

Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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Results for the First, Second, and Third Quarter Calendar Year 2015 Tank 50H WAC Slurry Samples

Chemical and Radionuclide Contaminants

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February 2016

SRNL-STI-2015-00313, Rev. 0

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Printed in the United States of America

**Prepared for
U.S. Department of Energy**

Keywords: *Tank 50, Waste Acceptance
Criteria, Saltstone*

Retention: *Permanent*

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Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



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EXECUTIVE SUMMARY

This report details the chemical and radionuclide contaminant results for the characterization of the Calendar Year (CY) 2015 First, Second, and Third Quarter sampling of Tank 50H for the Saltstone Waste Acceptance Criteria (WAC) in effect at that time.^{1,2} Information from this characterization will be used by Defense Waste Processing Facility (DWPF) & Saltstone Facility Engineering (D&S-FE) to support the transfer of low-level aqueous waste from Tank 50H to the Salt Feed Tank in the Saltstone Facility in Z-Area, where the waste will be immobilized. This information is also used to update the Tank 50H Waste Characterization System. Previous memoranda documenting the WAC analyses results have been issued for these three samples.^{3,4,5}

The following conclusions are drawn from the analytical results pertaining to the WAC provided in this report for the First, Second, and Third Quarter CY 2015 (1QCY15, 2QCY15, and 3QCY15) samples:

- SRR WAC targets or limits were met for all analyzed chemical and radioactive contaminants unless noted in this section.
- Of the forty-seven metal, anion, and organic contaminants listed in Attachment 8.1 and 8.2 of the WAC for 1QCY15 and 2QCY15 samples, six of the detectable species are above 10% of the WAC limit or target.¹ These contaminants are carbonate, total base (OH⁻), nitrate, nitrite, sulfate (2QCY15 only) and total mercury. For the 3QCY15 sample of the fifty metal, anion, and organic contaminants listed in Attachment 8.1 and 8.2 for the WAC, eight of the detectable species are above 10% of the WAC limit or target.² These contaminants are carbonate, total base (OH⁻), nitrate, nitrite, sulfate, total mercury, monomethyl mercury and dimethyl mercury.
- Of the fifty-five radionuclides contaminants listed in Attachment 8.3 and 8.4 of the WAC for 1QCY15 and 2QCY15 samples, four are above 10% of the WAC limit or target.¹ These radionuclide contaminants are ⁹⁹Tc, ¹²⁹I, ¹³⁷Cs, and ^{137m}Ba. Of the fifty-five radionuclides contaminants listed in Attachment 8.3 and 8.4 of the WAC for 3QCY15 samples, three are above 10% of the WAC limit or target.² These radionuclide contaminants are ¹²⁹I, ¹³⁷Cs, and ^{137m}Ba.
- NORPAR 13 and Isopar L have higher detection limits⁶ compared with the Saltstone WAC target (for NORPAR 13) and limit (for Isopar L).^{1,2} The data provided in this report are based upon concentrations in the sub-sample, and due to the limited solubility of these materials in aqueous solution, may not represent the concentrations of the analytes in Tank 50H.
- All other organic species listed in Attachment 8.1 and 8.2 of the WAC for 1QCY15, 2QCY15, and 3QCY15 are below the method detection limits, except for total organic carbon which is detected at levels (304 mg/L to 315 mg/L) that are below 10% of the WAC limit of 5,000 mg/L.^{1,2}

Additional conclusions are:

- Certain constituents requested in the Task Technical Request pertaining to Toxicity Characteristic Leaching Procedure (TCLP) and Underlying Hazardous Constituents (UHC) such as antimony, beryllium and thallium were not detected. The total beta and total solids are in the range of 6.53E+05 pCi/mL to 7.97E+05 pCi/mL and 27.10 wt% to 27.87 wt%, respectively.
- The specific gravity and total gamma activity are reported in the range of 1.2347 to 1.2362 and 5.19E+05 pCi/mL to 8.00E+05 pCi/mL, respectively.
- Minimum detection limits are reported for ^{59}Ni , ^{94}Nb , ^{247}Cm , ^{249}Cf , and ^{251}Cf as determined from the minimum detectable activity associated with the radiochemical methods used for these radionuclides for 1QCY15, 2QCY15, and 3QCY15 samples. The reported detection limits are above the requested SRR target minimum detection limit concentrations except for the 2QCY15 and 3QCY15 ^{59}Ni minimum detection limits which are below the SRR target minimum.⁷ However, the reported values for the radionuclides listed above, are below the estimated detection limits initially established by SRNL in 2009, except for the 2QCY15 ^{94}Nb value which is ~ 6% above the estimated detection limit set by SRNL in 2009.⁸

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LIST OF ABBREVIATIONS

AA	Atomic Absorption (spectroscopy)
AD	Analytical Development
ARP/MCU	Actinide Removal Process/Modular CSSX Unit
CLFL	Composite Lower Flammability Limit
CSSX	Caustic Side Solvent Extraction
CVAA	Cold Vapor Atomic Absorption
D&S-FE	DWPF & Saltstone Facility Engineering
DWPF	Defense Waste Processing Facility
E&CPT	Environmental & Chemical Process Technology
EDTA	Ethylenediaminetetraacetate
ETP	Effluent Treatment Project
GC/MS	Gas Chromatograph/Mass Spectrometer
HDPE	High Density Polyethylene
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICP-ES	Inductively Coupled Plasma – Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
L	Liter
LLW	Low Level Waste
LSC	Liquid Scintillation Counting
MRL	Method Reporting Limit
mg	Milligram
mL	Milliliter
NA	Not Applicable
pCi/mL	Picocurie per Milliliter
PHA	Pulse Height Analysis
RSD	Relative Standard Deviation
SC	Shielded Cells (Facility)
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SVOA	Semi-Volatile Organic Analysis
TCLP/UHC	Toxicity Characterization Leaching Procedure/Underlying Hazardous Constituent
TIC/TOC	Total Inorganic Carbon/Total Organic Carbon

TPB	Tetraphenylborate
TTR	Technical Task Request
UHC	Underlying Hazardous Constituent
VDS	Variable Depth Sample
VOA	Volatile Organic Analysis
WAC	Waste Acceptance Criteria
WT %	Weight Percent

1.0 Introduction

The Saltstone Facility is designed and permitted to treat low-level radioactive and hazardous liquid waste (salt solution) remaining from the processing of radioactive material at the Savannah River Site. Low-level waste (LLW) streams from the Effluent Treatment Project (ETP), H-Canyon, and the decontaminated salt solution product from the Actinide Removal Process/Modular Caustic Side Solvent Extraction (CSSX) Unit (ARP/MCU) process are stored in Tank 50H until the LLW can be transferred to the Saltstone Facility for treatment and disposal. The LLW must meet the specified Waste Acceptance Criteria (WAC) before it is processed into saltstone.^{1,2} The specific chemical and radionuclide contaminants and their respective WAC limits are in the current^a Saltstone WAC. Revision 13 of the WAC¹ applied to both First and Second Quarter Calendar Year 2015 samples and Revision 14 of the WAC² applied to the Third Quarter Calendar Year 2015 sample.

DWPF Saltstone Facility Engineering (D&S-FE) requested that the Savannah River National Laboratory (SRNL) perform quarterly analysis on Tank 50H salt solution feed samples per a Task Technical Request (TTR).⁹ The concentrations of chemical and radionuclide contaminants are measured to ensure the saltstone produced during each quarter complies with the current WAC.^{1,2,9,10} This report documents the concentrations of chemical and radionuclide contaminants and discusses those results for the First, Second and Third Quarter Calendar Year 2015 (1QCY15, 2QCY15, and 3QCY15) samples collected from Tank 50H on January 20, 2015, April 7, 2015 and July 9, 2015, respectively. In addition to the chemical and radionuclide contaminants associated with the WAC, other species in the quarterly Tank 50H sample are reported as requested by D&S-FE.⁷ These include constituents pertinent to the Toxicity Characteristic Leaching Procedure (TCLP) / Underlying Hazardous Constituents (UHC) and corrosion control and radionuclides associated with inventory reporting requirements.

2.0 Experimental

2.1 Technical

On January 20, 2015, a 1-L sampler (HTF-50-15-4) and a 200-mL sampler (HTF-50-15-3) were collected from Tank 50H for the 1QCY15 WAC analyses and delivered the same day to the SRNL Shielded Cells (SC).¹¹ The 200 mL sampler is a dip sample taken six inches below the surface and the 1-L variable depth sample (VDS) was pulled 35 inches from the bottom of the tank after running one agitator pump for at least 4.4 hours prior to pump shutdown and sampling. The VDS was left at depth for at least one hour for foil dissolution. On April 7, 2015, a 1-L sampler (HTF-50-15-39) and a 200-mL sampler (HTF-50-15-38) were collected from Tank 50H for the 2QCY15 WAC analyses and delivered the same day to the SRNL SC.¹² On July 9, 2015, a 1-L sampler (HTF-50-15-92) and a 200-mL sampler (HTF-50-15-91) were collected from Tank 50H for the 3QCY15 WAC analyses and delivered the same day to the SRNL SC.¹³

All of the Tank 50H WAC quarterly samples were processed in the same way. At SRNL, slurry samples from the 200-mL sampler were transferred with glass pipettes to glass vials with Teflon[®]-lined caps. The vials were completely filled to minimize the void space and the volatilization of organics. The aliquots were transferred to the Analytical Development (AD) Organic Analysis Laboratory for semi-volatile and volatile organic analysis (SVOA and VOA, respectively). Two additional aliquots were used for SVOA analysis to determine the concentration of Isopar L and NORPAR 13, respectively, in the sample.

^a This report references the Saltstone WAC revision in effect when the sample is pulled for analysis and/or that which was referenced during initial data reporting. This may or may not be the latest revision when this report receives its final approval signature.

After the samples for organic analyses were obtained, the remaining slurry in the 200-mL sampler was agitated to disperse any solids and poured into a 125-mL Teflon[®] bottle and set aside. The 1-L VDS sample was transferred into a 2-L high-density polyethylene (HDPE) bottle. The transferred slurry was left to settle in the 2-L bottle. No solids were observed after being allowed to settle overnight, but traces of solids were visible after sitting undisturbed for several days. Visual inspection of the inside of the sampler indicated there were no visible solids remaining in the sampler, thus no clear supernate was returned to the sampler for rinsing. The total weight of the transferred slurry was 1,490 g, 1,433 g, and 1,464 g, respectively for the 1QCY15, 2QCY15, and 3QCY15 samples.

The 2-L HDPE bottle was agitated to thoroughly disperse the limited suspended solids into the supernate. Aliquots of slurry samples were then immediately collected with slurry pipettes to minimize settling effects and placed into HDPE bottles

Slurry samples were submitted in triplicate to SRNL for the following analyses:

- Six-mL aliquots to the AD Ion Chromatography (IC) Laboratory for soluble anion and cation analyses.
- Three-mL aliquots to the AD Organic Analysis Laboratory for measurement of tetraphenylborate (TPB) and ethylenediaminetetraacetate (EDTA) by High Performance Liquid Chromatography (HPLC).
- Six-mL aliquots to the AD Wet Chemistry Laboratory for Total Inorganic Carbon/Total Organic Carbon (TIC/TOC) analyses.
- Three sets of 70-mL aliquots to AD Radiochemistry Laboratory for radiochemical separations and analyses.
- Twelve-mL aliquots of filtered supernate were prepared by filtering aliquots of supernate using a 0.45 micron syringe filter. The filtered supernate samples were then submitted to the AD Wet Chemistry Laboratory for TIC/TOC analyses and Total Base analyses.
- Twelve-mL aliquots were sent to the AD Dissolution Laboratory for digestion using an aqua regia method.¹⁴ Visual inspection of the digested sample by the AD Task Supervisor indicated that all the solids had dissolved. Aliquots of dissolved slurries were analyzed using Inductively Coupled Plasma – Emission Spectroscopy (ICP-ES), Inductively Coupled Plasma – Mass Spectrometry (ICP-MS), Atomic Absorption spectroscopy (AA) for As, K, Na, and Se, and Cold Vapor Atomic Absorption spectroscopy (CVAA) for total Hg.
- Quadruplicate three-gram samples of both slurry and filtrate were used to determine the quantitative solids by remote handling/drying in the SRNL SC. A 15 WT% NaCl standard solution was performed in parallel. Data from the slurry and filtrate solids measurements were used to calculate the total insoluble solids. These measurements and resulting calculations were performed per Environmental and Chemical Process Technology (E&CPT) procedure L29, ITS-0078, Rev. 1, ‘Weight Percent Solids Determination Using a Furnace or Oven’.¹⁵

A known mass of approximately 150 mL of the Tank 50H slurry was transferred to a radiochemical hood for preparation of a saltstone sample used for TCLP/UHC testing.

2.2 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. Data collected during this study are recorded in SRNL Electronic Notebooks.^{11,12,13}

3.0 Results and Discussion

3.1 First and Second Quarter Calendar Year 2015 WAC Samples

The following tables contain the results for the 1QCY15 and the 2QCY15 WAC analyses that pertain to Revision 13 of the WAC.¹ Results for the 3QCY15 WAC analyses that pertain to Revision 14 of the WAC² will then be presented after the 1QCY15 and 2QCY15 results. Each table provides the analyte of interest, the method used for measuring that analyte, the average concentration of the analyte based on triplicate samples (unless otherwise noted), and the standard deviation of the average. The WAC target or limit for the analyte concentration is shown in the final column of these tables. These WAC targets or limits apply to both 1QCY15 and the 2QCY15 WAC analyses presented in Tables 3-1 through 3-5.¹ Several of the contaminants were either not detected in the slurry samples or detected at values below the Method Reporting Limit (MRL). For those analytes, the result is preceded by a “<” which indicates the result is an upper limit based on the sensitivity of the method/equipment used to analyze the individual analyte. The standard deviation for these analytes cannot be calculated and is noted as not applicable (NA). Some of the contaminants shown in the following tables are the result of only a single detectable value (out of the triplicate analysis). In these cases the standard deviation cannot be calculated and is noted as NA. Tables 3-1, 3-2, 3-3, and 3-4 are based directly on Attachments 8.1, 8.2, 8.3, and 8.4, respectively, of the WAC.¹

The analytical methods shown in Table 3-1 and Table 3-2 include analyses (IC, TIC, Total Base, VOA, SVOA, TOC and HPLC) in which Tank 50H supernate or filtrate are directly analyzed with no preparation or digestion of the sample. These methods typically report the same MRL over time, i.e., from quarter to quarter. Other methods listed in these two tables (ICP-ES, AA, ICP-MS and CVAA) involve aqua regia digestion of nominally 15 g of supernate. Since the exact mass of supernate used for each triplicate digestion is different, and the sample density is different from each quarter (the density is used to correct reported results in mass analyte per mass supernate over to mass analyte per liter (mg/L)), these methods report different “<” values for analytes that are below the MRL. The radiochemical methods shown in Table 3-3 and Table 3-4 are predominately separations and counting techniques. One noted exception is gamma spectroscopy in which the supernate is analyzed as received. Some of the radionuclides shown in these two tables are derived from ICP-MS using known specific activities (Curies/gram) for these particular radionuclides.¹⁶ Since the radiochemical separation methods involve varying separation efficiencies from analysis to analysis, and the overall radioactivity of each quarterly sample is different, these techniques typically report different “<” values for analytes that are below the MRL.

Table 3-1. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Chemical Results and WAC Limits¹

Chemical Name (Formula)	Method	Average Concentration (1Q / 2Q) (mg/L)	Std. Dev. (1Q / 2Q)	WAC Limit (mg/L)
Aluminate ($\text{Al}(\text{OH})_4^-$)	ICP-ES	1.18E+04 ^a / 1.08E+04 ^a	1.74E+02 / 5.02E+01	4.08E+05
Ammonium (NH_4^+)	IC	< 1.00E+02 / < 1.00E+02	NA / NA	2.12E+02
Carbonate (CO_3^{2-})	TIC	1.57E+04 ^b / 1.54E+04 ^b	2.88E+01 / 5.77E+01	1.20E+05
Chloride (Cl^-)	IC	1.93E+02 / 2.07E+02	1.53E+00 / 1.53E+00	7.95E+03
Fluoride (F^-)	IC	< 1.00E+02 / < 1.00E+02	NA / NA	4.07E+03
Free Hydroxide (OH^-)	Total Base	2.78E+04 ^b / 2.66E+04 ^b	2.60E+02 / 5.20E+02	1.58E+05
Nitrate (NO_3^-)	IC	1.24E+05 / 1.15E+05	1.15E+03 / 5.77E+02	4.37E+05
Nitrite (NO_2^-)	IC	2.37E+04 / 2.44E+04	1.00E+02 / 2.08E+02	2.14E+05
Oxalate ($\text{C}_2\text{O}_4^{2-}$)	IC	3.54E+02 / 3.62E+02	6.93E+00 / 8.02E+00	2.72E+04
Phosphate (PO_4^{3-})	ICP-ES	4.43E+02 ^c / 4.98E+02 ^c	2.18E+01 / 1.53E+01	2.94E+04
Sulfate (SO_4^{2-})	IC	5.62E+03 / 5.96E+03	1.70E+02 / 1.73E+02	5.69E+04
Arsenic (As)	AA	< 1.13E-01 / < 1.10E-01	NA / NA	2.30E+01
Barium (Ba)	ICP-ES	< 1.09E+00 / < 1.06E+00	NA / NA	6.19E+02
Cadmium (Cd)	ICP-ES	< 2.09E+00 / < 2.04E+00	NA / NA	3.10E+02
Chromium (Cr)	ICP-ES	3.21E+01 / 3.51E+01	7.43E-01 / 4.26E+00	1.24E+03
Lead (Pb)	ICP-MS	6.09E-02 / 1.63E-01	2.12E-02 / 1.33E-02	6.19E+02
Total Mercury (Hg)	CVAA	9.97E+01 / 1.18E+02	2.16E+00 / 1.25E+01	3.25E+02
Selenium (Se)	AA	< 2.27E-01 / 4.87E-02 ^e	NA / 3.75E-03	4.46E+02
Silver (Ag)	ICP-ES	< 2.41E+00 / < 2.36E+00	NA / NA	6.19E+02
Aluminum (Al)	ICP-ES	3.34E+03 / 3.08E+03	4.93E+01 / 1.42E+01	1.16E+05
Potassium (K)	AA	1.01E+02 / 2.91E+02	2.75E+00 / 0.00E+00 ^g	3.03E+04
Nickel Hydroxide ($\text{Ni}(\text{OH})_2$)	ICP-ES	< 1.28E+01 ^d / < 1.25E+01 ^d	NA / NA	1.17E+03
n-Butanol ($\text{C}_4\text{H}_9\text{OH}$)	VOA	< 5.00E-01 ^e / < 5.00E-01 ^e	NA / NA	7.73E+00
i-Butanol ($\text{C}_4\text{H}_9\text{OH}$)	VOA	< 5.00E-01 ^e / < 5.00E-01 ^e	NA / NA	7.73E+00
i-Propanol ($\text{C}_3\text{H}_7\text{OH}$)	VOA	< 2.50E-01 ^e / < 2.50E-01 ^e	NA / NA	1.88E+00
Phenol ($\text{C}_6\text{H}_5\text{OH}$)	SVOA	< 1.00E+01 ^e / < 1.00E+01 ^e	NA / NA	7.50E+02
Isopar L (----)	SVOA	< 2.68E+01 ppm ^{e,f} / < 2.68E+01 ppm ^{e,f}	NA / NA	1.10E+01 ppm
Total Organic Carbon (----)	TOC	3.04E+02 ^b / 3.14E+02 ^b	4.04E+00 / 0.00E+00 ^g	5.00E+03
Tetraphenylborate [TPB anion] ($\text{B}(\text{C}_6\text{H}_5)_4^-$)	HPLC	< 5.00E+00 / < 5.00E+00	NA / NA	5.00E+00

a. Result is calculated from the measured Al concentration assuming all of the Al is present as the hydroxide compound.

b. Measurement performed on filtered supernate samples.

c. Result is calculated from the measured P concentration assuming all of the P is present as the oxide compound.

d. Result is calculated from the measured Ni concentration assuming all of the Ni is present as the hydroxide compound.

e. Measurement performed on duplicate samples rather than triplicate samples.

f. Result is calculated from the reported concentration of < 33 mg/L and the density in Table 3-8.

g. All replicates showed identical concentrations.

Table 3-2. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Chemical Results and WAC Targets¹

Chemical Name (Formula)	Method	Average Concentration (1Q / 2Q) (mg/L)	Std. Dev. (1Q / 2Q)	WAC Target (mg/L)
Boron (B)	ICP-ES	4.21E+01 / 4.57E+01	5.82E-01 / 7.12E-02	7.43E+02
Cobalt (Co)	ICP-MS	< 2.06E-02 / < 2.01E-02	NA / NA	1.75E+02
Copper (Cu)	ICP-ES	< 1.32E+00 / < 1.28E+00	NA / NA	7.43E+02
Iron (Fe)	ICP-ES	5.14E+00 / 2.04E+01	1.11E-01 / 1.62E+01	4.95E+03
Lithium (Li)	ICP-ES	1.57E+01 / 1.53E+01	3.10E-01 / 3.97E-01	7.43E+02
Manganese (Mn)	ICP-ES	1.73E+00 / 1.86E+00	9.96E-02 / 7.38E-01	7.43E+02
Molybdenum (Mo)	ICP-ES	< 4.57E+00 / < 4.47E+00	NA / NA	7.43E+02
Nickel (Ni)	ICP-ES	< 8.13E+00 / < 7.92E+00	NA / NA	7.43E+02
Silicon (Si)	ICP-ES	3.15E+01 / 3.97E+01	1.26E+00 / 2.24E+00	1.07E+04
Strontium (Sr)	ICP-ES	< 1.97E-01 / < 1.92E-01	NA / NA	7.43E+02
Zinc (Zn)	ICP-ES	1.26E+01 / 5.01E+00	1.42E-01 / 6.41E-02	8.03E+02
Benzene (C ₆ H ₆)	VOA	< 1.50E-01 ^a / < 1.50E-01 ^a	NA / NA	3.10E+02
Methanol (CH ₃ OH)	VOA	b / b	NA / NA	1.88E+00
Dibutylphosphate [DBP] (C ₈ H ₁₈ O ₄ P)	IC	< 2.50E+02 / < 2.50E+02	NA / NA	3.47E+02
Tributyl Phosphate [TBP] ((C ₄ H ₉ O) ₃ PO)	SVOA	< 7.50E-01 ^a / < 7.50E-01 ^a	NA / NA	7.50E+00
Toluene (C ₆ H ₅ CH ₃)	VOA	< 1.50E-01 ^a / < 1.50E-01 ^a	NA / NA	3.10E+02
EDTA (----)	HPLC	< 1.00E+02 / < 1.00E+02	NA / NA	3.10E+02
NORPAR 13 (----)	SVOA	< 7.50E-01 ^a / < 7.50E-01 ^a	NA / NA	1.00E-01

a. Measurement performed on duplicate samples rather than triplicate samples.

b. Currently, a routine method for detecting this species does not exist in AD.

Table 3-3. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Radionuclides Results and WACT Limits¹

Radionuclide	Method	Average Concentration (1Q / 2Q) (pCi/mL)	Std. Dev. (1Q / 2Q)	WAC Limit (pCi/mL)
Tritium (³ H)	Tritium counting	3.72E+02 / 3.60E+02	2.15E+01 / 4.72E+01	5.63E+05
Carbon-14 (¹⁴ C)	C-14 Liquid scintillation	5.86E+02 / 8.03E+02	1.80E+01 / 5.18E+01	1.13E+05
Nickel-63 (⁶³ Ni)	Ni-59/63	< 9.77E+00 / < 3.41E+00	NA / NA	1.13E+05
Strontium-90 (⁹⁰ Sr)	Sr-90 Liquid scintillation	4.95E+03 / 3.49E+03	1.24E+03 / 7.10E+02	3.15E+06
Technetium-99 (⁹⁹ Tc)	Tc-99 Liquid scintillation	1.71E+04 / 1.58E+04	1.53E+03 / 2.74E+02	8.70E+04
Iodine-129 (¹²⁹ I)	I-129 (w/ separation) Liquid scintillation	1.08E+01 / 9.08E+00	3.27E+00 / 1.20E+00	6.30E+01
Cesium-137 (¹³⁷ Cs)	Gamma Scan	8.45E+05 / 6.52E+05	1.13E+04 / 8.33E+04	3.96E+06
Uranium-233 (²³³ U)	ICP-MS	< 1.99E+02 / < 1.94E+02	NA / NA	1.13E+04
Uranium-235 (²³⁵ U)	ICP-MS	2.33E-01 / 2.58E-01	4.76E-03 / 4.22E-03	1.13E+02
Plutonium-241 (²⁴¹ Pu)	Pu238/241 Liquid scintillation	2.65E+02 / 2.40E+02	3.67E+01 / 2.01E+01	8.38E+05
Total Alpha	Liquid Scintillation Counting	< 6.71E+02 / < 3.88E+02	NA / NA	2.13E+05

Table 3-4. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Radionuclide Results and WAC Targets¹

Radionuclide	Method	Average Concentration (1Q / 2Q) (pCi/mL)	Std. Dev. (1Q / 2Q)	WAC Target (pCi/mL)
Aluminum-26 (²⁶ Al)	Gamma scan (Cs removed)	< 1.68E-01 / < 1.77E-01	NA / NA	2.88E+03
Cobalt-60 (⁶⁰ Co)	Gamma scan (Cs removed)	2.98E-01 ^a / 3.45E-01	8.44E-02 / 4.90E-02	9.747E+02
Potassium-40 (⁴⁰ K)	Gamma scan (Cs removed)	< 1.52E+00 / < 1.49E+00	NA / NA	1.00E+02
Nickel-59 (⁵⁹ Ni)	Ni-59/63	< 1.32E+01 / < 6.44E+00	NA / NA	1.13E+03
Selenium-79 (⁷⁹ Se)	Se-79	2.45E+01 / 3.70E+01	8.34E-01 / 8.02E+00	1.90E+04
Yttrium-90 (⁹⁰ Y)	Secular Equilibrium w/ Sr-90	4.95E+03 / 3.49E+03	1.24E+03 / 7.10E+02	3.15E+06
Zirconium-93 (⁹³ Zr)	ICP-MS	< 5.17E+01 / < 5.04E+01	NA / NA	1.00E+05
Niobium-94 (⁹⁴ Nb)	Gamma scan (Cs removed)	< 2.33E-01 / < 4.64E-01	NA / NA	1.53E+02
Rhodium-106 (¹⁰⁶ Rh)	Secular Equilibrium w/ Ru-106	< 3.77E+00 / < 3.43E+00	NA / NA	1.13E+06
Ruthenium-106 (¹⁰⁶ Ru)	Gamma scan (Cs removed)	< 3.77E+00 / < 3.43E+00	NA / NA	1.13E+06
Antimony-125 (¹²⁵ Sb)	Gamma scan (Cs removed)	7.34E+00 / 6.64E+00	2.74E-01 / 3.64E-01	7.988E+03
Tellurium-125m (^{125m} Te)	Secular Equilibrium w/ Sb-125	7.34E+00 / 6.64E+00	2.74E-01 / 3.64E-01	1.828E+03
Tin-126 (¹²⁶ Sn)	Gamma scan (Cs removed)	1.26E+02 / 8.70E+01	9.25E+00 / 4.44E+01	1.80E+04
Cesium-134 (¹³⁴ Cs)	Gamma Scan	< 3.13E+01 / < 6.58E+01	NA / NA	1.82E+04
Cesium-135 (¹³⁵ Cs)	ICP-MS	< 2.37E+01 / < 4.62E+01	NA / NA	2.50E+02
Barium-137m (^{137m} Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137)	8.00E+05 / 6.16E+05	1.07E+04 / 7.88E+04	3.75E+06
Cerium-144 (¹⁴⁴ Ce)	Gamma scan (Cs removed)	< 4.09E+00 / < 3.79E+00	NA / NA	1.13E+05
Promethium-147 (¹⁴⁷ Pm)	Pm-147/Sm-151 Liquid scintillation	< 3.02E+01 / < 6.58E+01	NA / NA	5.63E+06
Samarium-151 (¹⁵¹ Sm)	Pm-147/Sm-151 Liquid scintillation	< 3.59E+01 / < 6.44E+01	NA / NA	2.25E+04
Europium-154 (¹⁵⁴ Eu)	Gamma scan (Cs removed)	1.45E+00 / 1.40E+00	1.93E-01 / 3.25E-01	1.615E+03
Europium-155 (¹⁵⁵ Eu)	Gamma scan (Cs removed)	< 1.08E+00 / < 2.18E+00	NA / NA	1.13E+04
Radium-226 (²²⁶ Ra)	Ra-226	< 5.77E+00 / < 3.51E+00	NA / NA	1.00E+03
Radium-228 (²²⁸ Ra)	Gamma scan (Cs removed)	< 1.83E+00 / < 1.77E+00	NA / NA	1.00E+04
Actinium-227 (²²⁷ Ac)	Th-229/230	< 1.60E-02 / < 4.30E-02	NA / NA	1.00E+04
Thorium-229 (²²⁹ Th)	Th-229/230	< 1.87E-02 / < 3.56E-02	NA / NA	1.63E+05
Thorium-230 (²³⁰ Th)	Th-229/230	2.65E-02 ^b / < 3.89E-02	NA / NA	6.26E+03
Thorium-232 (²³² Th)	ICP-MS	< 2.26E-03 / 1.21E-02 ^a	NA / 6.12E-03	2.88E+03
Protactinium-231 (²³¹ Pa)	Pa-231	< 1.18E+00 / < 8.74E-01	NA / NA	1.00E+03
Uranium-232 (²³² U)	U-232	3.43E+00 ^a / 2.00E+00	8.70E-01 / 2.10E-01	9.06E+03
Uranium-234 (²³⁴ U)	ICP-MS	< 1.29E+02 / < 1.25E+02	NA / NA	1.13E+04
Uranium-236 (²³⁶ U)	ICP-MS	< 1.33E+00 / < 1.30E+00	NA / NA	1.13E+04
Uranium-238 (²³⁸ U)	ICP-MS	5.33E+00 / 5.88E+00	9.82E-02 / 6.69E-02	1.13E+04
Neptunium-237 (²³⁷ Np)	ICP-MS	< 1.45E+01 / < 1.41E+01	NA / NA	1.00E+04

Table 3-4. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Radionuclide Results and WAC Targets¹, cont.

Radionuclide	Method	Average Concentration (1Q / 2Q) (pCi/mL)	Std. Dev. (1Q / 2Q)	WAC Target (pCi/mL)
Plutonium-238 (²³⁸ Pu)	Pu238/241 Pu alpha PHA	1.00E+03 / 1.21E+03	1.37E+02 / 3.58E+01	2.13E+05
Plutonium-239 (²³⁹ Pu)	Pu238/241 Pu alpha PHA	6.82E+01 / 7.03E+01	1.56E+01 / 1.19E+00	2.13E+05
Plutonium-240 (²⁴⁰ Pu)	Pu238/241 Pu alpha PHA	6.82E+01 / 7.03E+01	1.56E+01 / 1.19E+00	2.13E+05
Plutonium-242 (²⁴² Pu)	ICP-MS	< 7.86E+01 / < 7.66E+01	NA / NA	2.13E+05
Plutonium-244 (²⁴⁴ Pu)	ICP-MS	< 3.65E-01 / < 3.56E-01	NA / NA	7.02E+04
Americium-241 (²⁴¹ Am)	Am/Cm	5.17E+00 / 6.58E+00	1.78E+00 / 1.78E+00	2.13E+05
Americium-242m (^{242m} Am)	Am/Cm	4.82E-02 ^b / < 1.37E-01	NA / NA	4.50E+05
Americium-243 (²⁴³ Am)	Am/Cm	< 4.33E-01 / < 4.64E-01	NA / NA	2.13E+05
Curium-242 (²⁴² Cm)	Am/Cm	3.98E-02 ^b / < 1.13E-01	NA / NA	1.13E+04
Curium-244 (²⁴⁴ Cm)	Am/Cm	2.40E+01 / 5.27E+01	1.55E+01 / 5.73E+01	2.13E+05
Curium-245 (²⁴⁵ Cm)	Am/Cm	< 1.14E+00 / < 1.72E+00	NA / NA	2.25E+05

a. Measurement represents data from duplicate samples rather than triplicate samples.

b. Measurement represents data from single sample rather than triplicate samples.

As shown in Table 3-1 through Table 3-4 all of the contaminants are within the WAC limits or targets with the exception of Isopar L and NORPAR 13.¹ Few contaminants are detected above 10% of the WAC limit or target. Carbonate, total base (OH⁻), nitrate, nitrite, sulfate (2QCY15 only), and total mercury are above 10% of the WAC Limits shown in Table 3-1. The ammonium detection limit of < 100 mg/L is above 10% of the WAC limit of 212 mg/L. All organic species listed in Attachment 8.1 and 8.2 of the WAC for 1QCY15, 2QCY15 are below the method detection limits except for total organic carbon which is detected at levels (304 mg/L to 315 mg/L) below 10% of the WAC limit of 5,000 mg/L. The radionuclides ⁹⁹Tc, ¹²⁹I, ¹³⁷Cs, and ^{137m}Ba are above 10% of the WAC limits and targets shown in Table 3-3 and Table 3-4. Some of the radionuclide concentrations shown in Table 3-4 are the result of only a single detectable value (out of the triplicate analysis). In these cases the standard deviation cannot be calculated and is noted as NA or not applicable. In October 2010, SRNL reviewed the MRLs for the organic constituents in Tank 50H.⁶ All of the MRLs are at or below the WAC targets for the organics with the exception of NORPAR 13 and Isopar L. NORPAR 13 has a MRL of 0.75 mg/L and is above the WAC target.¹ The MRL for Isopar L is < 26.8 ppm and above the WAC limit.¹ Isopar L and NORPAR 13 have negligible solubility in aqueous solutions, which makes it difficult to obtain reliable sub-samples of the original sample. The values reported are the concentrations as detected by the Gas Chromatography/Mass Spectrometry (GC/MS), but may not necessarily be an accurate representation of the actual concentrations of these analytes in Tank 50H.

In a memo from Savannah River Remediation (SRR), the requested detection limits for several radionuclides were lowered in order to accommodate future inventory reporting requirements.⁷ The reported detection limits of ⁵⁹Ni and ⁹⁴Nb shown in Table 3-4 are below the WAC targets. However, they are above the limits requested by SRR (6.59E+00 and 2.00E-03 pCi/mL, respectively) except for the 2QCY15 ⁵⁹Ni value (<6.44E+00 pCi/mL) which is below the SRR requested value.⁷ The reported ⁵⁹Ni detection limits (<1.32E+01 pCi/mL for 1QCY15 and <6.44E+00 pCi/mL for 2QCY15) are below the quantification limit established by SRNL (2.00E+01 pCi/mL).⁸ The ⁹⁴Nb detection limit (<2.33E-01 pCi/mL for 1QCY15) is below the quantification limit established by SRNL (4.38E-01 pCi/mL).⁸ The ⁹⁴Nb detection limit (<4.64E-01 pCi/mL for 2QCY15) is ~6% higher than the estimated SRNL quantification limit⁸ and is below the WAC target.¹

The radionuclide ^{137m}Ba is the radioactive daughter of 94.6% of the beta decay of ^{137}Cs . 5.4% of the ^{137}Cs decays to stable ^{137}Ba . The half-life of the parent radionuclide (^{137}Cs) is six million times longer than its daughter (^{137m}Ba), therefore, the two radionuclides are in secular equilibrium. Radionuclides in secular equilibrium have the same activity associated with their decay. Thus, the activity of ^{137m}Ba is 94.6% of the activity of the ^{137}Cs or $8.00\text{E}+05$ pCi/mL and $6.16\text{E}+05$ pCi/mL for 1QCY15 and 2QCY15, respectively. Other daughter radionuclides in secular equilibrium with their parent radionuclides are also calculated for the WAC analyses. They are parent/daughter: $^{90}\text{Sr}/^{90}\text{Y}$, $^{106}\text{Ru}/^{106}\text{Rh}$, $^{125}\text{Sb}/^{125m}\text{Te}$ and $^{93}\text{Zr}/^{93m}\text{Nb}$.

The concentration of ^{135}Cs is calculated by assigning all of the mass at mass number 135 from the ICP-MS results to cesium. It is assumed all the mass detected at mass 244 is ^{244}Pu . The Pu alpha Pulse Height Analysis (PHA) method does not resolve the alpha activities of ^{239}Pu and ^{240}Pu . To determine the maximum concentration of each radionuclide, the total activity is assigned to each radionuclide separately. As shown in Table 3-4, the reported activity is below the WAC target for each radionuclide.¹

Table 3-5 and Table 3-6 list the chemical contaminants that affect Saltstone Disposal Unit (SDU) flammability. These chemicals must be monitored to ensure flammable gases other than Isopar L, benzene, ammonia, and hydrogen do not contribute more than 10% of the Composite Lower Flammability Limit (CLFL).¹ To address the uncertainty of GC/MS results from earlier quarterly tank analyses, several modifications to SRNL sampling and analysis methodology have been implemented. First, either a second set of surface sample sub-samples are collected prior to combining the surface and dip samples, or alternatively, the entire surface sample is reserved for organic analyses. This additional sample material is held in reserve for later analysis pending the outcome of the first set of samples. For the 1QCY15 sample, a 200 mL surface sample was collected. Approximately 95 g of the surface sample was set aside and not combined with the dip sample. Secondly, the cell prepared blank is pH adjusted (made basic) and processed for analysis in the same manner as the actual tank samples to better detect contaminants introduced to the sample. Finally, the caustic used to adjust the blank pH is analyzed for contaminants.

Table 3-5. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Chemical Results Impacting Saltstone Disposal Unit (SDU) Flammability¹

Chemical Name (Formula)	Method	Average Concentration (1Q/2Q) (mg/L)	Std. Dev. (1Q / 2Q)	WAC Limit
Isopar L (----)	SVOA	< 2.68E+01 ppm ^{a,b} / < 2.68E+01 ppm ^{a,b}	NA / NA	1.10E+01 ppm
Tetraphenylborate [TPB anion] ($\text{B}(\text{C}_6\text{H}_5)_4^-$)	HPLC	< 5.00E+00 / < 5.00E+00	NA / NA	5.00E+00 mg/L
Ammonium (NH_4^+)	IC	< 1.00E+02 / < 1.00E+02	NA / NA	2.12E+02 mg/L

a. Measurement performed on duplicate samples rather than triplicate samples.

b. Result is calculated from the reported concentration of < 33 mg/L and the density in Table 3-8.

Table 3-6. Tank 50H 1QCY15 and 2QCY15 Slurry Sample “Other Organics” Results Impacting SDU Flammability¹

Chemical Name (Formula)	Method	Average Concentration (mg/L)	Std. Dev.	WAC Concentrations (mg/L)
Butanol (C ₄ H ₉ OH)	VOA	< 5.00E-01 / < 5.00E-01	NA / NA	0.75
Tributylphosphate ((C ₄ H ₉ O) ₃ PO)	SVOA	< 7.50E-01 / < 7.50E-01	NA / NA	1.0
Isopropanol(C ₃ H ₇ OH)	VOA	< 2.50E-01 / < 2.50E-01	NA / NA	0.25
Methanol (CH ₃ OH)	a	NA / NA	NA / NA	0.05
NORPAR 13 (----)	SVOA	< 7.50E-01 / < 7.50E-01	NA / NA	0.1

a. Currently, a routine method for detecting this species does not exist in SRNL.

Isopar L and NORPAR 13 are the only species considered in Table 3-5 or Table 3-6 with reported values above the WAC limit.¹ It should be noted that the detection limit for Isopar L was expected based on current SRNL capabilities.⁶ The reported detection limit for NORPAR 13 is above the WAC limit for both accident analysis (Table 3-2) and SDU flammability (Table 3-6), but it is the lowest achievable MRL for this analyte.⁶ As previously discussed, the insolubility of Isopar L and NORPAR 13 makes sub-sampling difficult, therefore, the reported results are not necessarily representative of the concentration of these analytes in the Tank 50H sample received by SRNL.

Table 3-7 provides results for the processing criteria for transfers into the Saltstone Facility. All of the results contained in Table 3-7 fall within the general processing criteria given in Table 5 of the WAC.¹ The pH was calculated using the free base concentration (OH⁻). There are no measurable insoluble solids in the slurry using the method of oven-dried slurry and filtrate at 110°C.¹⁵

Table 3-7. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Results for Saltstone Processing Criteria¹

Processing Criterion	Method	Value (1Q / 2Q)	Std. Dev. (1Q / 2Q)
pH > 10	Calculated	> 13 / > 13	NA / NA
2.5 M < [Na ⁺] < 7.0 M	ICP-ES / AA	4.75 M / 4.90 M	0.233 / 0.0231
Total Insoluble Solids <15 wt%	Calculated	~0 wt% / ~0 wt%	NA / NA

Table 3-8 provides constituents listed in the TTR⁹ but not contained in the WAC. The results from Table 3-8 are used to support the TCLP/UHC testing by a certified laboratory.¹⁷ Natural Tl is composed of two isotopes, ²⁰³Tl and ²⁰⁵Tl with fractional abundances of 0.295 and 0.705, respectively. The concentration of each isotope was divided by its fractional abundance, and the reported concentration of Tl is from the lowest detection limit determined from three replicates for the mass 203 isotope and three replicates for the mass 205 isotope.

Table 3-8. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Requests for Constituents for TCLP/UHC¹⁷

Constituent	Method	Average Value (1Q / 2Q) (mg/L, unless stated otherwise)	Std. Dev. (1Q / 2Q)
Antimony (Sb)	ICP-MS	< 3.60E-02 / < 3.51E-02	NA / NA
Beryllium (Be)	ICP-ES	< 2.30E-01 / < 2.25E-01	NA / NA
Cyanide (CN)	a	NA / NA	NA / NA
Thallium (Tl)	ICP-MS	< 2.92E-02 / < 2.84E-02	NA / NA
Density (slurry)	Measured ^b	1.2320 g/mL / 1.2336 g/mL	0.0006 / 0.0003
Total Beta	LSC	7.97E+05 pCi/mL / 7.88E+05 pCi/mL	2.22E+05 / 2.70E+04
Total Solids	Measured	27.10 wt% / 27.87 wt%	0.042 / 0.338

a. Currently, a routine method for detecting this species does not exist in SRNL.

b. Density for 1QCY15 measured at 21.8 °C; Density for 2QCY15 measured at 20.4 °C.

The tank corrosion species listed in Table 3-9 were requested by D&S-FE.^{11,12} The specific gravity was calculated by dividing the measured density of the slurry given in Table 3-8 by the density of water at the same temperature.¹⁸

Table 3-9. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Requests from the D&S-FE for Corrosion Species^{11,12}

Constituent	Method	Average Value (1Q / 2Q)	Std. Dev. (1Q / 2Q)
Specific Gravity	a	1.2347 / 1.2362	0.0006 / 0.0003
Total Gamma	b	8.00E+05 pCi/mL / 6.17E+05 pCi/mL	6.19E+03 ^c / 4.55E+04 ^c

a. Calculated from the measured density of slurry and density of water at 21.8 °C (1QCY15) and 21.2 °C (2QCY15).¹⁸

b. Calculated from the sum of measured gamma emitters ⁶⁰Co, ¹²⁵Sb, ¹²⁶Sb, ¹²⁶Sn, ^{137m}Ba, ¹⁵⁴Eu, and ²⁴¹Am.

c. Value is the “standard error of the mean” rather than the standard deviation of the measurements since its calculation involves multiple radionuclides.

The activities calculated for total gamma and ^{137m}Ba are expected to be close for this sample because the total gamma activity is dominated by ^{137m}Ba, the radioactive daughter of ¹³⁷Cs. The total gamma activity was calculated by summing the measured gamma activity of the major gamma emitters: ⁶⁰Co, ¹²⁵Sb, ¹²⁶Sb, ¹²⁶Sn, ^{137m}Ba, ¹⁵⁴Eu, and ²⁴¹Am.

Table 3-10 provides results for additional radionuclides required for quantification and support of inventory-reporting requirements as requested by D&S-FE.^{11,12}

Table 3-10. Tank 50H 1QCY15 and 2QCY15 Slurry Sample Radionuclides Results for Inventory Reporting Requirement^{11,12}

Radionuclide	Method	Average Concentration (1Q / 2Q) (pCi/mL)	Std. Dev. (1Q / 2Q)
Niobium-93m (^{93m} Nb)	Calculation (Secular Equilibrium w/ 97.5% of Zr-93)	< 5.04E+01 / < 4.91E+01	NA / NA
Silver-108m (^{108m} Ag)	Gamma scan (Cs removed)	< 6.89E-01 / < 6.40E-01	NA / NA
Barium-133 (¹³³ Ba)	Gamma scan (Cs removed)	< 1.05E+00 / < 9.55E-01	NA / NA
Bismuth-207 (²⁰⁷ Bi)	Gamma scan (Cs removed)	< 5.63E-01 / < 5.45E-01	NA / NA
Thorium-228 (²²⁸ Th)	Gamma scan (Cs removed)	< 1.41E+01 / < 1.31E+01	NA / NA
Curium-247 (²⁴⁷ Cm)	Am/Cm	< 1.75E+00 / < 2.36E+00	NA / NA
Californium-249 (²⁴⁹ Cf)	Am/Cm	< 1.82E+00 / < 2.55E+00	NA / NA
Californium-251 (²⁵¹ Cf)	Am/Cm	< 1.32E+00 / < 1.95E+00	NA / NA

3.2 Third Quarter Calendar Year 2015 WAC Samples

The following tables contain the results for the 3QCY15 WAC analyses that pertain to Revision 14 of the WAC.² Each table provides the analyte of interest, the method used for measuring that analyte, the average concentration of the analyte based on triplicate samples (unless otherwise noted), the standard deviation of the average, and, if applicable, the WAC target or limit for the analyte concentration.² Several of the contaminants were either not detected in the slurry samples or detected at values below the MRL. For those analytes, the result is preceded by a “<” which indicates the result is an upper limit based on the sensitivity of the method/equipment used to analyze the individual analyte. Table 3-11, Table 3-12, Table 3-13, and Table 3-14 are based directly on Attachments 8.1, 8.2, 8.3, and 8.4, respectively, of the WAC.²

Table 3-11. Tank 50H 3QCY15 Slurry Sample Chemical Results and WAC Limits²

Chemical Name (Formula)	Method	Average Concentration (mg/L)	Std. Dev.	WAC Limit (mg/L)
Aluminate ($\text{Al}(\text{OH})_4^-$)	ICP-ES	1.11E+04 ^a	4.34E+01	4.08E+05
Ammonium (NH_4^+)	IC	< 1.00E+02	NA	2.12E+02
Carbonate (CO_3^{2-})	TIC	1.58E+04 ^b	7.63E+01	1.20E+05
Chloride (Cl^-)	IC	2.18E+02	2.65E+00	7.95E+03
Fluoride (F^-)	IC	< 1.00E+02	NA	4.07E+03
Free Hydroxide (OH^-)	Total Base	2.72E+04 ^b	8.50E+02	1.58E+05
Nitrate (NO_3^-)	IC	1.29E+05	4.93E+03	4.37E+05
Nitrite (NO_2^-)	IC	2.55E+04	1.36E+03	2.14E+05
Oxalate ($\text{C}_2\text{O}_4^{2-}$)	IC	4.05E+02	2.33E+01	2.72E+04
Phosphate (PO_4^{3-})	ICP-ES	5.11E+02 ^c	5.78E+00	2.94E+04
Sulfate (SO_4^{2-})	IC	5.88E+03	6.14E+02	5.69E+04
Arsenic (As)	AA	< 1.13E-01	NA	2.30E+01
Barium (Ba)	ICP-ES	< 1.08E+00	NA	6.19E+02
Cadmium (Cd)	ICP-ES	< 2.08E+00	NA	3.10E+02
Chromium (Cr)	ICP-ES	3.26E+01	1.88E-01	1.24E+03
Lead (Pb)	ICP-MS	1.82E-01	9.17E-03	6.19E+02
Total Mercury (Hg)	CVAA	1.02E+02	7.12E-02	3.25E+02
Elemental Mercury	CVAFS	2.20E+00	9.24E-02	8.92E+01
Selenium (Se)	AA	< 2.26E-01	NA	4.46E+02
Silver (Ag)	ICP-ES	< 2.41E+00	NA	6.19E+02
Aluminum (Al)	ICP-ES	3.16E+03	1.23E+01	1.16E+05
Potassium (K)	AA	1.86E+02	2.36E+01	3.03E+04
Nickel Hydroxide ($\text{Ni}(\text{OH})_2$)	ICP-ES	< 1.28E+01 ^d	NA	1.17E+03
n-Butanol ($\text{C}_4\text{H}_9\text{OH}$)	VOA	< 5.00E-01 ^e	NA	7.73E+00
i-Butanol ($\text{C}_4\text{H}_9\text{OH}$)	VOA	< 5.00E-01 ^e	NA	7.73E+00
i-Propanol ($\text{C}_3\text{H}_7\text{OH}$)	VOA	< 2.50E-01 ^e	NA	1.88E+00
Phenol ($\text{C}_6\text{H}_5\text{OH}$)	SVOA	< 1.00E+01 ^e	NA	7.50E+02
Isopar L (----)	SVOA	< 2.68E+01 ppm ^{e,f}	NA	1.10E+01 ppm
Total Organic Carbon (----)	TOC	3.13E+02 ^b	4.36E+00	5.00E+03
Tetraphenylborate [TPB anion] ($\text{B}(\text{C}_6\text{H}_5)_4^-$)	HPLC	< 5.00E+00	NA	5.00E+00
Monomethyl Mercury	CVAFS w/ Distillation	5.33E+01	6.40E+00	3.25E+02

- Result is calculated from the measured Al concentration assuming all of the Al is present as the hydroxide compound.
- Measurement performed on filtered supernate samples.
- Result is calculated from the measured P concentration assuming all of the P is present as the oxide compound.
- Result is calculated from the measured Ni concentration assuming all of the Ni is present as the hydroxide compound.
- Measurement performed on duplicate samples rather than triplicate samples.
- Result is calculated from the reported concentration of < 33 mg/L and the density of the slurry sample listed in Table 3-18.

Table 3-12. Tank 50H 3QCY15 Slurry Sample Chemical Results and WAC Targets²

Chemical Name (Formula)	Method	Average Concentration (mg/L)	Std. Dev.	WAC Target (mg/L)
Boron (B)	ICP-ES	4.24E+01	1.23E-01	7.43E+02
Cobalt (Co)	ICP-MS	< 2.05E-02	NA	1.75E+02
Copper (Cu)	ICP-ES	< 1.31E+00	NA	7.43E+02
Iron (Fe)	ICP-ES	3.55E+00	2.07E-01	4.95E+03
Lithium (Li)	ICP-ES	1.52E+01	7.12E-02	7.43E+02
Manganese (Mn)	ICP-ES	< 1.31E+00	NA	7.43E+02
Molybdenum (Mo)	ICP-ES	< 4.56E+00	NA	7.43E+02
Nickel (Ni)	ICP-ES	< 8.10E+00	NA	7.43E+02
Silicon (Si)	ICP-ES	3.70E+01	1.15E+00	1.07E+04
Strontium (Sr)	ICP-ES	< 1.97E-01	NA	7.43E+02
Zinc (Zn)	ICP-ES	7.29E+00	1.61E-01	8.03E+02
Benzene (C ₆ H ₆)	VOA	< 1.50E-01 ^a	NA	3.10E+02
Methanol (CH ₃ OH)	VOA	b	NA	1.88E+00
Dibutylphosphate [DBP] (C ₈ H ₁₉ O ₄ P)	IC	< 2.50E+02	NA	3.47E+02
Tributyl Phosphate [TBP] ((C ₄ H ₉ O) ₃ PO)	SVOA	< 7.50E-01 ^a	NA	7.50E+00
Toluene (C ₆ H ₅ CH ₃)	VOA	< 1.50E-01 ^a	NA	3.10E+02
EDTA (----)	HPLC	< 1.00E+02	NA	3.10E+02
NORPAR 13 (----)	SVOA	< 7.50E-01 ^a	NA	1.00E-01
Dimethyl Mercury	CVAFS	1.43E-01	1.42E-02	1.00E+00

a. Measurement performed on duplicate samples rather than triplicate samples.

b. Currently, a routine method for detecting this species does not exist in AD.

Table 3-13. Tank 50H 3QCY15 Slurry Sample Radionuclides Results and WAC Limits²

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Limit (pCi/mL)
Tritium (³ H)	Tritium counting	3.47E+02	3.96E+01	5.63E+05
Carbon-14 (¹⁴ C)	C-14 Liquid scintillation	5.32E+02	2.34E+01	1.13E+05
Nickel-63 (⁶³ Ni)	Ni-59/63	< 2.77E+00	NA	1.13E+05
Strontium-90 (⁹⁰ Sr)	Sr-90 Liquid scintillation	2.60E+03	2.68E+02	3.15E+06
Technetium-99 (⁹⁹ Tc)	Tc-99 Liquid scintillation	1.56E+04	2.51E+02	2.11E+05
Iodine-129 (¹²⁹ I)	I-129 (w/ separation) Liquid scintillation	1.28E+01	1.03E+00	6.30E+01
Cesium-137 (¹³⁷ Cs)	Gamma Scan	5.48E+05	1.30E+04	3.96E+06
Uranium-233 (²³³ U)	ICP-MS	< 1.99E+02	NA	1.13E+04
Uranium-235 (²³⁵ U)	ICP-MS	2.50E-01	1.48E-03	1.13E+02
Plutonium-241 (²⁴¹ Pu)	Pu238/241 Liquid scintillation	1.55E+02	2.32E+01	8.38E+05
Total Alpha	Liquid Scintillation Counting	< 3.75E+02	NA	2.13E+05

Table 3-14. Tank 50H 3QCY15 Slurry Sample Radionuclide Results and WAC Targets²

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Target (pCi/mL)
Aluminum-26 (²⁶ Al)	Gamma scan (Cs removed)	< 8.78E-02	NA	2.88E+03
Cobalt-60 (⁶⁰ Co)	Gamma scan (Cs removed)	2.27E-01 ^a	NA	9.747E+02
Potassium-40 (⁴⁰ K)	Gamma scan (Cs removed)	< 1.06E+00	NA	1.00E+02
Nickel-59 (⁵⁹ Ni)	Ni-59/63	< 2.91E+00	NA	1.13E+03
Selenium-79 (⁷⁹ Se)	Se-79	2.80E+01	3.49E+00	1.90E+04
Yttrium-90 (⁹⁰ Y)	Secular Equilibrium w/ Sr-90	2.60E+03	2.68E+02	3.15E+06
Zirconium-93 (⁹³ Zr)	ICP-MS	< 5.15E+01	NA	1.00E+05
Niobium-94 (⁹⁴ Nb)	Gamma scan (Cs removed)	< 2.41E-01	NA	1.53E+02
Rhodium-106 (¹⁰⁶ Rh)	Secular Equilibrium w/ Ru-106	< 2.86E+00	NA	1.13E+06
Ruthenium-106 (¹⁰⁶ Ru)	Gamma scan (Cs removed)	< 2.86E+00	NA	1.13E+06
Antimony-125 (¹²⁵ Sb)	Gamma scan (Cs removed)	7.09E+00 ^b	3.50E-01 ^b	7.988E+03
Tellurium-125m (^{125m} Te)	Secular Equilibrium w/ Sb-125	7.09E+00 ^b	3.50E-01 ^b	1.828E+03
Tin-126 (¹²⁶ Sn)	Gamma scan (Cs removed)	1.20E+02	4.53E+00	1.80E+04
Cesium-134 (¹³⁴ Cs)	Gamma Scan	< 7.61E+01	NA	1.82E+04
Cesium-135 (¹³⁵ Cs)	ICP-MS	< 2.36E+01	NA	2.50E+02
Barium-137m (^{137m} Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137)	5.18E+05	1.23E+04	3.75E+06
Cerium-144 (¹⁴⁴ Ce)	Gamma scan (Cs removed)	< 2.11E+00	NA	1.13E+05
Promethium-147 (¹⁴⁷ Pm)	Pm-147/Sm-151 Liquid scintillation	< 3.59E+01	NA	5.63E+06
Samarium-151 (¹⁵¹ Sm)	Pm-147/Sm-151 Liquid scintillation	< 3.82E+01	NA	2.25E+04
Europium-154 (¹⁵⁴ Eu)	Gamma scan (Cs removed)	5.68E-01 ^a	NA	1.615E+03
Europium-155 (¹⁵⁵ Eu)	Gamma scan (Cs removed)	< 1.18E+00	NA	1.13E+04
Radium-226 (²²⁶ Ra)	Ra-226	< 3.73E+00	NA	1.00E+03
Radium-228 (²²⁸ Ra)	Gamma scan (Cs removed)	< 9.14E-01	NA	1.00E+04
Actinium-227 (²²⁷ Ac)	Th-229/230	6.58E-02 ^a	NA	1.00E+04
Thorium-229 (²²⁹ Th)	Th-229/230	< 1.12E-02	NA	1.63E+05
Thorium-230 (²³⁰ Th)	Th-229/230	< 4.29E-02	NA	6.26E+03
Thorium-232 (²³² Th)	ICP-MS	< 2.25E-03	NA	2.88E+03
Protactinium-231 (²³¹ Pa)	Pa-231	< 6.80E-01	NA	1.00E+03
Uranium-232 (²³² U)	U-232	5.76E+00	2.37E+00	9.06E+03
Uranium-234 (²³⁴ U)	ICP-MS	< 1.28E+02	NA	1.13E+04
Uranium-236 (²³⁶ U)	ICP-MS	< 1.33E+00	NA	1.13E+04
Uranium-238 (²³⁸ U)	ICP-MS	5.65E+00	5.43E-02	1.13E+04
Neptunium-237 (²³⁷ Np)	ICP-MS	< 1.45E+01	NA	1.00E+04

Table 3-14. Tank 50H 3QCY15 Slurry Sample Radionuclide Results and WAC Targets², cont.

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Target (pCi/mL)
Plutonium-238 (²³⁸ Pu)	Pu238/241 Pu alpha PHA	5.75E+02	8.94E+01	2.13E+05
Plutonium-239 (²³⁹ Pu)	Pu238/241 Pu alpha PHA	2.55E+01	3.66E+00	2.13E+05
Plutonium-240 (²⁴⁰ Pu)	Pu238/241 Pu alpha PHA	2.55E+01	3.66E+00	2.13E+05
Plutonium-242 (²⁴² Pu)	ICP-MS	< 7.83E+01	NA	2.13E+05
Plutonium-244 (²⁴⁴ Pu)	ICP-MS	< 3.64E-01	NA	7.02E+04
Americium-241 (²⁴¹ Am)	Am/Cm	< 3.38E+00	NA	2.13E+05
Americium-242m (^{242m} Am)	Am/Cm	< 2.81E-01	NA	4.50E+05
Americium-243 (²⁴³ Am)	Am/Cm	< 6.49E-01	NA	2.13E+05
Curium-242 (²⁴² Cm)	Am/Cm	< 2.32E-01	NA	1.13E+04
Curium-244 (²⁴⁴ Cm)	Am/Cm	3.41E+00	1.65E+00	2.13E+05
Curium-245 (²⁴⁵ Cm)	Am/Cm	< 2.05E+00	NA	2.25E+05

- a. Measurement represents data from single sample rather than triplicate samples.
b. Measurement represents data from duplicate samples rather than triplicate samples.

As shown in Table 3-11 through Table 3-14, all of the contaminants are within the WAC limits or targets with the exception of Isopar L and NORPAR 13.² Few contaminants are above 10% of the WAC limit or target. Carbonate, total base (OH-), nitrate, nitrite, sulfate, total mercury, monomethyl mercury and dimethyl mercury are above 10% of the WAC Limits shown in Table 3-11 and Table 3-12. The radionuclides ¹²⁹I, ¹³⁷Cs and ^{137m}Ba are above 10% of the WAC limits shown in Table 3-13 and Table 3-14. In October 2010, SRNL reviewed the MRLs for the organic constituents in Tank 50H.⁶ All of the MRLs are at or below the WAC targets for the organics with the exception of NORPAR 13 and Isopar L. NORPAR 13 has a MRL of 0.75 mg/L and is above the WAC target.² The MRL for Isopar L is < 26.8 ppm and above the WAC limit.² Isopar L and NORPAR 13 have negligible solubility in aqueous solutions, which makes it difficult to obtain reliable sub-samples of the original sample. The values reported are the concentrations as detected by the Gas Chromatography/Mass Spectrometry (GC/MS), but may not necessarily be an accurate representation of the actual concentrations of these analytes in Tank 50H.

In a memo from Savannah River Remediation (SRR), the requested detection limits for several radionuclides were lowered in order to accommodate future inventory reporting requirements.⁷ In Table 3-14, the reported detection limit of ⁵⁹Ni (< 2.91E+00 pCi/mL) is less than the limit requested by SRR (6.59E+00 pCi/mL) and the reported detection limit for ⁹⁴Nb (<2.41E-01 pCi/mL) is above the limit requested by SRR (2.00E-03 pCi/mL).⁷ Both of the reported detection limits for ⁵⁹Ni and ⁹⁴Nb are below the quantification limit established by SRNL (2.0E+01 for ⁵⁹Ni and 4.38E-01 for ⁹⁴Nb)⁸ and they are below the WAC targets.²

Table 3-15 and Table 3-16 list the chemical contaminants that affect SDU flammability. Isopar L and NORPAR 13 are the only species considered in Table 3-15 or Table 3-16 with reported values above the WAC limit.²

Table 3-15. Tank 50H 3QCY15 Slurry Sample Chemical Results Impacting SDU Flammability²

Chemical Name	Method	Average Concentration (mg/L)	Std. Dev.	WAC Limit
Isopar L	SVOA	< 2.68E+01 ppm ^{a,b}	NA	1.10E+01 ppm
Tetraphenylborate (TPB anion)	HPLC	< 5.00E+00	NA	5.00E+00 mg/L
Ammonium (NH ₄ ⁺)	IC	< 1.00E+01	NA	2.12E+02 mg/L
Total Mercury	CVAA	1.02E+02	7.12E-02	3.25E+02 mg/L
Monomethyl Mercury	CVAFS w/ Distillation	5.33E+01	6.40E+00	3.25E+02 mg/L
Dimethyl Mercury	CVAFS	1.43E-01	1.42E-02	1.00E+00 mg/L

- a. Measurement performed on duplicate samples rather than triplicate samples.
b. Result is calculated from the reported concentration of < 33 mg/L and the density in Table 3-18.

Table 3-16. Tank 50H 3QCY15 Slurry Sample “Other Organics” Results Impacting SDU Flammability²

Chemical Name	Method	Average Concentration (mg/L)	Std. Dev.	WAC Concentrations (mg/L)
Butanol	VOA	< 5.00E-01	NA	0.75
Tributylphosphate	SVOA	< 7.50E-01	NA	1.0
Propanol	VOA	< 2.50E-01	NA	0.25
Methanol	a	NA	NA	0.05
NORPAR 13	SVOA	< 7.50E-01	NA	0.1

- a. Currently, a routine method for detecting this species does not exist in SRNL.

Table 3-17 provides results for the processing criteria for transfers into the Saltstone Facility. All of the results contained in this table fall within the general processing criteria.² The pH was calculated using the free base concentration (OH⁻). There are no measurable insoluble solids in the slurry using the method of oven-dried slurry and filtrate at 110°C.¹⁵

Table 3-17. Tank 50H 3QCY15 Slurry Sample Results for Saltstone Processing Criteria²

Processing Criterion	Method	Value	Std. Dev.
pH > 10	Calculated	>13	NA
2.5 M < [Na ⁺] < 7.0 M	ICP-ES / AA	4.96 M	8.82E-02
Total Insoluble Solids <15 wt%	Calculated	~0 wt%	NA

Table 3-18 provides constituents listed in the TTR⁹ but not contained in the WAC. The results from this table are used to support the TCLP/UHC testing by a certified laboratory.¹⁷

Table 3-18. Tank 50H 3QCY15 Slurry Sample Requests for Constituents for TCLP/UHC¹⁷

Constituent	Method	Average Value (mg/L, unless stated otherwise)	Std. Dev.
Antimony (Sb)	ICP-MS	< 3.58E-02	NA
Beryllium (Be)	ICP-ES	< 2.29E-01	NA
Cyanide (CN)	a	NA	NA
Thallium (Tl)	ICP-MS	< 2.91E-02	NA
Density (slurry)	Measured (22.2°C)	1.2334 g/mL	0.0001
Total Beta	LSC	6.53E+05 pCi/mL	1.19E+04
Total Solids	Measured	27.74 wt%	0.303

a. Currently, a routine method for detecting this species does not exist in SRNL.

The tank corrosion species listed in Table 3-19 were requested by D&S-FE.¹³ The Specific gravity was calculated by dividing the measured density of the slurry given in Table 3-18 by the density of water at the same temperature.¹⁸

Table 3-19. Tank 50H 3QCY15 Slurry Sample Requests from the D&S-FE for Corrosion Species¹³

Constituent	Method	Average Value	Std. Dev.
Specific Gravity	a	1.2356	0.0001
Total Gamma	b	5.19E+05 pCi/mL	7.10E+03 ^c

- a. Calculated from the measured density of slurry and density of water at 20.2 °C.¹⁸
b. Calculated from the sum of measured gamma emitters ⁶⁰Co, ¹²⁵Sb, ¹²⁶Sb, ¹²⁶Sn, ^{137m}Ba, ¹⁵⁴Eu, and ²⁴¹Am.
c. Value is the “standard error of the mean” rather than the standard deviation of the measurements since its calculation involves multiple radionuclides.

Table 3-20 provides results for additional radionuclides required for quantification and support of inventory-reporting requirements as requested by D&S-FE.¹³

Table 3-20. Tank 50H 3QCY15 Slurry Sample Radionuclides Results for Inventory Reporting Requirement¹³

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.
Niobium-93m (^{93m} Nb)	Calculation (Secular Equilibrium w/ 97.5% of Zr-93)	< 5.02E+01	NA
Silver-108m (^{108m} Ag)	Gamma scan (Cs removed)	< 3.57E-01	NA
Barium-133 (¹³³ Ba)	Gamma scan (Cs removed)	< 5.77E-01	NA
Bismuth-207 (²⁰⁷ Bi)	Gamma scan (Cs removed)	< 2.79E-01	NA
Thorium-228 (²²⁸ Th)	Gamma scan (Cs removed)	< 7.61E+00	NA
Curium-247 (²⁴⁷ Cm)	Am/Cm	< 3.13E+00	NA
Californium-249 (²⁴⁹ Cf)	Am/Cm	< 3.24E+00	NA
Californium-251 (²⁵¹ Cf)	Am/Cm	< 2.45E+00	NA

4.0 Conclusions

The following conclusions are drawn from the analytical results pertaining to the WAC provided in this report:

- SRR WAC targets or limits were met for all analyzed chemical and radioactive contaminants unless noted in this section.
- Of the forty-seven metal, anion, and organic contaminants listed in Attachment 8.1 and 8.2 of the WAC for 1QCY15 and 2QCY15 samples, six of the detectable species are above 10% of the WAC limit or target.¹ These contaminants are carbonate, total base (OH⁻), nitrate, nitrite, sulfate (2QCY15 only) and total mercury. For the 3QCY15 sample of the fifty metal, anion, and organic contaminants listed in Attachment 8.1 and 8.2 for the WAC, eight of the detectable species are above 10% of the WAC limit or target.² These contaminants are carbonate, total base (OH⁻), nitrate, nitrite, sulfate, total mercury, monomethyl mercury and dimethyl mercury.
- Of the fifty-five radionuclides contaminants listed in Attachment 8.3 and 8.4 of the WAC for 1QCY15 and 2QCY15 samples, four are above 10% of the WAC limit or target.¹ These radionuclide contaminants are ⁹⁹Tc, ¹²⁹I, ¹³⁷Cs, and ^{137m}Ba. Of the fifty-five radionuclides contaminants listed in Attachment 8.3 and 8.4 of the WAC for 3QCY15 samples, three are above 10% of the WAC limit or target.² These radionuclide contaminants are ¹²⁹I, ¹³⁷Cs, and ^{137m}Ba.
- NORPAR 13 and Isopar L have higher detection limits⁶ compared with the Saltstone WAC target (for NORPAR 13) and limit (for Isopar L).^{1,2} The data provided in this report are based upon concentrations in the sub-sample, and due to the limited solubility of these materials in aqueous solution, may not represent the concentrations of the analytes in Tank 50H.
- All other organic species listed in Attachment 8.1 and 8.2 of the WAC for 1QCY15, 2QCY15, and 3QCY15 are below the method detection limits, except for total organic carbon which is detected at levels (304 mg/L to 315 mg/L) that are below 10% of the WAC limit of 5,000 mg/L.^{1,2}

Additional conclusions are:

- Certain constituents requested in the Task Technical Request pertaining to Toxicity Characteristic Leaching Procedure (TCLP) and Underlying Hazardous Constituents (UHC) such as antimony, beryllium and thallium were not detected. The total beta and total solids are in the range of 6.53E+05 pCi/mL to 7.97E+05 pCi/mL and 27.10 wt% to 27.87 wt%, respectively.
- The specific gravity and total gamma activity are reported in the range of 1.2347 to 1.2362 and 5.19E+05 pCi/mL to 8.00E+05 pCi/mL, respectively.

- Minimum detection limits are reported for ^{59}Ni , ^{94}Nb , ^{247}Cm , ^{249}Cf , and ^{251}Cf as determined from the minimum detectable activity associated with the radiochemical methods used for these radionuclides for 1QCY15, 2QCY15, and 3QCY15 samples. The reported detection limits are above the requested SRR target minimum detection limit concentrations except for the 2QCY15 and 3QCY15 ^{59}Ni minimum detection limits which are below the SRR target minimum.⁷ However, the reported values for the radionuclides listed above, are below the estimated detection limits initially established by SRNL in 2009, except for the 2QCY15 ^{94}Nb value which is ~ 6% above the estimated detection limit set by SRNL in 2009.⁸

5.0 References

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