

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).

Disclaimer:

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U. S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

- 1) warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
- 2) representation that such use or results of such use would not infringe privately owned rights; or
- 3) endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.



Results for the Third Quarter Calendar Year 2018 Tank 50 Salt Solution Sample

C. L. Crawford

January 2019

SRNL-STI-2018-00650, Revision 0



DISCLAIMER

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

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

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

Retention: *Permanent*

Results for the Third Quarter Calendar Year 2018 Tank 50 Salt Solution Sample

C. L. Crawford

January 2019

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



REVIEWS AND APPROVALS

AUTHORS:

C. L. Crawford, Author, Advanced Characterization and Processing	Date
------------------------------------------------------------------	------

TECHNICAL REVIEW:

J. H. Christian, Advanced Characterization and Processing, Reviewed per E7 2.60	Date
---------------------------------------------------------------------------------	------

APPROVAL:

B. J. Wiedenman, Manager Advanced Characterization and Processing	Date
----------------------------------------------------------------------	------

S. D. Fink, Acting Director Chemical Processing Technologies	Date
-----------------------------------------------------------------	------

E. J. Freed, Manager DWPF and Saltstone Facility Engineering	Date
-----------------------------------------------------------------	------

R. E. Edwards, Manager Nuclear Safety and Engineering Integration	Date
----------------------------------------------------------------------	------

EXECUTIVE SUMMARY

In this Technical Report, the chemical and radionuclide contaminant results from the Third Quarter Calendar Year 2018 (CY18) 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, where the waste will be treated and disposed in the Saltstone Disposal Facility. This Technical Report compares results, where applicable, to Saltstone Production Facility (SPF) Waste Acceptance Criteria (WAC) limits and targets.¹ The chemical and radionuclide contaminant results from the characterization of the Third Quarter CY18 sampling of Tank 50 were requested by SRR personnel via a Task Technical Request (TTR)² and details of the testing are presented in the Savannah River National Laboratory (SRNL) Task Technical and Quality Assurance Plan (TTQAP).³ This Technical Report is part of Deliverable 2 relating to Task 1 from the SRR request.² 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.³

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 has a higher detection limit⁴ compared with the current SPF WAC¹ value of 11 ppm that has been in effect since revision 12 of the WAC dating back to July of 2013.⁵
- Nitrosamines were not detected in the Tank 50 salt solution sample above the instrument detection limits of < 1 mg/L.
- The minimum detection limit is reported for ⁹⁴Nb 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.⁶ However, the minimum detection limit reported for the Third Quarter CY18 Tank 50 sample for ⁹⁴Nb is within 12% of the estimated detection limit initially established by SRNL in 2009.⁷ Thus per guidance from SRR,⁶ SRNL continues to achieve as low as practical detection limits for this radionuclide.

TABLE OF CONTENTS

LIST OF TABLES	vii
LIST OF ABBREVIATIONS	viii
1.0 Introduction	1
2.0 Experimental	1
2.1 Technical	1
2.2 Quality Assurance	2
3.0 Results and Discussion	2
4.0 Conclusions	8
5.0 Reference	9

LIST OF TABLES

Table 3-1. Chemical Contaminants from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Attachment 8.1 Limits ¹	3
Table 3-2. Chemical Contaminants from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Attachment 8.2 Targets ¹	4
Table 3-3. Radionuclide Contaminants from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Attachment 8.3 Limits ¹	4
Table 3-4. Radionuclide Contaminants from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Attachment 8.4 Targets ¹	5
Table 3-5. Chemical Contaminants Impacting Saltstone Disposal Unit (SDU) Flammability from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Table 2 Limits and Targets ¹	6
Table 3-6. Other Organics Impacting SDU Flammability from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Table 3 Concentrations ¹	7
Table 3-7. Processing Constituents from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Table 4 Limits ¹	7
Table 3-8. Additional Measured Constituents ³	8

LIST OF ABBREVIATIONS

AD	Analytical Development
CVAFS	Cold Vapor Atomic Fluorescence Spectroscopy
EDTA	ethylenediaminetetraacetate
HDPE	High-Density Polyethylene
HPLC	High Performance Liquid Chromatography
ICP-ES	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
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).¹ The quarterly Regulatory Compliance samples pulled in Tank 50 should be characterized for both Limit and Target acceptance criteria in this WAC.¹ 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.¹ A memorandum reporting the average Cs-137 value has been previously issued.⁸

2.0 Experimental

2.1 Technical

The Third Quarter CY18 Tank 50 samples [a 200-mL sample obtained 6" below the surface (HTF-50-18-74) and a 3-L variable depth sample (VDS) obtained 66" from the tank bottom (HTF-50-18-73)] were obtained on September 20, 2018 and received at Savannah River National Laboratory (SRNL) on September 20, 2018.⁹

The contents of the 3-L slurry in the steel variable depth sampler were transferred by pumping into three different high-density polyethylene (HDPE) 1-L bottles. The original 3-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 3-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. One liter of the total received 3-L sample was transferred out of the Shielded Cells and located in a radiochemical hood. The bottle was 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. The other two liters of the VDS were set aside within the Shielded Cells Facility for other testing within SRNL. 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 of the concentrations presented in the tables (except upper limits) are averages based on triplicate analyses of the Third Quarter CY18 Tank 50 samples. 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. 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.¹⁰ 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 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.¹¹

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.¹²

Total mercury was analyzed by SRNL using the ICP-MS method. 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).¹³ These species include elemental mercury (Hg(0)), monomethyl mercury, ethyl mercury, and dimethyl mercury. Monomethyl, ethyl, and dimethyl mercury are organomercury species. The concentration values for the organomercury species are calculated from the Hg speciation data on a mg Hg/L basis.¹³ As a sample calculation for monomethyl mercury, information from Reference 13 shows that the reported average monomethyl concentration on a mg Hg/L basis is 30.7 mg Hg/L. This value is then multiplied by the formula weight of monomethyl mercury from the WAC¹ (215.62 g monomethyl mercury/mole) divided by the molecular weight of Hg (200.6 g Hg/mole). Thus, the calculated concentration of the species monomethyl mercury is $30.7 \text{ mg Hg/L} \times (215.62 \text{ g monomethyl mercury/mole} / 200.6 \text{ g Hg/mole}) = 33.0 \text{ mg monomethyl mercury/L}$.

2.2 Quality Assurance

Quality Assurance requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60.¹⁴ SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.¹⁵

3.0 Results and Discussion

Analyzed nonradionuclide chemical concentrations, their standard deviations and their corresponding WAC Limits¹ are shown in Table 3-1 that correspond to the Attachment 8.1 Limits in the WAC.¹ Per the WAC, the Limits shown shall not be exceeded accounting for the analytical uncertainty in each measured concentration.¹ Analyzed nonradionuclide chemical concentrations, their standard deviations and their corresponding WAC Targets¹ are shown in Table 3-2 that correspond to the Attachment 8.2 Targets in the WAC.¹ Per the WAC, the Targets shown shall not be exceeded accounting for the analytical uncertainty in each measured concentration.¹ 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.¹ For the chemical contaminants and the radionuclides given in tables below, an analytical uncertainty of 2 sigma (2σ) 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 (1σ) values that are calculated from the normal ‘standard deviation’ function for either duplicate or triplicate values from within Excel[®] spreadsheets.

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.¹ The minimum detection limit reported for ⁹⁴Nb of <4.91E-01 pCi/mL in Table 3-4 is above the requested SRR target minimum detection limit of 2.0E-03 pCi/mL⁶ but is within 12% of the estimated detection limit initially established by SRNL of 4.38E-01 pCi/mL in 2009.⁷

**Table 3-1. Chemical Contaminants from Third Quarter CY18 Tank 50 Samples and SPF WAC,
Revision 17, Attachment 8.1 Limits¹**

Chemical Name (Formula)	Method	Average Concentration (mg/L)	Std. Dev.	WAC Limit (mg/L)
Aluminate (Al(OH) ₄ ⁻)	ICP-ES	1.71E+04 ^a	2.06E+02	4.08E+05
Ammonium (NH ₄ ⁺)	IC	<1.00E+02	NA	2.12E+02
Carbonate (CO ₃ ²⁻)	TIC	1.57E+04 ^b	7.63E+01	1.20E+05
Chloride (Cl ⁻)	IC	5.16E+02	2.08E+00	7.95E+03
Fluoride (F ⁻)	IC	<1.00E+02	NA	4.07E+03
Free Hydroxide (OH ⁻)	Total Base	3.29E+04 ^b	2.60E+02	1.58E+05
Nitrate (NO ₃ ⁻)	IC	1.18E+05	5.77E+02	4.37E+05
Nitrite (NO ₂ ⁻)	IC	2.74E+04	5.77E+01	2.14E+05
Oxalate (C ₂ O ₄ ²⁻)	IC	5.33E+02	3.79E+00	2.72E+04
Phosphate (PO ₄ ³⁻)	IC	3.51E+02	6.11E+00	2.94E+04
Sulfate (SO ₄ ²⁻)	IC	4.48E+03	2.08E+01	5.69E+04
Arsenic (As)	ICP-MS	<1.66E-01	NA	2.30E+01
Barium (Ba)	ICP-ES	1.97E+00	2.54E-01	6.19E+02
Cadmium (Cd)	ICP-ES	<1.74E-01	NA	3.10E+02
Chromium (Cr)	ICP-ES	5.46E+01	2.57E+00	1.24E+03
Lead (Pb)	ICP-MS	5.44E-01	3.02E-01	6.19E+02
Total Mercury (Hg)	ICP-MS	6.74E+01	3.98E-01	3.25E+02
Elemental Mercury (Hg(0))	CVAFS	1.64E+00 ^e	5.08E-02	1.82E+01
Monomethyl Mercury (CH ₃ Hg)	CVAFS w/ Distillation	3.30E+01 ^e	1.02E+00	3.50E+02
Ethyl Mercury (C ₂ H ₅ Hg)	CVAFS w/ Distillation	<1.02E+00 ^e	NA	3.73E+02
Selenium (Se)	ICP-MS	<1.66E-01	NA	4.46E+02
Silver (Ag)	ICP-ES	<4.29E-01	NA	6.19E+02
Aluminum (Al)	ICP-ES	4.85E+03	5.84E+01	1.16E+05
Potassium (K)	ICP-ES	4.20E+02	1.47E+01	3.03E+04
Butanol (C ₄ H ₉ OH)	VOA	<5.00E-01 ^c	NA	7.73E+00
Propanol (C ₃ H ₇ OH)	VOA	<2.50E-01 ^c	NA	1.88E+00
Phenol (C ₆ H ₅ OH)	SVOA	<1.00E+01 ^c	NA	7.50E+02
Isopar L (----)	SVOA	<2.67E+01 ppm ^{c,d}	NA	1.10E+01 ppm
Total Organic Carbon (----)	TOC	2.37E+02 ^b	2.31E+00	5.00E+03
Tetraphenylborate [TPB] (B(C ₆ H ₅) ₄ ⁻)	HPLC	<5.00E+00	NA	5.00E+00

- 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. 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 8.
e. Mercury species calculated from data presented in Reference 13.

Table 3-2. Chemical Contaminants from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Attachment 8.2 Targets¹

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Target (mg/L)</u>
Boron (B)	ICP-ES	4.46E+01	2.57E+00	7.43E+02
Cobalt (Co)	ICP-MS ^a	<1.66E-02	NA	1.75E+02
Copper (Cu)	ICP-ES	<6.18E+00	NA	7.43E+02
Iron (Fe)	ICP-ES	9.43E+00	4.44E+00	4.95E+03
Lithium (Li)	ICP-ES	<1.24E+01	NA	7.43E+02
Manganese (Mn)	ICP-ES	<4.14E-02	NA	7.43E+02
Molybdenum (Mo)	ICP-ES	2.49E+01	4.06E+00	7.43E+02
Nickel (Ni)	ICP-ES	<4.14E+00	NA	7.43E+02
Silicon (Si)	ICP-ES	2.05E+01	2.60E+00	1.07E+04
Strontium (Sr)	ICP-ES	<3.42E-02	NA	7.43E+02
Zinc (Zn)	ICP-ES	7.41E+00	5.52E-01	8.03E+02
Benzene (C ₆ H ₆)	VOA	<1.50E-01 ^b	NA	3.10E+02
Methanol (CH ₃ OH)	VOA	c	NA	1.88E+00
Dibutylphosphate [DBP] (C ₈ H ₁₉ O ₄ P)	IC	<2.50E+02	NA	3.47E+02
Tributylphosphate [TBP] (C ₄ H ₉ O) ₃ PO	SVOA	<7.50E-01 ^b	NA	7.50E+00
Toluene (C ₆ H ₅ CH ₃)	VOA	<1.50E-01 ^b	NA	3.10E+02
EDTA (C ₁₀ H ₁₂ N ₂ O ₈ ⁴⁻)	HPLC	<1.00E+02	NA	3.10E+02
NORPAR 13 (C _n H _{2.n})	SVOA	<7.50E-01 ^b	NA	7.50E-01
Dimethyl Mercury (CH ₃) ₂ Hg	CVAFS	2.94E-02 ^d	2.56E-03	1.00E+00

- a. Cobalt based on the stable Co-59 isotope.
b. Measurement performed on duplicate samples rather than triplicate samples.
c. Currently, a routine method for detecting this species does not exist in Analytical Development (AD).
d. Mercury species calculated from data presented in Reference 13.

Table 3-3. Radionuclide Contaminants from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Attachment 8.3 Limits¹

<u>Radionuclide</u>	<u>Method</u>	<u>Average Concentration (pCi/mL)</u>	<u>Std. Dev.</u>	<u>WAC Limit (pCi/mL)</u>
Tritium (³ H)	Tritium Counting	1.22E+03	8.02E+01	5.63E+05
Carbon-14 (¹⁴ C)	C-14 Liquid Scintillation	5.38E+02	7.77E+01	1.13E+05
Nickel-63 (⁶³ Ni)	Ni-59/63	<2.08E+01	NA	1.13E+05
Strontium-90 (⁹⁰ Sr)	Sr-90 Liquid Scintillation	4.48E+04	2.83E+03	3.15E+06
Technetium-99 (⁹⁹ Tc)	Tc-99 Liquid Scintillation	4.50E+04	1.37E+03	2.11E+05
Iodine-129 (¹²⁹ I)	I-129 (w/ separation) Liquid Scintillation	3.27E+01	2.05E+00	6.30E+01
Cesium-137 (¹³⁷ Cs)	Gamma Scan	7.33E+05	5.20E+03	3.96E+06
Uranium-233 (²³³ U)	ICP-MS	<1.60E+02	NA	1.13E+04
Uranium-235 (²³⁵ U)	ICP-MS	2.05E-01	3.43E-03	1.13E+02
Plutonium-241 (²⁴¹ Pu)	Pu238/241 Liquid Scintillation	7.85E+03	5.28E+02	8.38E+05
Total Alpha	Liquid Scintillation Counting (Cs removed)	<1.32E+04	NA	2.13E+05

**Table 3-4. Radionuclide Contaminants from Third Quarter CY18 Tank 50 Samples and SPF
WAC, Revision 17, Attachment 8.4 Targets¹**

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Target (pCi/mL)
Aluminum-26 (²⁶ Al)	Gamma Scan (Cs removed)	1.56E-01 ^a	NA	2.88E+03
Cobalt-60 (⁶⁰ Co)	Gamma Scan (Cs removed)	<2.35E-01	NA	9.75E+02
Potassium-40 (⁴⁰ K)	Gamma Scan (Cs removed)	2.59E+00 ^a	NA	1.00E+02
Nickel-59 (⁵⁹ Ni)	Ni-59/63	<1.59E+01	NA	1.13E+03
Selenium-79 (⁷⁹ Se)	Se-79	2.88E+01	1.03E+01	1.90E+04
Yttrium-90 (⁹⁰ Y)	Secular Equilibrium w/ 100% of Sr-90	4.48E+04	2.83E+03	3.15E+06
Zirconium-93 (⁹³ Zr)	ICP-MS	<4.17E+01	NA	1.00E+05
Niobium-94 (⁹⁴ Nb)	Nb-94	<4.91E-01	NA	1.53E+02
Rhodium-106 (¹⁰⁶ Rh)	Secular Equilibrium w/ 100% of Ru-106	<2.39E+00	NA	1.13E+06
Ruthenium-106 (¹⁰⁶ Ru)	Gamma Scan (Cs removed)	<2.39E+00	NA	1.13E+06
Antimony-125 (¹²⁵ Sb)	Gamma Scan (Cs removed)	7.45E+00	2.23E-01	7.99E+03
Tellurium-125m (^{125m} Te)	Secular Equilibrium w/ 100% of Sb-125	7.45E+00	2.23E-01	1.83E+03
Tin-126 (¹²⁶ Sn)	Gamma Scan (Cs removed)	4.23E+02	6.76E+00	1.80E+04
Cesium-134 (¹³⁴ Cs)	Gamma Scan	<4.59E+01	NA	1.82E+04
Cesium-135 (¹³⁵ Cs)	Cs-135	4.00E+00	1.82E-01	2.50E+02
Barium-137m (^{137m} Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137)	6.93E+05	4.92E+03	3.75E+06
Cerium-144 (¹⁴⁴ Ce)	Gamma Scan (Cs removed)	<2.12E+00	NA	1.13E+05
Promethium-147 (¹⁴⁷ Pm)	Pm-147/Sm-151 Liquid Scintillation	<3.77E+01	NA	5.63E+06
Samarium-151 (¹⁵¹ Sm)	Pm-147/Sm-151 Liquid Scintillation	<2.50E+01	NA	2.25E+04
Europium-154 (¹⁵⁴ Eu)	Gamma Scan (Cs removed)	<6.53E-01	NA	NA
Europium-155 (¹⁵⁵ Eu)	Gamma Scan (Cs removed)	<1.06E+00	NA	1.13E+04
Radium-226 (²²⁶ Ra)	Ra-226	<4.64E+00	NA	1.00E+03
Radium-228 (²²⁸ Ra)	Gamma Scan (Cs removed)	<1.86E+00	NA	1.00E+04
Actinium-227 (²²⁷ Ac)	Th-229/230	<1.96E-02	NA	1.00E+04
Thorium-229 (²²⁹ Th)	Th-229/230	<1.07E-02	NA	1.63E+05
Thorium-230 (²³⁰ Th)	Th-229/230	<1.20E-02	NA	6.26E+03
Thorium-232 (²³² Th)	ICP-MS	<1.82E-03	NA	2.88E+03
Protactinium-231 (²³¹ Pa)	Pa-231	<2.09E+00	NA	1.00E+03
Uranium-232 (²³² U)	U-232	2.17E+00 ^b	1.11E-01	9.06E+03
Uranium-234 (²³⁴ U)	ICP-MS	<1.04E+02	NA	1.13E+04
Uranium-236 (²³⁶ U)	ICP-MS	1.12E+00	1.11E-02	1.13E+04
Uranium-238 (²³⁸ U)	ICP-MS	3.52E+00	1.58E-02	1.13E+04

Table 3-4. Radionuclide Contaminants from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Attachment 8.4 Targets¹, 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 (²³⁷ Np)	ICP-MS	<1.17E+01	NA	1.00E+04
Plutonium-238 (²³⁸ Pu)	Pu238/241 Pu alpha PHA	2.34E+04	1.57E+03	2.13E+05
Plutonium-239 (²³⁹ Pu)	Pu238/241 Pu alpha PHA	5.77E+02	7.02E+01	2.13E+05
Plutonium-240 (²⁴⁰ Pu)	Pu238/241 Pu alpha PHA	5.77E+02	7.02E+01	2.13E+05
Plutonium-242 (²⁴² Pu)	ICP-MS	<6.33E+01	NA	2.13E+05
Plutonium-244 (²⁴⁴ Pu)	ICP-MS	<2.94E-01	NA	7.02E+04
Americium-241 (²⁴¹ Am)	Am/Cm	5.19E+00	9.18E-01	2.13E+05
Americium-242m (^{242m} Am)	Am/Cm	<3.34E-02	NA	4.50E+05
Americium-243 (²⁴³ Am)	Am/Cm	<2.57E-01	NA	2.13E+05
Curium-242 (²⁴² Cm)	Am/Cm	<2.76E-02	NA	1.13E+04
Curium-244 (²⁴⁴ Cm)	Am/Cm	1.73E+00	3.75E-01	2.13E+05
Curium-245 (²⁴⁵ Cm)	Am/Cm	<1.05E+00	NA	2.25E+05

- a. Only one detectable value from the analyzed triplicate set.
b. 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.¹

Table 3-5. Chemical Contaminants Impacting Saltstone Disposal Unit (SDU) Flammability from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Table 2 Limits and Targets¹

<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 ^{a,b}	NA	1.10E+01 ppm (Limit)
Tetraphenylborate [TPB] (B(C ₆ H ₅) ₄) ⁻	HPLC	<5.00E+00	NA	5.00E+00 mg/L (Limit)
Ammonium (NH ₄ ⁺)	IC	<1.00E+02	NA	2.12E+02 mg/L (Limit)
Total Mercury (Hg)	ICP-MS	6.74E+01	3.98E-01	3.25E+02 mg/L (Limit)
Monomethyl Mercury (CH ₃ Hg)	CVAFS w/ Distillation	3.30E+01 ^c	1.02E+00	3.50E+02 mg/L (Limit)
Dimethyl Mercury ((CH ₃) ₂ Hg)	CVAFS	2.94E-02 ^c	2.56E-03	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 8.
c. Mercury species calculated from data presented in Reference 13.

Table 3-6. Other Organics Impacting SDU Flammability from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Table 3 Concentrations¹

<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 ₄ H ₉ OH)	VOA	<5.00E-01	NA	0.75
Tributylphosphate[TBP] ((C ₄ H ₉ O) ₃ PO)	SVOA	<7.50E-01	NA	1.0
Isopropanol (C ₃ H ₇ OH)	VOA	<2.50E-01	NA	0.25
Methanol (CH ₃ OH)	a	NA	NA	0.05
NORPAR 13 (C _n H _{2.n})	SVOA	<7.50E-01	NA	0.75

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

Table 3-7. Processing Constituents from Third Quarter CY18 Tank 50 Samples and SPF WAC, Revision 17, Table 4 Limits¹

<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	5.26 M	6.80E-02	2.5 M < [Na ⁺] < 7.0 M
Total Insoluble Solids	Calculated	~0 wt%	NA	< 15 wt%

Table 3-8 contains additional measured constituents per the TTQAP.³ This table also includes formate analysis and the Total Organic Carbon (TOC) (minus formate & oxalate) as shown in Appendix 1 of the WAC. The average and standard deviation that may be used to determine the 95% confidence interval for the Third Quarter CY18 are shown for TOC (minus formate & oxalate). These standard deviations include the variance in each triplicate analysis set as well as the one-sigma instrument uncertainty of 10% reported for each value. These values were calculated using the Guide for the Expression of Uncertainty in Measurement (GUM) Workbench statistical package.¹⁶ These data indicate that, at the 95% confidence interval, the calculated TOC (minus formate & oxalate) could be in the approximate range of 0 to 95 mg/L for the Third Quarter CY18 sample, i.e., the average \pm 2X Std. Dev.

Table 3-8. Additional Measured Constituents³

<u>Constituent</u>	<u>Method</u>	<u>Average Value</u>	<u>Std. Dev.</u>
Density (slurry)	Measured (24.4°C)	1.2354 g/mL	0.0001
Specific Gravity	a	1.2392	0.0001
Total Solids	Measured	27.26 wt%	0.03
Total Beta	LSC	1.98E+05 pCi/mL	3.20E+03
Total Gamma	b	6.94E+05 pCi/mL	1.51E+03 ^c
Beryllium (Be)	ICP-ES	<1.94E-02	NA
N-Nitrosodimethylamine (C₂H₆N₂O)	SVOA	<1 mg/L	NA
N-Dioctylnitrosamine (C₁₆H₃₄N₂O)	SVOA	<1 mg/L	NA
Formate (HCO₂)^d	IC	2.04E+02	1.00E+00
Total Organic Carbon (minus formate & oxalate)^e	Calculated	3.77E+01 mg/L	2.84E+01 ^f

a. Calculated from the measured density of slurry and density of water at 25.4 °C.¹⁷

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.

d. Formate is not required by the WAC but is used in the Total Organic Carbon (minus formate & oxalate) calculation.¹

e. Total Organic Carbon (minus formate & oxalate) as shown in Appendix 1 of the WAC.¹

f. Standard deviation includes uncertainty in the triplicate analysis and the one-sigma instrument uncertainty.

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 compared with the current SPF WAC value of 11 ppm that has been in effect since revision 12 of the WAC dating back to July of 2013.
- Nitrosamines were not detected in the Tank 50 salt solution sample above the instrument detection limits of < 1 mg/L.
- The minimum detection limit is reported for ⁹⁴Nb 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.⁶ However, the minimum detection limit reported for the Third Quarter CY18 Tank 50 sample for ⁹⁴Nb is within 12% of the estimated detection limit initially established by SRNL in 2009.⁷ Thus per guidance from SRR,⁶ SRNL continues to achieve as low as practical detection limits for this radionuclide.

5.0 Reference

- ¹ Ray, J. W., "Waste Acceptance Criteria for Aqueous Waste Sent to the Z-Area Saltstone Production Facility", Savannah River Remediation, X-SD-Z-00001, Rev. 17, March 2017.
- ² Ray, J. W., "Routine Saltstone Support for Salt Solution and Grout Analyses – FY2018", Savannah River Remediation, X-TTR-Z-00012, Rev. 1, June 2018.
- ³ Hill, K. A., "Task Technical and Quality Assurance Plan for Salt Solution and Grout Sample Preparation and Analyses – FY2018", Savannah River National Laboratory, SRNL-RP-2017-00658, Rev. 0, December 2017.
- ⁴ 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.
- ⁵ 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.
- ⁶ Dixon, D. B., "Minimum Detection Limits for Saltstone Quarterly WAC Analyses", Savannah River Remediation, SRR-WSE-2013-00005, Rev. 1, January 2013.
- ⁷ 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.
- ⁸ Crawford, C. L., "Results for the Third Quarter Calendar Year 2018 Tank 50 Salt Solution Sample: Cs-137", Savannah River National Laboratory, SRNL-L3100-2018-00075, Rev.0, November 2018.
- ⁹ Crawford, C. L., "3Q CY18 Tank 50 WAC Characterization", B9108-00026-48, SRNL E-Notebook (Production), Savannah River National Laboratory, September 2018.
- ¹⁰ "Aqua Regia Dissolution of Sludge for Elemental Analysis"; Savannah River National Laboratory, Manual L16.1, Procedure ADS-2226, Rev. 10, July 2013.
- ¹¹ "Weight Percent Solids Determination Using a Furnace or Oven", Savannah River National Laboratory, Manual L29, Procedure ITS-0078, Rev. 1, October 2012.
- ¹² "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 September 14, 2018).
- ¹³ Bannochie, C. J., "Results of Hg Speciation Testing on the 3Q18 Tank 50 Sample", Savannah River National Laboratory, SRNL-L3300-2018-00069, Rev. 0, December 2018.
- ¹⁴ "Technical Reviews", Savannah River Site, Manual E7, Procedure 2.60, Latest Revision.
- ¹⁵ "Savannah River National Laboratory Technical Report Design Check Guidelines", Westinghouse Savannah River Company, WSRC-IM-2002-00011, Rev. 2, August 2004.
- ¹⁶ Metrodata GmbH, "GUM workbench: User Manual for Version 1.2, 2.3, and 2.4," Weil am Rhein, Germany, 2009.
- ¹⁷ *CRC Handbook of Chemistry and Physics*, 99th ed.; 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 September 14, 2018).

Distribution:

Name:	
P. M. Almond	C. A. Langton
J. P. Arnold	K. R. Liner
T. R. Ball	M. J. Mahoney
C. J. Bannochie	K. B. Martin
M. J. Barnes	M. W. McCoy
M. N. Borders	P. W. Norris
J. M. Bricker	J. E. Occhipinti
K. M. Brotherton	F. M. Pennebaker
L. W. Brown	R. C. Player
T. B. Brown	M. M. Potvin
N. F. Chapman	J. W. Ray
J. H. Christian	C. Ridgeway
W. A. Condon	L. B. Romanowski
A. D. Cozzi	K. H. Rosenberger
C. L. Crawford	A. Samadi-Dezfouli
C. C. DiPrete	D. C. Sherburne
K. D. Dixon	F. M. Smith
R. E. Edwards	A. V. Staub
S. D. Fink	J. Stevens
E. J. Freed	C. B. Sudduth
E. W. Harrison	B. J. Wiedenman
C. C. Herman	T. L. White
K. A. Hill	A. W. Wiggins
P. J. Hill	W. R. Wilmarth
J. F. Iaukea	R. H. Young
V. Jain	Records Administration (EDWS)
V. M. Kmiec	