limit for samples YMTFA72C and YMTFA65C at 120 and 113 respectively. The laboratory case narrative attributed this to sample matrix. The lab narrative further noted that the samples were truncated to 100% to reduce any potential bias. However, applicable guideline, es/er/ms-5 states "...recoveries greater than expected (> 100%) are indicative of instrumental problems or contamination, as carriers fortified into samples are not expected to be recovered at levels greater than spiked." Total β Sr was therefore qualified as estimated (J) in these two samples.

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

Summary

- Ra-226 results for all samples were qualified as estimated (J) in because Ra-226 was detected in the method blank and the normalized difference for each sample was < 2.58.
- Th-230 results for all samples were qualified as estimated (J) in because Th-230 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 samples because the Th-230 duplicate RER <1.
- U-238 results for all samples were qualified estimated (J) because the duplicate U-238 RER <1.
- The total β Sr results for samples YMTFA72C and YMTFA65C were qualified estimated (J) because the carrier > the 110% QC limit.
- PCB-1260 and Total PCBs were qualified estimated (J) for sample YMTFA56C because the intercolumn RPD >40%.

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
YMTFA72C	Ra-226	0.402	0.402	pCi/g		J
YMTFA72C	Th-230	0.0741	0.0741	pCi/g		J
YMTFA72C	U-238	0.108	0.108	pCi/g		J
YMTFA72C	Total β Sr	0.240	0.240	pCi/g		J
YMTFA65C	Ra-226	0.392	0.392	pCi/g		J
YMTFA65C	Th-230	0.102	0.102	pCi/g		J
YMTFA65C	U-238	0.197	0.197	pCi/g		J
YMTFA65C	Total β Sr	0.162	0.162	pCi/g		J
YMTFA58C	Ra-226	0.411	0.411	pCi/g		J
YMTFA58C	Th-230	0.187	0.187	pCi/g		J
YMTFA58C	U-238	0.209	0.209	pCi/g		J
YMTFA57C	Ra-226	0.469	0.469	pCi/g		J
YMTFA57C	Th-230	0.0983	0.0983	pCi/g		J
YMTFA57C	U-238	0.0772	0.0772	pCi/g		J
YMTFA56C	PCB-1260	0.027	0.027	mg/K g	J	J
YMTFA56C	Total PCBs	0.027	0.027	mg/K g	J	J
YMTFA56C	Ra-226	0.557	0.557	pCi/g		J
YMTFA56C	Th-230	0.135	0.135	pCi/g		J
YMTFA56C	U-238	0.0910	0.0910	pCi/g		J
YMTFA55C	Ra-226	0.691	0.691	pCi/g		J
YMTFA55C	Th-230	0.159	0.159	pCi/g		J
YMTFA55C	U-238	0.0209	0.0209	pCi/g		J

Appendix A
Verification Summary Tables

Data Verification	Y	N	N/A	Comment
Custody of Samples				
Are samples traceable through inspection of signature records on field and laboratory chains of custody (COCs)?	Y			COC No. 160-4416-2171.2; COC 160-91913.2 for lab to lab sample transportation. Custody seals were present at receipt on the cooler received on the field but not the container used for lab-to-lab transfer.
Has contractual turn-around time been met for all samples?			N/A	Samples rec'd by lab on 8/16/16 and reported on 10/5/2016. Contractual TAT for lab was not available to the validator.
Have all samples been preserved correctly and pertinent documentation included?	Y			Samples received at 0.4°C.
Is the laboratory log in sample receipt checklist present	Y			
Are any sample receipt non-conformances noted?			N/A	
Standard Traceability				
Have certificate(s) been included for the LCS and MS?	Y			
Standards have not exceeded the certificate expiration date	Y			
Are chemical standards and reference materials traceable to a reliable source? (Reagent traceability summary)	Y			
Analytical Completeness				
Are all COC samples and associated analytical results reported in the laboratory data package?		N		Two expansion joint samples, YMTFA53EJ and YMTFA52EJ, are listed on the COC. The validator confirmed with management that these two samples were reported in a different SDG, so there is no impact on completeness.
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 does not identify all issues. See validation checklists and DVR for details.

Data Verification	Y	N	N/A	Comment
Other data summary forms are present as applicable (detection, sample results, surrogate, tracer/carrier, QC results and association, prep and analysis chronicle, method and sample summaries)	Y			
Sample Data				
Is the Sample Data included for each COC requested analytical method?	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?	Y				Samples received at 0.4°C.
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			
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				

Metals by ICP (SW6010)	Y	N	N/A	Qualifier	Comment or Reason Code
Mercury by CVAA (SW7470A)					
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		
Serial Dilution					
Was the Serial Dilution within acceptance limits?			N/A		SD on project sample was NC due to low levels and nondetects. SD was qualitatively acceptable.
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				Yes. The case narrative noted that samples were diluted during

Metals by ICP (SW6010) Mercury by CVAA (SW7470A)	Y	N	N/A	Qualifier	Comment or Reason Code
					TCLP prep due to the nature of the sample matrix, and that samples were high in salts. There were no resulting data issues.

Polychlorinated Biphenyl	Y	N	N/A	Qualifier	Comment or Reason Code
Preservation and Holding Times					
Were samples properly preserved?	Y				Samples received at 0.4°C.
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 on multiple ICV peaks were slightly > 20% for multiple peaks. In all cases, minimum # acceptable peaks (3) were available.
Calibration Verification					
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. In all but one CCV, minimum # acceptable peaks (3) were available. However, PCB-1254 in two separate calibrations had %D at 98.1 and 96.9%. PCB-1254 was not detected in any samples so no qualifications were assigned. However, this should be noted in the DVR
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				

Polychlorinated Biphenyl	Y	N	N/A	Qualifier	Comment or Reason Code
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?	Y				Surrogate out high in one CCV, but okay in all samples.
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 with sample counts up to 20 samples?	Y				MS/MSD run on sample not in this SDG.
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				
Target Analyte Identification					
Do the positively identified compounds meet the identification criteria?		N			PCB-1260 and Total PCBs qualified J in sample YMTFA56C for intercolumn RPD >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?	Y				Samples were received at 0.4°C in bags.
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.
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 resolutions within control criteria?	Y				
LCS					
Has at least one LCS been prepared for up to 20 samples?	Y				
Is the LCS the same matrix as the samples in the reporting batch?	Y				
Are LCS %D (or %R) within QC acceptance limit?	Y				
Laboratory Duplicate					
Has at least one laboratory duplicate been prepared for up to 20 samples?	Y				
Are RPD and DER within QC acceptance limit?		N			RER >1 for Th-230 at 2.5 and U-238 at 1.6. Neptunium-237 RER also >1 at 2 but both results were U, so these are considered to be

Radionuclide Analyses: Alpha Spectrometry Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
					comparable. J qualifiers assigned to all samples for Th-230 and U-238 results.
Matrix Spike					
Has at least one MS been prepared for up to 20 samples?		N			MS for Tritium only; Tritium MS acceptable. Project sample.
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			All blank results ND except Th-230 and Ra-226. The normalized difference was calculated for all samples and determined to be < 2.58 in all samples for both parameters. Ra-226 and Th-230 results for all samples were qualified J. See table inserted after checklist for values.
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			For Total β Strontium, the narrative noted samples YMTFA72C and YMTFA65C had carrier recoveries above the 110% QC

Radionuclide Analyses: Alpha Spectrometry Gas Flow Proportional Counting Liquid Scintillation Counting	Y	N	N/A	Qualifier	Comment or Reason Code
					<p>limit due to matrix interferences, noting the LCS had an acceptable spike recovery demonstrating acceptable sample preparation and instrument performance. The lab narrative further noted that the samples were truncated to 100% to reduce any potential bias.</p> <p>Per es/er/ms-5 “...recoveries greater than expected (> 100%) are indicative of instrumental problems or contamination, as carriers fortified into samples are not expected to be recovered at levels greater than spiked.”</p> <p>The Sr(C) recoveries in these samples were 120 and 113, respectively (limit 40-110).</p> <p>The strontium detections in these two samples were qualified as J.</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.

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

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	Th-230	pCi/g	0.0537	0.055		
YMTFA72 C	Th-230	pCi/g	0.0741	0.057	0.257547839	J
YMTFA65 C	Th-230	pCi/g	0.102	0.086	0.473143015	J
YMTFA58 C	Th-230	pCi/g	0.187	0.1	1.167995806	J
YMTFA57 C	Th-230	pCi/g	0.0983	0.073	0.487963821	J
YMTFA56 C	Th-230	pCi/g	0.135	0.092	0.758489252	J
YMTFA55 C	Th-230	pCi/g	0.159	0.092	0.982397518	J
Blank	Ra-226	pCi/g	0.2916	0.104		
YMTFA72 C	Ra-226	pCi/g	0.402	0.102	0.75787291	J
YMTFA65 C	Ra-226	pCi/g	0.392	0.104	0.682630008	J
YMTFA58 C	Ra-226	pCi/g	0.411	0.0966	0.841188057	J
YMTFA57 C	Ra-226	pCi/g	0.469	0.112	1.160692548	J
YMTFA56 C	Ra-226	pCi/g	0.557	0.123	1.647684594	J
YMTFA55 C	Ra-226	pCi/g	0.691	0.147	2.218033331	J

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				

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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		

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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				