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# Results for the First Quarter Calendar Year 2019 Tank 50 Salt Solution Sample

**C. L. Crawford**

June 2019

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## EXECUTIVE SUMMARY

In this Technical Report, the chemical and radionuclide contaminant results from the First Quarter Calendar Year 2019 (CY19) sample of Tank 50 salt solution are presented in tabulated form. The information from this characterization will be used by Savannah River Remediation (SRR) for the transfer of aqueous waste from Tank 50 to the Saltstone Production Facility (SPF), where the waste will be treated and disposed in the Saltstone Disposal Facility. This Technical Report compares results, where applicable, to SPF Waste Acceptance Criteria (WAC) Limits and Targets.<sup>1</sup> The chemical and radionuclide contaminant results from the characterization of the First Quarter CY19 sampling of Tank 50 were requested by SRR personnel via a Task Technical Request (TTR)<sup>2</sup> and details of the testing are presented in the Savannah River National Laboratory (SRNL) Task Technical and Quality Assurance Plan (TTQAP).<sup>3</sup> This Technical Report is part of Deliverable 2 relating to Task 1 from the SRR request.<sup>2</sup> Data pertaining to the regulatory limits for Resource Conservation and Recovery Act (RCRA) metals will be documented at a later time per the TTQAP for the Tank 50 Saltstone task.<sup>3</sup>

The following facts pertaining to the WAC are drawn from the analytical results provided in this report.

- WAC Targets or Limits were met for all analyzed chemical and radioactive contaminants for which the detection limits are below the WAC Targets or Limits.
- Isopar L<sup>a</sup> has a higher detection limit<sup>4</sup> compared with the current SPF WAC Limit value of 11 ppm<sup>1</sup> associated with flammability that has been in effect since revision 12 of the WAC dating back to July of 2013.<sup>5</sup>
- Nitrosamines were not detected in the Tank 50 salt solution sample above the instrument detection limits of < 1 mg/L.
- The minimum detection limit (<1.97E-01 pCi/mL) is reported for <sup>94</sup>Nb as determined from the minimum detectable activity associated with the radiochemical method used for this radionuclide. The reported detection limit is above the requested SRR target minimum detection limit concentration.<sup>6</sup> However, the minimum detection limit reported for the First Quarter CY19 Tank 50 sample for <sup>94</sup>Nb is lower than the estimated detection limit of 4.38E-01 pCi/mL initially established by SRNL in 2009.<sup>7</sup> Thus per guidance from SRR,<sup>6</sup> SRNL continues to achieve as low as practical detection limits for this radionuclide.

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<sup>a</sup> Isopar L is a trademark chemical (Isopar™ L) manufactured by ExxonMobil. It is a synthetic isoparaffinic hydrocarbon that is manufactured from a petroleum based raw material.

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## LIST OF ABBREVIATIONS

AD	Analytical Development
CVAFS	Cold Vapor Atomic Fluorescence Spectroscopy
DMA	Direct Mercury Analyzer
DSA	Documented Safety Analysis
EDTA	ethylenediaminetetraacetate
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
MRL	Minimum Reporting Limit
PHA	Pulse Height Analysis (alpha PHA)
RCRA	Resource Conservation and Recovery Act
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SVOA	Semi-Volatile Organic Analysis
TIC/TOC	Total Inorganic Carbon/Total Organic Carbon
TPB	tetraphenylborate
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
VDS	Variable Depth Sample
VOA	Volatile Organic Analysis
WAC	Waste Acceptance Criteria

## 1.0 Introduction

Tank 50 aqueous waste is analyzed on a quarterly basis and the results are compared to the Waste Acceptance Criteria (WAC) of the Z-Area Saltstone Production Facility (SPF).<sup>1</sup> The quarterly Regulatory Compliance samples pulled in Tank 50 should be characterized for both Limit and Target acceptance criteria in this WAC.<sup>1</sup> The information from this characterization will be used by Savannah River Remediation (SRR) for the transfer of aqueous waste from Tank 50 to SPF, where the waste will be treated and disposed in the Saltstone Disposal Facility. This Technical Report compares results, where applicable, to SPF WAC Limits and Targets.<sup>1</sup> A memorandum reporting the average Cs-137 value has been previously issued.<sup>8</sup>

## 2.0 Experimental

### 2.1 Technical

The First Quarter CY19 Tank 50 samples [a 200-mL sample obtained 6" below the surface (HTF-50-19-7) and a 2-L variable depth sample (VDS) obtained 66" from the tank bottom (HTF-50-19-8)] were obtained on February 5, 2019 and received at Savannah River National Laboratory (SRNL) on February 5, 2019.<sup>9</sup>

The contents of the 2-L slurry in the steel variable depth sampler were initially mixed by recycling some of the slurry using the transfer pump with both ends of the transfer line submerged in the sample. After initial mixing, two small ~ 30-mL aliquots of the Tank 50 sample were pumped into a Teflon<sup>®</sup> and a glass container with zero headspace. These two samples were used for Hg speciation testing.<sup>10</sup> The remaining contents were then transferred by pumping into two different high-density polyethylene (HDPE) 1-L bottles. The original 2-L slurry was not composited into a single container prior to distribution into the individual 1-L bottles since the Tank 50 sample contains very little suspended solids and pumping occurred immediately after handling and positioning of the 2-L sample within the variable depth sampler inside the SRNL Shielded Cells Facility. The transferred slurry was left to settle in the bottles. Visual inspection of the inside of the steel sampler indicated there were no visible solids remaining in the sampler, so no clear supernate was returned to the sampler for rinsing. The entire sample was transferred out of the Shielded Cells and located in a radiochemical hood. The two small zero headspace vials were put in shrouded containers and transferred to storage in a refrigerator. All transfers out of the Shielded Cells were made on the same day as sample collection. The 1-L bottles were agitated to thoroughly disperse the extremely limited suspended solids into the supernate. These suspended solids are typically only visible as trace solids at the bottom of the container upon prolonged storage of the material under static conditions. Aliquots of slurry samples were promptly collected with slurry pipettes to minimize settling effects and placed into HDPE bottles. Samples were removed from the 200-mL surface sample from within a radiochemical hood and were transferred using glass pipettes into clean glass sample vials with Teflon lined caps. Amber colored glass sample vials were used for the samples that were analyzed for nitrosamines to minimize exposure to light.

Unless otherwise stated, all concentrations presented in the tables (except upper limits) are averages based on analyses of triplicate aliquots of the First Quarter CY19 Tank 50 sample. The standard deviation of each average is also presented. Several of the contaminants were either not detected in the slurry samples or detected at values below the method reporting limit (MRL). For contaminants not detected or detected below the MRL, the result is preceded by a "<", which indicates the result is an upper limit based on the sensitivity of the method used to analyze the individual analyte. If only one value out of the triplicate analysis is above the detection limit, then that single value is reported and noted in the tables. Also, if only two values out of the triplicate analyses are above the detection limit, then the average of those two values is reported and noted in the tables. All volatile organic analysis (VOA) and semi-volatile organic analysis (SVOA) were performed on the surface sample and all other analyses were performed on the variable depth sample. The VOA method is performed per SRNL AD Procedure L16.1, ADS-2656.<sup>11</sup> This method is

based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) process that involves dilution of 1 mL of Tank 50 supernate with 4 mL of reagent water. The SVOA method is performed per SRNL AD Procedure L16.1, ADS-2657.<sup>12</sup> Both of these methods use discrete standards as detailed in the procedures.<sup>11,12</sup> The SVOA method uses organic solvents to extract SVOA analytes that are analyzed by GC/MS. A 2 mL dichloromethane (or methylene chloride, CH<sub>2</sub>Cl<sub>2</sub>) volume is used to initially extract 10 mL of Tank 50 supernate for phenol. The Tank 50 supernate is then extracted with 2 additional 2-mL volumes of dichloromethane. The dichloromethane extracts are combined and concentrated before analysis. Tributyl phosphate is analyzed from a 2 mL hexane (C<sub>6</sub>H<sub>14</sub>) extract of 10 mL of Tank 50 supernate. Isopar L and Norpar 13 are analyzed from a 2 mL hexane extraction of 5 mL of Tank 50 supernate. Nitrosamines are analyzed by a separate SVOA method that uses 2 mL of dichloromethane as extractant and 10 mL of Tank 50 supernate with deuterated N-nitrosodimethylamine-d6 (NDMA-d6) as a standard along with a separate GC/MS analysis methodology. Data reported for inductively coupled plasma atomic emission spectroscopy (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS) are derived from the digested Tank 50 supernate by the aqua regia method.<sup>13</sup> Anion and the ammonium cation analyses are determined from Ion Chromatography (IC). Total Inorganic Carbon/Total Organic Carbon (TIC/TOC) analysis was used to measure the TIC (carbonate) and TOC components. The tetraphenylborate (TPB) anion and ethylenediaminetetraacetate (EDTA) were analyzed using High Performance Liquid Chromatography (HPLC). All the above analyses excluding VOA and SVOA used approximately 150 mL of the 1-L variable depth sample. A 3-mL sample of the slurry was used to determine the density of the slurry using an Anton-Paar DMA 35n portable density meter. Total and soluble weight percent solids were determined on portions of the Tank 50 sample using the “Weight Percent Solids Determination Using a Furnace or Oven” procedure from the Environmental & Chemical Process Technology research programs section.<sup>14</sup>

Approximately 630 mL of the VDS were used to determine all the measured radionuclide concentrations in triplicate. Radionuclides reported using the ICP-MS method are converted from a reported mass per volume basis to activity per volume units using the specific activities (Ci/g) reported from the Department of Energy 1996 Integrated Data Base Report.<sup>15</sup>

Mercury analyses performed at SRNL by Analytical Development (AD) included Total mercury using the Direct Mercury Analyzer (DMA) method and monomethyl mercury by Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS). Other mercury (Hg) speciation data shown in Table 3-1, Table 3-2 and Table 3-5 are calculated from previous work as analyzed by Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS) by the Eurofins Frontier Global Sciences, Inc. laboratory in Seattle, WA.<sup>10</sup> The parent sample for all mercury analyses performed at either SRNL or at Eurofins was obtained from the original Tank 50 sample within two days of sample receipt. The parent sample was obtained in near zero-headspace containers that were immediately refrigerated after removal from the Shielded Cells Facility on the same day of preparation. The Hg species reported from Eurofins include elemental mercury (Hg (0)), ethyl mercury, and dimethyl mercury. Monomethyl, ethyl, and dimethyl mercury are organomercury species. The concentration values for the organomercury species ethyl mercury and dimethyl mercury are calculated from the Hg speciation data on a mg Hg/L basis.<sup>10</sup> As a sample calculation for dimethyl mercury, information from Reference 10 shows that the reported average dimethyl mercury concentration on a mg Hg/L basis is 0.00908 mg Hg/L. This value is then multiplied by the formula weight of dimethyl mercury from the WAC<sup>1</sup> (230.7 g dimethyl mercury/mole) divided by the molecular weight of Hg (200.6 g Hg/mole). Thus, the calculated concentration of the species dimethyl mercury is 0.00908 mg Hg/L x (230.7 g dimethyl mercury/mole / 200.6 g Hg/mole) = 0.0104 mg dimethyl mercury/L.

## 2.2 Quality Assurance

Quality Assurance requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60.<sup>16</sup> SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.<sup>17</sup> The customer requested that a

Functional Classification of Safety Significant apply to this work.<sup>2</sup> Data collection and analysis methods used in this work comply with this requirement as detailed in the TTQAP.<sup>3</sup>

### 3.0 Results and Discussion

Analyzed nonradionuclide chemical concentrations, their standard deviations and their corresponding WAC Limits<sup>1</sup> are shown in Table 3-1 that correspond to the Attachment 8.1 Limits in the WAC.<sup>1</sup> Per the WAC, the Limits shown shall not be exceeded accounting for the analytical uncertainty in each measured concentration.<sup>1</sup> Analyzed nonradionuclide chemical concentrations, their standard deviations and their corresponding WAC Targets<sup>1</sup> are shown in Table 3-2 that correspond to the Attachment 8.2 Targets in the WAC.<sup>1</sup> Per the WAC, the Targets shown shall not be exceeded accounting for the analytical uncertainty in each measured concentration.<sup>1</sup> The Limits refer to a type of acceptance criteria that, if not satisfied, will have an adverse impact on repository requirements, whereas the Targets refer to a type of acceptance criteria that is set as a guideline to protect a Limit.<sup>1</sup> For the chemical contaminants and the radionuclides given in tables below, an analytical uncertainty of 2 sigma ( $2\sigma$ ) shall be accounted for in sample analyses used to determine the analytical uncertainty vs. either the Limit or Target. The standard deviations given in tables for this WAC report are taken as 1 sigma ( $1\sigma$ ) values that are calculated from the normal 'standard deviation' function for either duplicate or triplicate values from within Excel<sup>®</sup> spreadsheets.

Comparison of the average analyzed detectable values shown in Table 3-1 to the WAC Limits indicates that free hydroxide and nitrate anions are the highest analytes relative to the WAC Limits at 20% and 26%, respectively. Comparison of the average analyzed values shown in Table 3-2 to the WAC Targets indicates that aluminum is the highest analyte relative to the WAC Target at 68%, with average TOC at a lower ratio to WAC Target of 26%. Good agreement for both total Hg and monomethyl mercury was obtained between the two analytical laboratories, i.e. the numbers are the same within analytical uncertainty. SRNL AD analysis indicates a total Hg average value of  $59.8 \text{ mg/L} \pm 0.4 \text{ mg/L}$  compared to the Eurofins 12% higher average value of  $67.4 \text{ mg/L} \pm 3.8 \text{ mg/L}$ . The uncertainty associated with the AD and Eurofins total Hg analysis is 20%, so these reported average total Hg values are equivalent to within the method uncertainty. SRNL AD analysis of monomethyl mercury indicates a monomethyl mercury average value of  $26.2 \text{ mg/L} \pm 1.65 \text{ mg/L}$  compared to the Eurofins average value of  $25.8 \text{ mg monomethyl mercury/L} \pm 1.57 \text{ mg/L}$  that is calculated from the reported average monomethyl mercury of  $24.0 \text{ mg Hg/L}$ .<sup>10</sup> The uncertainty associated with the AD and Eurofins monomethyl mercury analysis is 10% and 30%, respectively, so these reported average total Hg values are equivalent to within the method uncertainty. No VOA analytes (butanol, propanol, benzene and toluene) were detected above the indicated method detection limits from duplicate analyses as shown in Table 3-1 and Table 3-2.

Analyzed radionuclide concentrations and the respective radiochemical analysis methods, their standard deviations and their corresponding WAC Limits and Targets are shown in Table 3-3 and Table 3-4, respectively. These tables correspond to Attachment 8.3 Limits and Attachment 8.4 Targets, respectively, from the WAC.<sup>1</sup> The minimum detection limit reported for <sup>94</sup>Nb of  $<1.97\text{E-}01 \text{ pCi/mL}$  in Table 3-4 is above the requested SRR target minimum detection limit of  $2.0\text{E-}03 \text{ pCi/mL}$ <sup>6</sup> but is lower than the estimated detection limit initially established by SRNL of  $4.38\text{E-}01 \text{ pCi/mL}$  in 2009.<sup>7</sup> It is noted at the bottom of Table 3-4 that one of the triplicate Cm-244 values was reported as  $\sim 70\text{X}$  higher than the other two replicates. However, the average Cm-244 value from all three replicates of  $1.70\text{E+}01 \text{ pCi/mL}$  is still three orders of magnitude lower than the WAC target of  $6.67\text{E+}04 \text{ pCi/mL}$ . It is unlikely that the Tank 50 supernate sample was cross-contaminated with Cm-244 from the Shielded Cells which should have rendered all of the aliquots biased high for Cm-244. All of the aliquots were prepared from the same Tank 50 parent sample in a radiochemical hood after removal from the Shielded Cells.

Comparison of the average analyzed detectable values shown in Table 3-3 to the WAC Limits indicates that I-129 is the highest analyte relative to the WAC Limit at 44%. Comparison of the average analyzed

detectable values shown in Table 3-4 to the WAC Targets indicates that Pu-238 and total alpha are the highest analytes relative to the WAC Targets at 37% and 32%, respectively. Similar trends were observed for the previous Fourth Quarter 2018 Tank 50 sample for I-129 being the highest analyte relative to the WAC Limit at 44%, and for both Pu-238 and total alpha at 31% and 30% of the WAC Targets, respectively.<sup>18</sup>

Isopar L has a higher detection limit<sup>4</sup> compared with the current SPF WAC Limit value of 11 ppm<sup>1</sup> shown in Table 3-5 associated with flammability that has been in effect since revision 12 of the WAC dating back to July of 2013.<sup>5</sup>

**Table 3-1. Chemical Contaminants from First Quarter CY19 Tank 50 Samples and SPF WAC, Revision 18, Attachment 8.1 Limits<sup>1</sup>**

<b>Chemical Name (Formula)</b>	<b>Method</b>	<b>Average Concentration (mg/L)</b>	<b>Std. Dev.</b>	<b>WAC Limit (mg/L)</b>
Aluminate (Al(OH) <sub>4</sub> <sup>-</sup> )	ICP-ES	1.68E+04 <sup>a</sup>	6.79E+02	4.08E+05
Ammonium (NH <sub>4</sub> <sup>+</sup> )	IC	<1.00E+02	NA	2.12E+02
Carbonate (CO <sub>3</sub> <sup>2-</sup> )	TIC	1.44E+04 <sup>b</sup>	7.63E+01	1.20E+05
Chloride (Cl <sup>-</sup> )	IC	4.72E+02	1.55E+01	7.95E+03
Fluoride (F <sup>-</sup> )	IC	<1.00E+02	NA	4.07E+03
Free Hydroxide (OH <sup>-</sup> )	Total Base	3.19E+04 <sup>b</sup>	2.60E+02	1.58E+05
Nitrate (NO <sub>3</sub> <sup>-</sup> )	IC	1.13E+05	7.77E+03	4.37E+05
Nitrite (NO <sub>2</sub> <sup>-</sup> )	IC	2.25E+04	1.53E+03	2.14E+05
Oxalate (C <sub>2</sub> O <sub>4</sub> <sup>2-</sup> )	IC	5.57E+02	2.05E+01	2.72E+04
Phosphate (PO <sub>4</sub> <sup>3-</sup> )	IC	2.73E+02	9.71E+00	3.14E+04
Sulfate (SO <sub>4</sub> <sup>2-</sup> )	IC	3.84E+03	1.53E+02	5.69E+04
Arsenic (As)	ICP-MS	<1.96E-01	NA	1.97E+02
Barium (Ba)	ICP-ES	7.00E+00 <sup>c</sup>	7.68E-01	6.19E+02
Cadmium (Cd)	ICP-ES	<1.48E+00	NA	3.10E+02
Chromium (Cr)	ICP-ES	4.90E+01	2.36E+00	1.50E+03
Lead (Pb)	ICP-MS	3.83E-01	4.33E-02	7.50E+02
Total Mercury (Hg)	DMA	5.98E+01	4.04E-01	3.25E+02
Elemental Mercury (Hg(0))	CVAFS	2.31E+00 <sup>d</sup>	2.36E+00	3.25E+02
Monomethyl Mercury (CH <sub>3</sub> Hg)	CVAFS	2.62E+01	1.65E+00	3.50E+02
Ethyl Mercury (C <sub>2</sub> H <sub>5</sub> Hg)	CVAFS w/ Distillation	<1.01E+00 <sup>d</sup>	NA	3.73E+02
Selenium (Se)	ICP-MS	<1.96E-01	NA	3.75E+02
Silver (Ag)	ICP-ES	<2.12E+00	NA	6.19E+02
Aluminum (Al)	ICP-ES	4.78E+03	1.93E+02	1.16E+05
Potassium (K)	ICP-ES	2.90E+02	2.22E+01	3.03E+04
Butanol (C <sub>4</sub> H <sub>9</sub> OH)	VOA	<5.00E-01 <sup>e</sup>	NA	7.73E+00
Propanol (C <sub>3</sub> H <sub>7</sub> OH)	VOA	<2.50E-01 <sup>e</sup>	NA	1.88E+00
Phenol (C <sub>6</sub> H <sub>5</sub> OH)	SVOA	<1.00E+01 <sup>e</sup>	NA	7.50E+02
Tetraphenylborate [TPB] (B(C <sub>6</sub> H <sub>5</sub> ) <sub>4</sub> <sup>-</sup> )	HPLC	<5.00E+00	NA	5.00E+00
Total Organic Carbon (----)	TOC	1.92E+02 <sup>b</sup>	2.08E+00	4.50E+03
Isopar L (----)	SVOA	<3.30E+01 <sup>e</sup>	NA	8.75E+01 <sup>f</sup>

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

b. Measurement performed on filtered supernate samples.

c. A similar value was also determined for the blank so the average value reported is likely high biased.

d. Mercury species calculated from data presented in Reference 10.

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

f. The WAC Limit shown in this table is based on bounding DSA concentrations for accident consequence analysis. A more restrictive limit for Isopar L is set to protect assumptions associated with flammability as shown in Table 3-5 below.<sup>1</sup>

**Table 3-2. Chemical Contaminants from First Quarter CY19 Tank 50 Samples and SPF WAC, Revision 18, Attachment 8.2 Targets<sup>1</sup>**

<b>Chemical Name (Formula)</b>	<b>Method</b>	<b>Average Concentration (mg/L)</b>	<b>Std. Dev.</b>	<b>WAC Target (mg/L)</b>
<b>Aluminum (Al)</b>	ICP-ES	4.78E+03	1.93E+02	<b>7.00E+03<sup>f</sup></b>
<b>Boron (B)</b>	ICP-ES	4.49E+01	1.61E+00	<b>7.43E+02</b>
<b>Cobalt (Co)</b>	ICP-MS <sup>a</sup>	<2.94E-02	NA	<b>1.45E+02</b>
<b>Copper (Cu)</b>	ICP-ES	<8.12E-01	NA	<b>7.43E+02</b>
<b>Iron (Fe)</b>	ICP-ES	2.86E+01 <sup>b</sup>	NA	<b>4.95E+03</b>
<b>Lithium (Li)</b>	ICP-ES	<9.61E+00	NA	<b>7.43E+02</b>
<b>Manganese (Mn)</b>	ICP-ES	<3.28E+00	NA	<b>7.43E+02</b>
<b>Molybdenum (Mo)</b>	ICP-ES	<2.21E+01	NA	<b>7.43E+02</b>
<b>Nickel (Ni)</b>	ICP-ES	<4.11E+00	NA	<b>7.43E+02</b>
<b>Silicon (Si)</b>	ICP-ES	<3.28E+01	NA	<b>1.07E+04</b>
<b>Strontium (Sr)</b>	ICP-ES	4.07E-01	2.87E-01	<b>7.43E+02</b>
<b>Zinc (Zn)</b>	ICP-ES	1.26E+01 <sup>b</sup>	NA	<b>8.03E+02</b>
<b>Dimethyl Mercury ((CH<sub>3</sub>)<sub>2</sub>Hg)</b>	CVAFS	1.04E-02 <sup>c</sup>	5.74E-04	<b>1.00E+00</b>
<b>Benzene (C<sub>6</sub>H<sub>6</sub>)</b>	VOA	<1.50E-01 <sup>d</sup>	NA	<b>3.10E+02</b>
<b>Methanol (CH<sub>3</sub>OH)</b>	VOA	e	NA	<b>1.88E+00</b>
<b>Toluene (C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub>)</b>	VOA	<1.50E-01 <sup>d</sup>	NA	<b>3.10E+02</b>
<b>Dibutylphosphate [DBP] (C<sub>8</sub>H<sub>19</sub>O<sub>4</sub>P)</b>	IC	<2.50E+02	NA	<b>3.47E+02</b>
<b>Tributylphosphate [TBP] ((C<sub>4</sub>H<sub>9</sub>O)<sub>3</sub>PO)</b>	SVOA	<7.50E-01 <sup>d</sup>	NA	<b>7.50E+00</b>
<b>Total Organic Carbon (TOC)</b>	TOC	1.92E+02	2.08E+00	<b>7.50E+02<sup>e</sup></b>
<b>EDTA (C<sub>10</sub>H<sub>12</sub>N<sub>2</sub>O<sub>8</sub><sup>4-</sup>)</b>	HPLC	<1.00E+02	NA	<b>3.10E+02</b>
<b>NORPAR 13 (C<sub>n</sub>H<sub>2n</sub>)</b>	SVOA	<7.50E-01 <sup>d</sup>	NA	<b>7.50E-01</b>
<b>Formate (CHOO<sup>-</sup>)</b>	IC	1.83E+02	5.57E+00	<b>6.38E+03</b>

a. Cobalt based on the stable Co-59 isotope.

b. Only one detectable value from the analyzed triplicate set.

c. Mercury species calculated from data presented in Reference **Error! Bookmark not defined.**

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

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

f. The WAC Targets for Al and TOC shown in this table are more restrictive than the corresponding WAC Limits shown in Table 3-1 to protect assumptions associated with thermolytic hydrogen generation.<sup>1</sup>

**Table 3-3. Radionuclide Contaminants from First Quarter CY19 Tank 50 Samples and SPF WAC,  
Revision 18, Attachment 8.3 Limits<sup>1</sup>**

<b><u>Radionuclide</u></b>	<b><u>Method</u></b>	<b><u>Average Concentration</u> (pCi/mL)</b>	<b><u>Std. Dev.</u></b>	<b><u>WAC Limit</u> (pCi/mL)</b>
<b>Tritium (<sup>3</sup>H)</b>	Tritium Counting	1.16E+03	1.45E+02	<b>5.63E+05</b>
<b>Carbon-14 (<sup>14</sup>C)</b>	C-14 Liquid Scintillation	3.47E+02	7.46E+00	<b>1.13E+05</b>
<b>Nickel-63 (<sup>63</sup>Ni)</b>	Ni-59/63	<7.12E+00	NA	<b>1.13E+05</b>
<b>Strontium-90 (<sup>90</sup>Sr)</b>	Sr-90 Liquid Scintillation	2.91E+04	1.78E+03	<b>2.62E+06</b>
<b>Technetium-99 (<sup>99</sup>Tc)</b>	Tc-99 Liquid Scintillation	4.29E+04	6.98E+02	<b>2.11E+05</b>
<b>Iodine-129 (<sup>129</sup>I)</b>	I-129 (w/ separation) Liquid Scintillation	2.75E+01	1.31E+00	<b>6.30E+01</b>
<b>Cesium-137 (<sup>137</sup>Cs)</b>	Gamma Scan	6.68E+05	5.34E+04	<b>3.96E+06</b>
<b>Uranium-233 (<sup>233</sup>U)</b>	ICP-MS	< 1.90E+02	NA	<b>1.13E+04</b>
<b>Uranium-235 (<sup>235</sup>U)</b>	ICP-MS	1.87E-01	4.31E-03	<b>1.13E+02</b>
<b>Plutonium-241 (<sup>241</sup>Pu)</b>	Pu238/241 Liquid Scintillation	7.94E+03	4.19E+02	<b>8.38E+05</b>
<b>Total Alpha<sup>a</sup></b>	Liquid Scintillation Counting (Cs removed)	2.14E+04	1.40E+03	<b>2.13E+05</b>

a. Only two detectable values from the analyzed triplicate set



**Table 3-4. Radionuclide Contaminants from First Quarter CY19 Tank 50 Samples and SPF WAC,  
Revision 18, Attachment 8.4 Targets<sup>1</sup>**

<b>Radionuclide</b>	<b>Method</b>	<b>Average Concentration (pCi/mL)</b>	<b>Std. Dev.</b>	<b>WAC Target (pCi/mL)</b>
Aluminum-26 ( <sup>26</sup> Al)	Gamma Scan (Cs removed)	1.58E-01 <sup>a</sup>	N/A	2.88E+03
Potassium-40 ( <sup>40</sup> K)	Gamma Scan (Cs removed)	<1.34E+00	NA	1.00E+02
Cobalt-60 ( <sup>60</sup> Co)	Gamma Scan (Cs removed)	<1.42E-01	NA	9.75E+02
Nickel-59 ( <sup>59</sup> Ni)	Ni-59/63	<5.27E+00	NA	1.13E+03
Selenium-79 ( <sup>79</sup> Se)	Se-79	2.15E+01	2.40E+00	1.90E+04
Yttrium-90 ( <sup>90</sup> Y)	Secular Equilibrium w/ 100% of Sr-90	2.91E+04	1.78E+03	2.62E+06
Zirconium-93 ( <sup>93</sup> Zr)	ICP-MS	<7.40E+01	NA	1.00E+05
Niobium-94 ( <sup>94</sup> Nb)	Nb-94	<1.97E-01	NA	1.53E+02
Rhodium-106 ( <sup>106</sup> Rh)	Secular Equilibrium w/ 100% of Ru-106	<1.68E+00	NA	3.12E+05
Ruthenium-106 ( <sup>106</sup> Ru)	Gamma Scan (Cs removed)	<1.68E+00	NA	3.12E+05
Antimony-125 ( <sup>125</sup> Sb)	Gamma Scan (Cs removed)	7.24E+00	7.01E-01	7.99E+03
Tellurium-125m ( <sup>125m</sup> Te)	Secular Equilibrium w/ 100% of Sb-125	7.24E+00	7.01E-01	1.83E+03
Tin-126 ( <sup>126</sup> Sn)	Gamma Scan (Cs removed)	4.36E+02	4.75E+01	1.80E+04
Cesium-134 ( <sup>134</sup> Cs)	Gamma Scan	<6.62E+01	NA	1.82E+04
Cesium-135 ( <sup>135</sup> Cs)	Cs-135	3.60E+00	4.02E-01	2.50E+02
Barium-137m ( <sup>137m</sup> Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137)	6.32E+05	5.05E+04	3.75E+06
Cerium-144 ( <sup>144</sup> Ce)	Gamma Scan (Cs removed)	<3.13E+00	NA	3.12E+04
Praseodymium-144 ( <sup>144</sup> Pr)	Secular Equilibrium w/ 100% of Ce-144	<3.13E+00	NA	3.12E+04
Promethium-147 ( <sup>147</sup> Pm)	Pm-147/Sm-151 Liquid Scintillation	<3.52E+01	NA	1.57E+06
Samarium-151 ( <sup>151</sup> Sm)	Pm-147/Sm-151 Liquid Scintillation	<3.81E+01	NA	2.25E+04
Europium-154 ( <sup>154</sup> Eu)	Gamma Scan (Cs removed)	<4.43E-01	NA	1.62E+03
Radium-226 ( <sup>226</sup> Ra)	Ra-226	<2.22E+00	NA	1.00E+03
Radium-228 ( <sup>228</sup> Ra)	Gamma Scan (Cs removed)	<1.01E+00	NA	1.00E+04
Actinium-227 ( <sup>227</sup> Ac)	Th-229/230	<7.84E-03	NA	1.00E+04
Thorium-229 ( <sup>229</sup> Th)	Th-229/230	<1.44E+00	NA	1.63E+05
Thorium-230 ( <sup>230</sup> Th)	Th-229/230	<4.01E-01	NA	6.26E+03
Thorium-232 ( <sup>232</sup> Th)	ICP-MS	<3.23E-03	NA	2.88E+03
Protactinium-231 ( <sup>231</sup> Pa)	Pa-231	<5.68E-01	NA	1.00E+03
Uranium-232 ( <sup>232</sup> U)	U-232	2.07E+00	1.41E-01	2.27E+03
Uranium-233 ( <sup>233</sup> U)	ICP-MS	<1.90E+02	NA	3.12E+03
Uranium-234 ( <sup>234</sup> U)	ICP-MS	<1.23E+02	2.84E+00	3.12E+03
Uranium-236 ( <sup>236</sup> U)	ICP-MS	<1.27E+00	NA	3.12E+03
Uranium-238 ( <sup>238</sup> U)	ICP-MS	3.12E+00	9.25E-02	3.12E+03

**Table 3-4. Radionuclide Contaminants from First Quarter CY19 Tank 50 Samples and SPF WAC, Revision 18, Attachment 8.4 Targets<sup>1</sup>, continued**

<u>Radionuclide</u>	<u>Method</u>	<u>Average Concentration (pCi/mL)</u>	<u>Std. Dev.</u>	<u>WAC Target (pCi/mL)</u>
Neptunium-237 ( <sup>237</sup> Np)	ICP-MS	<1.38E+01	NA	1.00E+04
Plutonium-238 ( <sup>238</sup> Pu)	Pu238/241 Pu alpha PHA	2.49E+04	1.25E+03	6.67E+04
Plutonium-239 ( <sup>239</sup> Pu)	Pu238/241 Pu alpha PHA	6.44E+02	2.81E+01	6.67E+04
Plutonium-240 ( <sup>240</sup> Pu)	Pu238/241 Pu alpha PHA	6.44E+02	2.81E+01	6.67E+04
Plutonium-242 ( <sup>242</sup> Pu)	ICP-MS	<7.49E+01	NA	6.67E+04
Plutonium-244 ( <sup>244</sup> Pu)	ICP-MS	<3.48E-01	NA	7.02E+04
Americium-241 ( <sup>241</sup> Am)	Am/Cm	3.05E+00	4.56E-01	6.67E+04
Americium-242m ( <sup>242m</sup> Am)	Am/Cm	<1.20E-02	NA	4.50E+05
Americium-243 ( <sup>243</sup> Am)	Am/Cm	1.72E-01 <sup>a</sup>	NA	6.67E+04
Curium-242 ( <sup>242</sup> Cm)	Am/Cm	<9.95E-03	NA	1.13E+04
Curium-244 ( <sup>244</sup> Cm)	Am/Cm	1.70E+01 <sup>b</sup>	2.82E+01	6.67E+04
Curium-245 ( <sup>245</sup> Cm)	Am/Cm	<3.10E-01	NA	2.25E+05
Total Alpha	Liquid Scintillation Counting (Cs removed)	2.14E+04 <sup>c</sup>	1.40E+03	6.67E+04

- a. Only one detectable value from the analyzed triplicate set.  
b. One Cm-244 measurement was ~ 70X the other two measurements. If only two replicates are included in the calculation, the average Cm-244 value is 7.25E-01 ± 5.73E-02 pCi/mL.  
c. Only two detectable values from the analyzed triplicate set.

The following tables show various chemical contaminants (Table 3-5), organic species (Table 3-6) and processing constituents (Table 3-7) related to the Saltstone Disposal Unit (SDU) that are referred to in the WAC per Tables 2, 3 and 4, respectively.<sup>1</sup>

**Table 3-5. Chemical Contaminants Impacting Saltstone Disposal Unit (SDU) Flammability from First Quarter CY19 Tank 50 Samples and SPF WAC, Revision 18, Table 2 Limits and Targets<sup>1</sup>**

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Limit/Target</u>
Isopar L (----)	SVOA	<2.67E+01 ppm <sup>a,b</sup>	NA	1.10E+01 ppm (Limit)
Tetraphenylborate [TPB] (B(C <sub>6</sub> H <sub>5</sub> ) <sub>4</sub> <sup>-</sup> )	HPLC	<5.00E+00	NA	5.00E+00 mg/L (Limit)
Ammonium (NH <sub>4</sub> <sup>+</sup> )	IC	<1.00E+02	NA	2.12E+02 mg/L (Limit)
Total Mercury (Hg)	DMA	5.98E+01	4.04E-01	3.25E+02 mg/L (Limit)
Monomethyl Mercury (CH <sub>3</sub> Hg)	CVAFS	2.62E+01	1.65E+00	3.50E+02 mg/L (Limit)
Dimethyl Mercury ((CH <sub>3</sub> ) <sub>2</sub> Hg)	CVAFS	1.04E-02 <sup>c</sup>	5.74E-04	1.00E+00 mg/L (Target)

- 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 of the slurry sample listed in Table 3-8.  
c. Mercury species calculated from data presented in Reference **Error! Bookmark not defined.**

**Table 3-6. Other Organics Impacting SDU Flammability from First Quarter CY19 Tank 50 Samples and SPF WAC, Revision 18, Table 3 Concentrations<sup>1</sup>**

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Concentrations (mg/L)</u>
Butanol (C <sub>4</sub> H <sub>9</sub> OH) <sup>a</sup>	VOA	<5.00E-01	NA	0.75
Tributylphosphate[TBP] ((C <sub>4</sub> H <sub>9</sub> O) <sub>3</sub> PO) <sup>a</sup>	SVOA	<7.50E-01	NA	1.0
Propanol (C <sub>3</sub> H <sub>7</sub> OH) <sup>a</sup>	VOA	<2.50E-01	NA	0.25
Methanol (CH <sub>3</sub> OH)	b	NA	NA	0.05
NORPAR 13 (C <sub>n</sub> H <sub>2.n</sub> ) <sup>a</sup>	SVOA	<7.50E-01	NA	0.75

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 3-7. Processing Constituents from First Quarter CY19 Tank 50 Samples and SPF WAC, Revision 18, Table 4 Limits<sup>1</sup>**

<u>Processing Constituents</u>	<u>Method</u>	<u>Value</u>	<u>Std. Dev.</u>	<u>WAC Limit</u>
pH	Calculated	>13	NA	> 10
Sodium Concentration	ICP-ES	5.38 M	1.26E-01	2.5 M < [Na <sup>+</sup> ] < 7.0 M
Total Insoluble Solids	Calculated	~0 wt %	NA	< 15 wt %

Table 3-8 contains additional measured constituents per the TTQAP.<sup>3</sup> There were no detectable nitrosamine species in the Tank 50 surface sample via the SVOA analyses shown in Table 3-8 as was the case in the previous Fourth Quarter 2018 Tank 50 sample.<sup>18</sup>

**Table 3-8. Additional Measured Constituents<sup>3</sup>**

<b><u>Constituent</u></b>	<b><u>Method</u></b>	<b><u>Average Value</u></b>	<b><u>Std. Dev.</u></b>
<b>Density (slurry)</b>	Measured (21.9°C)	1.2339 g/mL	0.0007
<b>Specific Gravity</b>	a	1.2369	0.0007
<b>Total Solids</b>	Measured	26.90 wt %	0.08
<b>Total Beta</b>	LSC	8.84E+05 pCi/mL	9.38E+04
<b>Total Gamma</b>	b	6.33E+05 pCi/mL	7.68E+03 <sup>c</sup>
<b>Beryllium (Be)</b>	ICP-ES	<7.23E-01 mg/L	NA
<b>N-Nitrosodimethylamine (C<sub>2</sub>H<sub>6</sub>N<sub>2</sub>O)</b>	SVOA	<1 mg/L	NA
<b>N-Dioctylnitrosamine (C<sub>16</sub>H<sub>34</sub>N<sub>2</sub>O)</b>	SVOA	<1 mg/L	NA

a. Calculated from the measured density of slurry and density of water at 23.0 °C.<sup>19</sup>

b. Calculated from the sum of gamma emitters (Sb-126, Sn-126, Sb-125, Eu-154, Am-241, Co-60 and Ba-137m).

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

## 4.0 Conclusions

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

- WAC Targets or Limits were met for all analyzed chemical and radioactive contaminants for which the detection limits are below the WAC Targets or Limits.
- Isopar L has a higher detection limit<sup>4</sup> compared with the current SPF WAC Limit value of 11 ppm<sup>1</sup> associated with flammability that has been in effect since revision 12 of the WAC dating back to July of 2013.<sup>5</sup>
- Nitrosamines were not detected in the Tank 50 salt solution surface sample above the instrument detection limits of < 1 mg/L.
- The minimum detection limit (<1.97E-01 pCi/mL) is reported for <sup>94</sup>Nb as determined from the minimum detectable activity associated with the radiochemical method used for this radionuclide. The reported detection limit is above the requested SRR target minimum detection limit concentration.<sup>6</sup> However, the minimum detection limit reported for the First Quarter CY19 Tank 50 sample for <sup>94</sup>Nb is lower than the estimated detection limit of 4.38E-01 pCi/mL initially established by SRNL in 2009.<sup>7</sup> Thus per guidance from SRR,<sup>6</sup> SRNL continues to achieve as low as practical detection limits for this radionuclide.

## 5.0 Reference

- <sup>1</sup> Brown, M. K., "Waste Acceptance Criteria for Aqueous Waste Sent to the Z-Area Saltstone Production Facility", Savannah River Remediation, X-SD-Z-00001, Rev. 18, November 2018.
- <sup>2</sup> Ray, J. W., "Routine Saltstone Support for Salt Solution and Grout Analyses – FY2019", Savannah River Remediation, X-TTR-Z-00013, Rev. 1, November 2018.
- <sup>3</sup> Crawford, C. L., Hill, K. A., "Task Technical and Quality Assurance Plan for Salt Solution and Grout Sample Preparation and Analyses – FY2019", Savannah River National Laboratory, SRNL-RP-2018-01082, Rev. 0, December 2018.
- <sup>4</sup> Crump, S. L., "Determination of Method Reporting Limits for Select Analytes by GC/MS", Savannah River National Laboratory, SRNL-TR-2010-00206, Rev. 0, October 2010.
- <sup>5</sup> Potvin, M. M., "Waste Acceptance Criteria for Aqueous Waste Sent to the Z-Area Saltstone Production Facility", Savannah River Remediation, X-SD-Z-00001, Rev. 12, July 2013.
- <sup>6</sup> Dixon, D. B., "Minimum Detection Limits for Saltstone Quarterly WAC Analyses", Savannah River Remediation, SRR-WSE-2013-00005, Rev. 1, January 2013.
- <sup>7</sup> DiPrete, C. C., "Overview of Capability to Measure Radionuclides of Interest for Saltstone", Savannah River National Laboratory, SRNL-L4000-2009-00028, Rev. 0, June 2009.
- <sup>8</sup> Crawford, C. L., "Results for the First Quarter Calendar Year 2019 Tank 50 Salt Solution Sample: Cs-137", Savannah River National Laboratory, SRNL-L3100-2019-00012, Rev.0, April 2019.
- <sup>9</sup> Crawford, C. L., "1Q CY19 Tank 50 WAC Characterization", B9108-00327-02, SRNL E-Notebook (Production), Savannah River National Laboratory, January 2019.
- <sup>10</sup> Bannochie, C. J., "Results of Hg Speciation Testing on the 1Q19 Tank 50 Sample", Savannah River National Laboratory, SRNL-L3300-2019-00018, Rev. 0, April 2019.
- <sup>11</sup> "Gas Chromatography/Mass Spectrometry for Volatile Organics: Contract Laboratory Program Methods", Manual L16.1, Procedure ADS-2656, Rev. 9, October 2016.
- <sup>12</sup> "Gas Chromatography/Mass Spectrometry for Semivolatile Organics Including Polychlorinated Biphenyls", Manual L16.1, Procedure ADS-2657, Rev. 7, May 2016.
- <sup>13</sup> "Aqua Regia Dissolution of Sludge for Elemental Analysis"; Savannah River National Laboratory, Manual L16.1, Procedure ADS-2226, Rev. 10, July 2013.
- <sup>14</sup> "Weight Percent Solids Determination Using a Furnace or Oven", Savannah River National Laboratory, Manual L29, Procedure ITS-0078, Rev. 1, October 2012.
- <sup>15</sup> "Integrated Data Base Report – 1996: U.S. Spent Nuclear Fuel and Radioactive Waste Inventories, Projections, and Characteristics", Oak Ridge National Laboratory, DOE/RW-0006, Rev. 13, December 1997. <https://www.nrc.gov/docs/ML1028/ML102850100.pdf> (accessed April 5, 2019).
- <sup>16</sup> "Technical Reviews", Savannah River Site, Manual E7, Procedure 2.60, Latest Revision.
- <sup>17</sup> "Savannah River National Laboratory Technical Report Design Check Guidelines", Westinghouse Savannah River Company, WSRC-IM-2002-00011, Rev. 2, August 2004.

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- <sup>18</sup> Crawford, C. L., “Results of the Fourth Quarter Calendar Year 2018 Tank 50 Salt Solution Sample”, SRNL-STI-2019-00148, Rev. 0, May 2019.
- <sup>19</sup> *CRC Handbook of Chemistry and Physics*, 99th Print Edition, Internet Version 2018; Section 6: Fluid Properties. Edited by Rumble, J. R., CRC Press Taylor and Francis Group, Boca Raton, FL, Internet Version 2018. <http://hbcponline.com/faces/contents/ContentsSearch.xhtml> (accessed March 12, 2019).

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