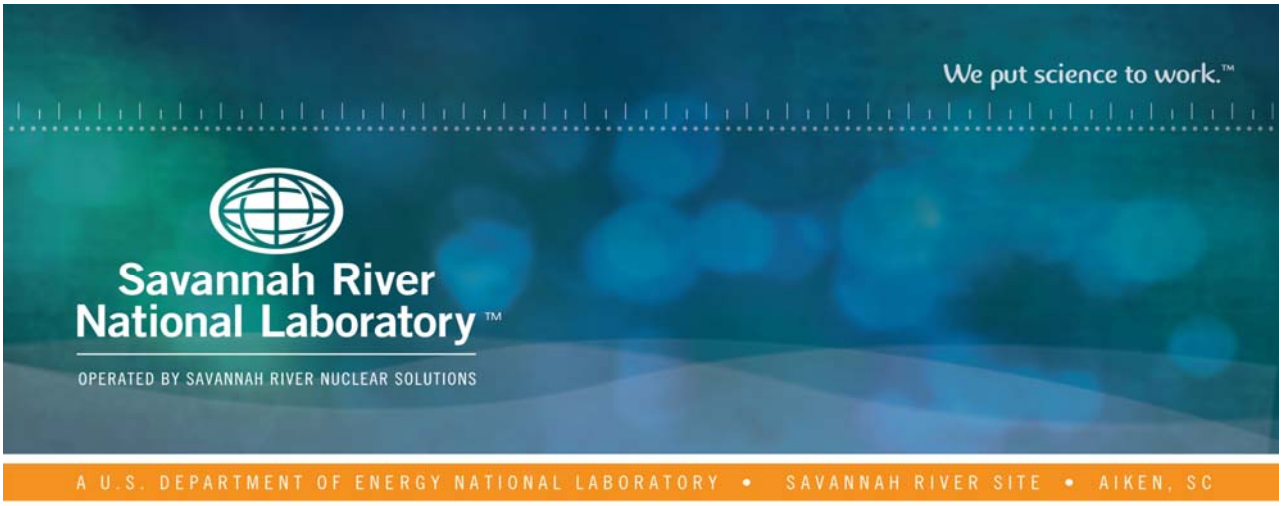

Results for the Fourth Quarter 2012 Tank 50 WAC Slurry Sample

Chemical and Radionuclide Contaminants

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September 2014

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EXECUTIVE SUMMARY

This report details the chemical and radionuclide contaminant results for the characterization of the 2012 Fourth Quarter sampling of Tank 50 for the Saltstone Waste Acceptance Criteria (WAC).[1] Information from this characterization will be used by Waste Solidification Engineering (WSE) to support the transfer of low-level aqueous waste from Tank 50 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 50 Waste Characterization System.

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

- The concentration of the reported chemical and radioactive contaminants were less than their respective WAC Limits and Targets, unless noted in this section.

- Norpar 13 and Isopar L have higher detection limits[5] compared with the Saltstone WAC[1]. The data provided in this report is based upon the concentrations in the sub-sample, and due to the limited solubility in aqueous solution, may not represent the concentrations of the analytes in Tank 50.

- Diisooctyl adipate (or diisooctyl hexanedioate) was measured at 1.30E+00 mg/L in one of two replicate measurements conducted on an at-depth sample.[a] The organic analysis of the at-depth sample was conducted at the request of SRR.[4] This analyte was below the detection limit in the surface sample.

- The low insoluble solids content increases the measurement uncertainty for insoluble species.

[a] Revision 1 of this document removes the previously reported 5-methyl-3-hexanol SVOA analyte per SRNL-STI-2014-00088 (Ref. 12).

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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
DDA	Deliquification, Dissolution and Adjustment
EPA	Environmental Protection Agency
ETP	Effluent Treatment Project
GC/MS	Gas Chromatograph/Mass Spectrometer
HDPE	High Density Polyethylene
HPLC	High Performance Liquid Chromatography
IC	Ion Chromatography
ICP-AES	Inductively coupled plasma – atomic emission spectroscopy
ICP-MS	Inductively coupled plasma – mass spectrometry
L	Liter
LLW	Low Level Waste
LSC	Liquid Scintillation Counting
MDL	Method Detection Limit
MRL	Method Reporting Limit
mg	Milligram
mL	Milliliter
NA	Not Applicable
ND	Not Determined
pCi/mL	Picocurie per milliliter
RSD	Relative Standard Deviation
SC	Shielded Cells (Facility)
SDF	Saltstone Disposal Facility
SFT	Salt Feed Tank
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site

SVOA	Semi-volatile Organic Analysis
TCLP/UHC	Toxic Characterization Leaching Procedure/Underlying Hazardous Constituent
TIC	Tentatively Identifiable Compound
TIC/TOC	Total inorganic carbon/total organic carbon
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
VOA	Volatile organic analysis
WAC	Waste Acceptance Criteria
WCS	Waste Characterization System
WSE	Waste Solidification Engineering
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.[1] 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 50 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] The specific chemical and radionuclide contaminants and their respective WAC limits are in the current Saltstone WAC.[1]

Waste Solidification Engineering (WSE) requested that the Savannah River National Laboratory (SRNL) perform quarterly analysis on saltstone samples.[6] The concentrations of chemical and radionuclide contaminants are measured to ensure the saltstone produced during each quarter is in compliance with the current WAC.[1, 6, 7] This report documents the concentrations of chemical and radionuclide contaminants and discusses those results for the 2012 Fourth Quarter samples collected from Tank 50 on October 9, 2012.

2.0 Experimental

On October 9, 2012, six 200-mL samplers (HTF-50-12-88, -89, -90, -91, -92, -93) were collected from Tank 50 for the Fourth Quarter 2012 (4Q12) WAC analyses and delivered the next day to the SRNL Shielded Cells (SC). The first 200 mL sampler (HTF-50-12-88) is a dip sample taken six inches below the surface and the remaining five samplers were pulled 66 inches from the bottom of the tank with one agitator pump running.

At SRNL, slurry samples (~15 mL each) from HTF-50-12-88 and HTF-50-12-89 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). Four additional 15-mL aliquots (for duplicate analyses) from HTF-50-12-88 were used for SVOA analysis to determine the concentration of Isopar L and Norpar 13, respectively, in the sample.

After the samples for organic analyses were obtained, the slurries in the steel samplers were combined into a 2-L high density polyethylene (HDPE) bottle. The 200 mL steel samplers were agitated to disperse solids in the slurry and poured into the 2-L HDPE bottle. The transferred slurry was left to settle in the 2-L bottle. A portion of the clear supernate was returned to each steel sampler, mixed to mobilize any remaining solids, and again returned to the 2-L HDPE bottle. Visual inspection of the inside of sampler indicated there were no visible solids remaining in the samplers. The total weight of the transferred slurry was 1074.70g.

The 2-L HDPE bottle was agitated to thoroughly disperse the extremely limited solids into the supernate. Aliquots of slurry samples were promptly collected with slurry pipettes to minimize settling effects and placed into HDPE bottles. A three milliliter sample of the slurry was used to determine the density of the slurry using an Anton-Paar DMA 35n portable density meter.

Slurry samples were submitted in triplicate to SRNL laboratories 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 and ethylenediaminetetraacetate 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.
- Approximately 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[8]. 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 – atomic emission spectroscopy (ICP-AES), 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 Hg.

3.0 Results and Discussion

The following tables contain the results for the 4Q12 WAC analyses. 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. 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.

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]

Table -3-1. Results for the 4th Quarter 2012 Tank 50 Slurry Samples for Chemical Contaminants Listed in Attachment 8.1 of the Saltstone WAC, Revision 11

Chemical Name	Method	Average Concentration (mg/L)	Std. Dev.	WAC Limit (mg/L)
Ammonium (NH_4^+)	IC	<1.00E+02	NA	**5.88E+03**
Carbonate (CO_3^{2-})	TIC	1.07E+04[a]	6.61E+01	**1.20E+05**
Chloride (Cl^-)	IC	1.25E+02	1.78E+01	**7.95E+03**
Fluoride (F^-)	IC	<1.00E+02	NA	**4.07E+03**
Free Hydroxide (OH^-)	Total base	3.29E+04[a]	4.28E+02	**1.58E+05**
Nitrate (NO_3^-)	IC	1.04E+05	1.06E+04	**4.37E+05**
Nitrite (NO_2^-)	IC	1.91E+04	1.65E+03	**2.14E+05**
Oxalate ($C_2O_4^{2-}$)	IC	4.91E+02	3.78E+01	**2.72E+04**
Phosphate (PO_4^{3-})	IC/ICP-ES	4.03E+02	3.44E+01	**2.94E+04**
Sulfate (SO_4^{2-})	IC	3.29E+03	3.84E+02	**5.69E+04**
Arsenic (As)	AA	<1.02E-01	NA	**1.50E+02**
Barium (Ba)	ICP-ES	<8.97E-01	NA	**6.19E+02**

Chemical Name	Method	Average Concentration (mg/L)	Std. Dev.	WAC Limit (mg/L)
Cadmium (Cd)	ICP-ES	<1.23E+00	NA	**3.10E+02**
Chromium (Cr)	ICP-ES	4.06E+01	6.70E-01	**1.24E+03**
Lead (Pb)	ICP-MS	4.50E-01	3.05E-02	**6.19E+02**
Mercury (Hg)	AA	3.67E+01	8.10E-01	**3.25E+02**
Selenium (Se)	AA	<2.03E-01	NA	**4.46E+02**
Silver (Ag)	ICP-ES	<1.61E+00	NA	**6.19E+02**
Aluminum (Al)	ICP-ES	3.20E+03	4.39E+01	**1.16E+05**
Potassium	AA	2.62E+02	1.17E+01	**3.03E+04**
Nickel Hydroxide	ICP-ES	<5.40E+00[e]	NA	**1.17E+03**
n-Butanol	VOA	6.40E-01[b]	NA	**7.73E+00**
i-Butanol	VOA	6.40E-01[b]	NA	**7.73E+00**
i-Propanol	VOA	<2.50E-01[b]	NA	**1.88E+00**
Phenol	SVOA	<1.00E+01[b]	NA	**7.50E+02**
Isopar L	SVOA	<2.71E+01 ppm[c,d]	NA	**6.56E+01 ppm**
Total organic carbon	TOC	4.27E+02[a]	5.77E+00	**5.00E+03**
Tetraphenylborate (TPB anion)	HPLC	<5.00E+00	NA	**5.00E+00**

a. Measurement performed on filtered supernate samples.
b. Measurement performed on quadruplicate samples rather than triplicate samples.
c. Measurement performed on duplicate samples rather than triplicate samples.
d. Result is calculated from the reported concentration of < 33 mg/L and the density of the slurry sample listed in Table 3-8.
e. Result is calculated from the measured Ni concentration assuming all of the Ni is present as the hydroxide compound.

Table -3-2. Results for the 4th Quarter 2012 Tank 50 Slurry Samples for Chemical Contaminants Listed in Attachment 8.2 of the Saltstone WAC, Revision 11

Chemical Name	Method	Average Concentration (mg/L)	Std. Dev.	WAC TARGET (mg/L)
Boron (B)	ICP-ES	7.76E+01	1.37E+00	**7.43E+02**
Cobalt (Co)	ICP-MS	<1.85E-02	NA	**7.43E+02**
Copper (Cu)	ICP-ES	8.08E-01	7.61E-02	**7.43E+02**
Iron (Fe)	ICP-ES	8.71E+00	6.15E-01	**4.95E+03**
Lithium (Li)	ICP-ES	1.33E+01	2.43E-01	**7.43E+02**
Manganese (Mn)	ICP-ES	2.90E+00	4.39E-02	**7.43E+02**
Molybdenum (Mo)	ICP-ES	7.50E+00	4.51E-01	**7.43E+02**
Nickel (Ni)	ICP-ES	<3.42E+00	NA	**7.43E+02**
Silicon (Si)	ICP-ES	4.13E+01	2.31E+01	**1.07E+04**
Strontium (Sr)	ICP-ES	<1.39E-01	NA	**7.43E+02**
Zinc (Zn)	ICP-ES	5.88E+00	1.44E-01	**8.03E+02**
Benzene	VOA	<1.50E-01[a]	NA	**3.10E+02**
Methanol	VOA	c	NA	**1.88E+00**
Toluene	VOA	<1.50E-01[a]	NA	**3.10E+02**
Dibutylphosphate (DBP)	IC	<2.75E+02	NA	**3.47E+02**
Tributyl Phosphate (TBP)	SVOA	<7.50E-01[a]	NA	**7.50E+00**
EDTA	HPLC	<1.00E+02	NA	**3.10E+02**
Norpar 13	SVOA	<7.50E-01[b]	NA	**1.00E-01**

a. Measurement performed on quadruplicate samples rather than triplicate samples.
b. Measurement performed on duplicate samples rather than triplicate samples.
c. Currently, a routine method for detecting this species does not exist in SRNL.

As indicated in Tables 3-1 and 3-2, all of the contaminants are within the WAC limits with the exception of Norpar 13. In October 2010, SRNL reviewed the MRL's for the organic constituents in Tank 50. All of the MRL's are at or below the WAC limits for the organics with the exception of Norpar 13 which has an MRL of 0.75 mg/L, which is above the WAC limit.[5] 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 in these tables are the concentrations as detected by the GC/MS but may not necessarily be an accurate representation of the concentrations of these analytes in Tank 50.

Table -3-3. Results for 4th Quarter 2012 Tank 50 Slurry Samples and WAC Limits for Radionuclide Contaminants Listed in Attachment 8.3 of the Saltstone WAC, Revision 11

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC LIMIT (pCi/mL)
Tritium (^{3}H)	Tritium counting	4.17E+02	4.11E+01	**5.63E+05**
Carbon-14 (^{14}C)	C-14 Liquid scintillation	4.12E+02	8.46E+01	**1.13E+05**
Nickel-63 (^{63}Ni)	Ni-59/63	<4.22E+00	NA	**1.13E+05**
Strontium-90 (^{90}Sr)	Sr-90 Liquid scintillation	1.98E+03	1.45E+01	**2.25E+07**
Technetium-99 (^{99}Tc)	Tc-99 Liquid scintillation	2.50E+04	1.86E+03	**2.11E+05**
Iodine-129 (^{129}I)	I-129 (w/ separation) Liquid scintillation	1.41E+01	2.96E+00	**3.80E+02**
Cesium-137 (^{137}Cs)	Gamma Scan	1.39E+06	1.19E+04	**7.13E+06**
Uranium-233 (^{233}U)	ICP-MS	<1.79E+02	NA	**1.13E+04**
Uranium-235 (^{235}U)	ICP-MS	2.50E-01	9.48E-03	**1.13E+02**
Plutonium-241 (^{241}Pu)	Pu238/241 Liquid scintillation	<1.97E+02	NA	**8.38E+05**
Total Alpha	Liquid Scintillation Counting	<7.30E+02	NA	**2.50E+05**

As shown in Table 3-4, none of the radionuclide contaminants exceed the targets listed in the latest revision of the WAC. 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.[2] The reported detection limits of ^{59}Ni and ^{94}Nb are above the limits requested by SRR (6.59E+00 and 2.00E-03 pCi/mL, respectively)[2] but below the quantification limits established by SRNL (2.00E+01 and 4.38E-01 pCi/mL, respectively).[3]

The concentration of ^{135}Cs is calculated by assigning all of the mass at 135 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 limit for each radionuclide.

Table 3-4. Results for the 4th Quarter 2012 Tank 50 Slurry Samples and WAC Targets for Radionuclide Contaminants Listed in Attachment 8.4 of the Saltstone WAC, Revision 11

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC TARGET (pCi/mL)
Aluminum-26 (^{26}Al)	Gamma scan (Cs removed)	<1.59E-01	NA	**2.88E+03**
Cobalt-60 (^{60}Co)	Gamma scan (Cs removed)	3.39E-01	1.37E-02	**4.87E+03**
Potassium-40 (^{40}K)	Gamma scan (Cs removed)	<1.40E+00	NA	**1.00E+02**
Nickel-59 (^{59}Ni)	Ni-59/63	<1.22E+01	NA	**1.13E+03**
Selenium-79 (^{79}Se)	Se-79	2.93E+01	1.29E+01	**1.90E+04**
Zirconium-93 (^{93}Zr)	Zr-93	<1.70E+01	NA	**1.00E+05**
Niobium-94 (^{94}Nb)	Gamma scan (Cs removed)	<1.98E-01	NA	**1.53E+02**
Ruthenium-106 (^{106}Ru)	Gamma scan (Cs removed)	<1.65E+00	NA	**1.13E+06**
Antimony-125 (^{125}Sb)	Gamma scan (Cs removed)	2.11E+02	2.75E+00	**3.99E+04**
Tin-126 (^{126}Sn)	Gamma scan (Cs removed)	1.15E+02	4.06E+00	**1.80E+04**
Cesium-134 (^{134}Cs)	Gamma Scan	<8.29E+01	NA	**3.28E+04**
Cesium-135 (^{135}Cs)	ICP-MS	<2.13E+01	NA	**1.50E+03**
Cerium-144 (^{144}Ce)	Gamma scan (Cs removed)	<2.00E+00	NA	**1.13E+05**
Promethium-147 (^{147}Pm)	Pm-147/Sm-151 Liquid scintillation	<2.66E+01	NA	**5.63E+06**
Samarium-151 (^{151}Sm)	Pm-147/Sm-151 Liquid scintillation	<2.67E+01	NA	**2.25E+04**
Europium-154 (^{154}Eu)	Gamma scan (Cs removed)	1.87E+00	3.64E-01	**8.03E+03**
Europium-155 (^{155}Eu)	Gamma scan (Cs removed)	<1.17E+00	NA	**1.13E+04**
Radium-226 (^{226}Ra)	Gamma scan (Cs removed)	<6.13E+00	NA	**1.00E+03**
Radium-228 (^{228}Ra)	Gamma scan (Cs removed)	<7.57E-01	NA	**1.00E+04**
Actinium-227 (^{227}Ac)	Gamma scan (Cs removed)	<4.42E+00	NA	**1.00E+04**
Thorium-229 (^{229}Th)	ICP-MS	<3.93E+03	NA	**1.63E+05**
Thorium-230 (^{230}Th)	ICP-MS	<3.90E+02	NA	**6.26E+03**
Thorium-232 (^{232}Th)	ICP-MS	<2.03E-03	NA	**2.88E+03**
Protactinium-231 (^{231}Pa)	Gamma scan (Cs removed)	<1.20E+01	NA	**1.00E+03**
Uranium-232 (^{232}U)	U-232	1.97E+00	9.59E-01	**1.71E+05**
Uranium-234 (^{234}U)	ICP-MS	<1.16E+02	NA	**1.13E+04**
Uranium-236 (^{236}U)	ICP-MS	<1.20E+00	NA	**1.13E+04**
Uranium-238 (^{238}U)	ICP-MS	4.92E+00	1.03E-01	**1.13E+04**
Neptunium-237 (^{237}Np)	ICP-MS	<1.30E+01	NA	**1.00E+04**

Plutonium-238 (^{238}Pu)	Pu238/241 Pu alpha PHA	5.95E+02	9.37E+01	**2.50E+05**
Plutonium-239 (^{239}Pu)	Pu238/241 Pu alpha PHA	1.38E+02	3.54E+01	**2.50E+05**
Plutonium-240 (^{240}Pu)	Pu238/241 Pu alpha PHA	1.38E+02	3.54E+01	**2.50E+05**
Plutonium-242 (^{242}Pu)	ICP-MS	<7.06E+01	NA	**2.50E+05**
Plutonium-244 (^{244}Pu)	ICP-MS	<3.28E-01	NA	**7.02E+04**
Americium-241 (^{241}Am)	Gamma scan (Cs removed)	2.25E+00[a]	NA	**2.50E+05**
Americium-242m (^{242m}Am)	Am/Cm	<3.90E-02	NA	**4.50E+05**
Americium-243 (^{243}Am)	Am/Cm	<9.73E-01	NA	**2.50E+05**
Curium-242 (^{242}Cm)	Am/Cm	<3.23E-02	NA	**1.13E+04**
Curium-244 (^{244}Cm)	Am/Cm	1.42E+01	4.15E+00	**2.50E+05**
Curium-245 (^{245}Cm)	Am/Cm	<8.51E+00	NA	**2.25E+05**

a. Measurement based on a single sample with a long 55 hour count.

Tables 3-5 and 3-6 list the chemical contaminants that impact vault flammability. These chemicals must be monitored to ensure flammable gases do not contribute more than 10% of the Composite Lower Flammability Limit (CLFL).[1]

Table -3-5. Results for the 4th Quarter 2012 Tank 50 Slurry Samples for Acceptance Criteria Limits for Chemical Contaminants Impacting Vault Flammability, Listed in Table 3 of the Saltstone WAC, Revision 11

Chemical Name	Method	Average Concentration (mg/L)	Std. Dev.	WAC Limit
Isopar L	SVOA	<2.71E+01 ppm[a]	NA	**1.10E+01 ppm**
Tetraphenylborate (TPB anion)	HPLC	<5.00E+00	NA	**5.00E+00 mg/L**
Ammonium (NH_4^+)	IC	<1.00E+02	NA	**2.12E+02 mg/L**

a. Result is calculated from the reported concentration of < 33 mg/L and the density of the slurry sample.

Table -3-6. Results for the 4th Quarter 2012 Tank 50 Slurry Samples for Concentrations of "Other Organics" Impacting Vault Flammability, Listed in Table 4 of the Saltstone WAC, Revision 11

Chemical Name	Method	Average Concentration (mg/L)	Std. Dev.	WAC Concentrations
n-Butanol	VOA	6.40E-01	NA	**0.75 mg/L**
Tributylphosphate	SVOA	<7.50E-01	NA	**1.0 mg/L**
i-Propanol	VOA	<2.50E-01	NA	**0.25 mg/L**
Methanol	a	NA	NA	**0.25 mg/L**
Norpar 13	SVOA	<7.50E-01	NA	**0.1 mg/L**

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 Tables 3-5 or 3-6 with reported values above the WAC limit. Although the reported detection limit for Isopar L is greater than the WAC limit for vault flammability, it is below the WAC limits for accident analysis as shown in Table 3-1. It should be noted that the detection limit for Isopar L was expected based on current SRNL capabilities.[5] The reported detection limit for Norpar is above the WAC limit for both accident analysis (Table 3-2) and vault flammability (Table 3-6) but it is the lowest achievable MRL for this analyte.[5] 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 50 sample received by SRNL.

Diisooctyl adipate (or diisooctyl hexanedioate) was measured at 1.30E+00 mg/L in one of two replicate measurements conducted on an at-depth sample. Analysis of an at-depth sample for plasticizers was requested by SRR.[4] The compound was not measured above the detection limit in the surface dip sample, nor a parallel blank sample prepared in the SRNL Shielded Cells. This material is a plasticizer. It is unclear if this material is lab carryover from composite SRNL LAW tank samples that arrived in plastic bottles or from a customer working on Tank 48 simulant with plastic wear. The material did not appear in laboratory blanks analyzed on the same instrument, so the results are reported here. Diisooctyl adipate is not likely to be a flammability concern due to its high boiling point of 210 ºC at 4 mmHg[9]. The previous revision of this document also reported 5-methyl-3-hexanol as a component of one of the two replicate measurements conducted on the at-depth sample. It has now been learned that this substituted alcohol is an artifact produced during the sample preparation for phenol determination when the Tank 50 sample, containing added internal standards, is over acidified.[12]

Table 3-7 provides results for the processing criteria for transfers into the Saltstone Facility.

Table -3-7. Results for the 4th Quarter 2012 Tank 50 Slurry Samples for Saltstone Processing Criteria WAC Limits, Listed in Table 5 of the Saltstone WAC, Revision 11

Processing Criterion	Method	Value	Std. Dev.
pH > 10	Calculated	>13	NA
2.5 M < [Na^+] < 7. 0 M	AA/ICP-ES	4.85 M	0.154
Total Insoluble Solids <15 wt%	Calculated	0.421 wt%	0.621

All of the results contained in Table 3-7 fall within the general processing criteria. The pH was calculated using the free base concentration (OH^-). The low insoluble solids content, visible but not measureable, makes subsampling difficult.

Table 3-8 provides constituents listed in the Technical Task Request but not contained in the WAC.

Table -3-8. Requests for Constituents for TCLP/UHC Support as well as from the TTR for the 4th Quarter 2012 Tank 50 Slurry Samples; Results Not Contained in Previous Tables

Constituent	Method	Average Value (mg/L, unless stated otherwise)	Std. Dev.
Antimony (Sb)	ICP-ES	<3.35E+01	NA
Beryllium (Be)	ICP-ES	<2.21E-01	NA
Cyanide (CN)	a	NA	NA
Thallium (Tl)	ICP-MS	6.56E-02	NA
Density (slurry)	Measured (20.7ºC)	1.2165	0.0019
Total Beta	LSC	1.54E+06 pCi/mL	5.41E+04
Total Solids	Measured	26.85 wt%	0.065

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

The results from Table 3-8 are used to support TCLP/UHC testing by a certified laboratory.[10] The density of the slurry was measured at 20.7 ºC. Natural Tl is composed of two isotopes, ^{203}Tl and ^{205}Tl 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 that determined from a single replicate for ^{205}Tl with a value above the detection limit.

The tank corrosion species listed in Table 3-9 were requested by Waste Solidification Engineering (WSE).[11] Specific gravity was calculated by dividing the measured density of the slurry (given in Table 3-8 at 20.7 °C) by the density of water at the same temperature.[9]

Table -3-9. Requests from the WSE for Corrosion Species from the 4th Quarter 2012 Tank 50 Slurry Samples; Results Not Contained in Previous Tables

Constituent	Method	Average Value	Std. Dev.
Specific Gravity	a	1.2189	0.0019
Ba-137m	b	1.31E+06 pCi/mL	1.13E+04
Total Gamma	c	1.31E+06 pCi/mL	6.51E+03

a. Calculated from the measured density of slurry and density of water at 20.7 °C[9].
b. Calculated from the measured concentration of Cs-137.
c. Calculated from the sum of measured gamma emitters.

The radionuclide ^{137m}Ba is the radioactive daughter of 94.6% of the beta decay of ^{137}Cs. 5.3% of the ^{137}Cs decays to stable ^{137}Ba. The half-life of the parent radionuclide, ^{137}Cs, is five 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 1.31E+06 pCi/mL. 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 ^{137}Cs. The total gamma activity was calculated by summing the measured gamma activity of the major gamma emitters: ^{60}Co, ^{125}Sb, ^{126}Sb, ^{126}Sn, ^{137}Cs (via ^{137m}Ba), ^{154}Eu, and ^{241}Am.

Table 3-10 provides results for additional radionuclides not listed in the WAC but which now require quantification in order to support inventory reporting requirements.

Table -3-10. Additional Radionuclides Requested for Inventory Reporting Requirements

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.
Niobium-93m (^{93m}Nb)	ICP-MS	<4.53E+01	NA
Silver-108m (^{108m}Ag)	Gamma scan (Cs removed)	<3.04E-01	NA
Barium-133 (^{133}Ba)	Gamma scan (Cs removed)	<4.95E-01	NA
Bismuth-207 (^{207}Bi)	Gamma scan (Cs removed)	<2.27E-01	NA
Thorium-228 (^{228}Th)	Gamma scan (Cs removed)	<7.34E+00	NA
Curium-247 (^{247}Cm)	Am/Cm	<2.72E+00	NA
Californium-249 (^{249}Cf)	Am/Cm	<2.94E+00	NA
Californium-251 (^{251}Cf)	Am/Cm	<2.01E+00	NA

4.0 Conclusions

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

- The concentration of the reported chemical and radioactive contaminants were less than their respective WAC Limits and Targets, unless noted in this section.
- Norpar 13 and Isopar L have higher detection limits[5] compared with the Saltstone WAC[1]. The data provided in this report is based upon the concentrations in the sub-sample, and due to the limited solubility in aqueous solution, may not represent the concentrations of the analytes in Tank 50.
- Diisooctyl adipate (or diisooctyl hexanedioate) was measured at 1.30E+00 mg/L in one of two replicate measurements conducted on an at-depth sample.[a] The organic analysis of the at-depth sample was conducted at the request of SRR.[4] This analyte was below the detection limit in the surface sample.
- The low insoluble solids content increases the measurement uncertainty for insoluble species.

[a] Revision 1 of this document removes the previously reported 5-methyl-3-hexanol SVOA analyte per SRNL-STI-2014-00088 (Ref. 12).

5.0 References

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6.0 Distribution

Name:	Location:	Name:	Location:
P. M. Almond	773-43A	C. A. Langton	773-43A
C. J. Bannochie	773-42A	J. N. Leita	704-30S
M. J. Barnes	773-A	K. R. Liner	704-S
L. W. Brown	773-A	M. J. Mahoney	766-H
P. L. Bovan	704-27S	S. L. Marra	773-A
N. F. Chapman	766-H	D. J. Martin	241-246H
C. K. Chiu	704-27S	P. W. Norris	704-Z
L. H. Connelly	773-A	E. Patten	704-Z
J. S. Contardi	766-H	F. M. Pennebaker	773-42A
A. D. Cozzi	999-W	J. W. Ray	704-S
C. C. DiPrete	773-A	M. M. Reigel	999-W
K. D. Dixon	704-14Z	L. B. Romanowski	766-H
C. E. Duffey	704-61H	E. R. Seldon	704-Z
A. D. England	704-14Z	A. R. Shafer	704-27S
S. D. Fink	773-A	D. C. Sherburne	704-S
K. M. Fox	999-W	F. M. Smith	705-1C
E. J. Freed	704-56H	A. V. Staub	704-Z
J. C. Griffin	773-A	K. H. Subramanian	249-8H
E. W. Harrison	704-60H	J. R. Vitali	704-71F
C. C. Herman	999-W	T. L. White	773-A
P. J. Hill	766-H	A. W. Wiggins	704-61H
P. R. Jackson	703-46A	R. H. Young	773-A
V. Jain	704-Z		