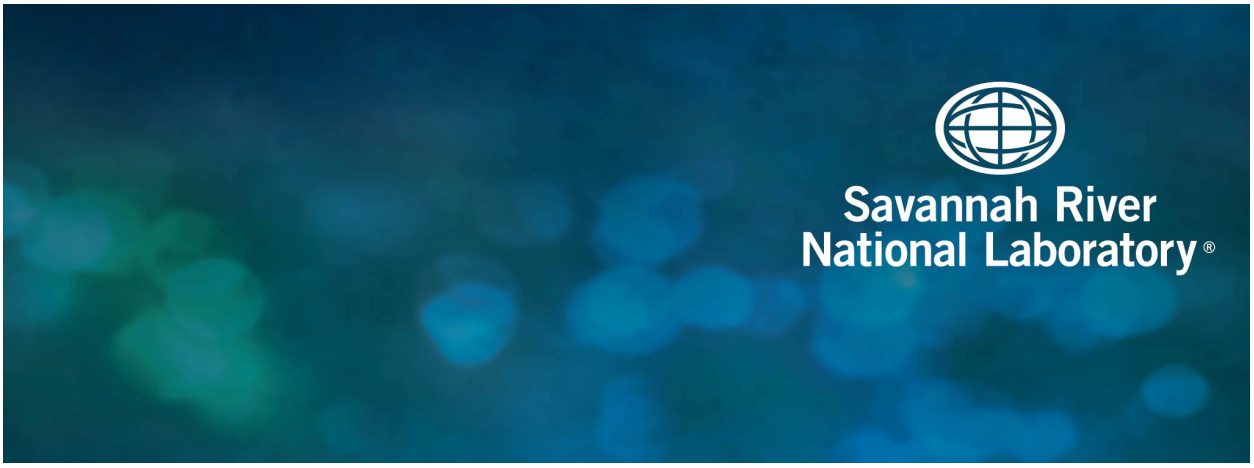
Contract No:

This document was prepared in conjunction with work accomplished under Contract No. 89303321CEM000080 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Disclaimer:



A U.S. DEPARTMENT OF ENERGY NATIONAL LAB • SAVANNAH RIVER SITE • AIKEN, SC • USA

Results for the Second Quarter Calendar Year 2021 Tank 50 Salt Solution Sample

C. L. Crawford

December 2021

SRNL-STI-2021-00338, Revision 1

DISCLAIMER

Printed in the United States of America

Prepared for
U.S. Department of Energy

SRNL-STI-2021-00338
Revision 1

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

Retention: *Permanent*

Results for the Second Quarter Calendar Year 2021 Tank 50 Salt Solution Sample

C. L. Crawford

December 2021

Savannah River National Laboratory is operated by Battelle Savannah River Alliance for the U.S. Department of Energy under Contract No. 89303321CEM000080.

REVIEWS AND APPROVALS

AUTHOR:

C. L. Crawford, Author, Applied Materials Research

TECHNICAL REVIEW:

J. H. Christian, Separation Sciences & Engineering, Reviewed per E7 2.60

APPROVAL:

A. D. Cozzi, Manager
Applied Materials Research

F. M. Pennebaker, Director
Chemical Processing

T. H. Huff, Manager
DWPF and Saltstone Facility Engineering

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

EXECUTIVE SUMMARY

In this Technical Report, the chemical and radionuclide contaminant results from the Second Quarter Calendar Year 2021 (CY21) 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 that were established at the time the Tank 50 sample was obtained.[1] The chemical and radionuclide contaminant results from the characterization of the Second Quarter CY21 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 per Task 2 from the SRR request, will be obtained semi-annually for the 1QCY21 and 3QCY21 Tank 50 samples.

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.[1]

- Nitrosamines were not detected in the Tank 50 salt solution sample above the instrument detection limits of <1 mg/L.

- The minimum detection limit (<2.68E-01 pCi/mL) is reported for ^{94}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.[4] However, the minimum detection limit reported for the Second Quarter CY21 Tank 50 sample for ^{94}Nb is lower than the estimated detection limit of 4.38E-01 pCi/mL initially established by SRNL in 2009.[5] Thus, per guidance from SRR,[4] SRNL continues to achieve as low as practical detection limits for this radionuclide.

LIST OF REVISIONS		
Revision Number	**Summary of Changes**	**Date**
0	Initial Issue	September 2021
1	Mercury data corrected in Table 3-1 and Table 3-5. Detailed information pertaining to corrected mercury data given in text of page 3. Footnote to Table 3-4 in original Revision 0 was in error and has been removed. The value for Isopar L in the text of page 8 has been revised from original <2.70 ppm to the correct <2.69 ppm value as shown in Table 3-5.	December 2021

TABLE OF CONTENTS

LIST OF TABLES

LIST OF ABBREVIATIONS

CVAFS	Cold Vapor Atomic Fluorescence Spectroscopy
DMA	Direct Mercury Analyzer
EDTA	ethylenediaminetetraacetate
GC/MS	Gas Chromatographic/Mass Spectrometric
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
LSC	Liquid Scintillation Counting
MRL	Minimum Reporting Limit
P&T-TD-CVAFS	Purge & Trap, Thermal Desorption, Cold Vapor Atomic Fluorescence Spectroscopy
Pu alpha PHA	Plutonium alpha Pulse Height Analysis
RCRA	Resource Conservation and Recovery Act
SaM	Sensing & Metrology
SDI	Salt Disposition Integration
SDU	Saltstone Disposal Unit
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).[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 and comparison to WAC Limits has been previously issued.[6]

2.0 Experimental

2.1 Technical

The Second Quarter CY21 Tank 50 samples [a 200-mL sample obtained 6" below the surface (HTF-50-21-35) and a 1-L variable depth sample (VDS) obtained 66" from the tank bottom (HTF-50-21-36)] were obtained and received at Savannah River National Laboratory (SRNL) on April 28, 2021.[7]

The contents of the 1-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, a 30-mL aliquot and a 15-mL aliquot of the Tank 50 sample were pumped into a Teflon® and a glass container, respectively, with zero headspace. These two samples were used for Hg speciation testing. The remaining contents were then transferred by pumping into two different high-density polyethylene (HDPE) 1-L bottles. The transferred slurry was left to settle in the bottles and no suspended or settled solids were observed during the brief storage in the Shielded Cells. 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 promptly transferred out of the Shielded Cells on the same day as it was collected from the steel variable depth sampler and placed in a radiochemical hood. The two small zero headspace vials for Hg speciation testing 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 for volatile organic analysis (VOA) and semi-volatile organic analysis (SVOA) were removed from the 200-mL surface sample from within a radiochemical hood and were transferred using glass pipettes into clean amber 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 Second Quarter CY21 Tank 50 sample. The 1-sigma 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 VOA and 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 Sensing & Metrology (SaM) Procedure L16.1, ADS-2656.[8] 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 SaM Procedure L16.1, ADS-2657.[9] Both of these methods use discrete standards as detailed in the procedures.[8,9] The SVOA method uses organic solvents to extract SVOA analytes that are subsequently measured by GC/MS. A 3 mL dichloromethane (also known as methylene chloride, CH_2Cl_2) volume is used to initially extract 10 mL of Tank 50 supernate for phenol. The Tank 50 supernate is then extracted with 2 additional 3-mL volumes of dichloromethane. The dichloromethane extracts are combined and concentrated to 1 mL before analysis. Tributyl phosphate is analyzed from a 0.01 mL hexane (C_6H_{14}) extract of 10 mL of Tank 50 supernate. Isopar L[a] and Norpar 13 are analyzed from a 2.5 mL hexane extraction of 10 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 (1 mL supernate diluted to 50 mL total volume) by the aqua regia method.[10] The aqua regia method heats the Tank 50 supernate mixed with a 1:3 mixture of nitric acid/hydrochloric acid for 2 hours in sealed Teflon containers in an oven at 115 ºC. 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). The IC, TIC/TOC and HPLC methods all used undiluted samples. All the above analyses excluding VOA and SVOA used approximately 150 mL of the 1-L variable depth sample. Densities were measured on triplicate samples of the Tank 50 slurry by SRNL SaM. 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.[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] The Cs-137 and C-134 radionuclides are determined from gamma spectroscopy. Total beta is measured from a radscreen method using Liquid Scintillation Counting (LSC). Plutonium isotopes (Pu-238, Pu-239 and Pu-240) are determined from a Plutonium alpha Pulse Heigh Analysis (Pu alpha PHA) method. The total alpha is measured from the same method after removal of Cs-137 from the sample using ammonium phosphomolybdate. This methodology for measuring total alpha concentrations results in upper limits rather than minimum detectable activities when the total alpha concentration is not high enough for a detectable concentration measurement.

Mercury analyses performed at SRNL by SaM included Total mercury using the Direct Mercury Analyzer (DMA) method[13] and monomethyl mercury and ethyl mercury by Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS).[14] Soluble elemental mercury (Hg(0)) was measured by SaM using a Purge & Trap, Thermal Desorption, CVAFS (P&T-TD-CVAFS) method.[15] A variation of this method was also developed by SaM to analyze for inorganic mercury (Hg(I/II)).[16] Dimethyl mercury was analyzed using a semi-quantitative SaM VOA method that involves extraction followed by GC/MS.[8] Total mercury and other mercury (Hg) species will be calculated from separate aliquots analyzed by CVAFS by the Eurofins Frontier Global Sciences, Inc. laboratory in Seattle, WA, and reported in a separate memorandum at a later time per the TTQAP.[3] The parent sample for all mercury analyses performed at SRNL was obtained from the original Tank 50 sample within two days of sample receipt. As discussed above, the parent Tank 50 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.[17] Total mercury, Monomethyl and ethyl mercury

[a] 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.

are determined from the Tank 50 parent sample obtained in the 30-mL Teflon bottle. All other species are determined from the 15-mL Tank 50 parent stored in the glass bottle. Samples of Tank 50 submitted to SRNL SaM for mercury speciation analysis were submitted without dilution. These samples were diluted within the AD laboratories to meet the targeted calibration range of either the DMA instrument for total Hg or the CVAFS instrument for other Hg species and for VOA analysis for dimethyl mercury.

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.[18] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[19] The customer requested that a Functional Classification of Safety Significant apply to this work.[2] Data collection and analysis methods used in this work comply with this requirement as detailed in the TTQAP.[3]

3.0 Results and Discussion

Analyzed nonradionuclide chemical concentrations, their 1-sigma standard deviations and their corresponding WAC Limits[1] 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 Targets[1] 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.[1] 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.

Comparison of the average analyzed detectable values shown in Table 3-1 to the WAC Limits indicates that free hydroxide and nitrate anions and total mercury are the highest analytes relative to the WAC Limits at 20%, 28% and 20%, respectively. The corrected mercury data shown in Table 3-1 derive from two different sources. The original values given in Revision 0 of this report for both Total mercury and Monomethyl mercury (shown in both Table 3-1 and Table 3-5) were in error due to inadvertent use of more recent 3Q21 Tank 50 WAC data that was erroneously used in the 2Q21 Revision 0 report. Revision of these values results in only minor changes for these two analytes, as the original erroneous reported data for Total mercury and Monomethyl mercury of 62.2 mg/L and 26.4 mg/L, respectively, in the Revision 0 document changed to the correct values reported here in Revision 1 of Total mercury and Monomethyl mercury of 63.8 mg/L and 22.2 mg/L, respectively. Elemental mercury - measured as the single value shown in Table 3-1 - necessitated reanalysis due to initial complications with P&T-TD-CVAFS instrumentation. The revised value for Elemental mercury shown in Table 3-1 of 4.78 mg/L is significantly lower than originally reported at 26.5 mg/L. 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 74%, with average TOC at a lower ratio to WAC Target of 29%. 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[1] Limits and Targets are shown in Table 3-3 and Table 3-4, respectively. The minimum detection limit reported for Nb-94 of (<2.68E-01 pCi/mL) in Table 3-4 is above the requested SRR target minimum detection limit of 2.8E-03 pCi/mL "to meet future inventory reporting requirements"[4] but is lower than the estimated detection limit initially

established by SRNL of 4.38E-01 pCi/mL in 2009.[5] All of these Nb-94 values (analyzed, 2013-requested and 2009 estimated detection limit) are orders of magnitude below the WAC target for Nb-94 of 1.52E+02 pCi/mL shown in Table 3-4.

Comparison of the average analyzed detectable values shown in Table 3-3 to the WAC Limits[1] indicates that Tc-99 and I-129 are the highest analytes relative to the WAC Limits at 15% and 23%, respectively. Comparison of the average analyzed detectable values shown in Table 3-4 to the WAC Targets indicates that none of the radionuclides are higher than 10% of the WAC Targets.[1] Table 3-4 indicates that the upper limit determined in the triplicate samples analyzed for total alpha for the 2Q21 sample is 3.87E+03 pCi/mL.

Table 3-1. Chemical Contaminants from Second Quarter CY21 Tank 50 Samples and SPF WAC, Attachment 8.1 Limits

Chemical Name (Formula)	Method	Average Concentration (mg/L)	Std. Dev.	WAC Limit (mg/L)
Aluminate ($Al(OH)_4^-$)	ICP-ES	1.81E+04[a]	1.32E+02	**4.08E+05**
Ammonium (NH_4^+)	IC	<5.00E+00	NA	**2.12E+02**
Carbonate (CO_3^{2-})	TIC	1.78E+04 [b]	2.88E+02	**1.20E+05**
Chloride (Cl^-)	IC	4.47E+02	4.93E+00	**7.95E+03**
Fluoride (F^-)	IC	<1.00E+02	NA	**4.07E+03**
Free Hydroxide (OH^-)	Total Base	3.23E+04 [b]	3.40E+02	**1.58E+05**
Nitrate (NO_3^-)	IC	1.22E+05	4.21E+04	**4.37E+05**
Nitrite (NO_2^-)	IC	2.67E+04	3.79E+02	**2.14E+05**
Oxalate ($C_2O_4^{2-}$)	IC	4.52E+02	5.86E+00	**2.72E+04**
Phosphate (PO_4^{3-})	IC	3.15E+02	4.62E+00	**3.14E+04**
Sulfate (SO_4^{2-})	IC	5.13E+03	2.42E+02	**5.69E+04**
Arsenic (As)	ICP-MS	<1.10E-01	NA	**1.97E+02**
Barium (Ba)	ICP-ES	6.51E+00 [c]	4.09E+00	**6.19E+02**
Cadmium (Cd)	ICP-ES	<7.68E-01	NA	**3.10E+02**
Chromium (Cr)	ICP-ES	4.36E+01	6.17E-01	**1.50E+03**
Lead (Pb)	ICP-MS	3.43E-01	9.94E-02	**7.50E+02**
Total Mercury (Hg)	DMA	6.38E+01	5.29E-01	**3.25E+02**
Elemental Mercury (Hg(0))	P&T-TD-CVAFS	4.78E+00[d]	NA[d]	**3.25E+02**
Monomethyl Mercury (CH_3Hg)	CVAFS	2.22E+01	7.00E-01	**3.50E+02**
Ethyl Mercury (C_2H_5Hg)	CVAFS	<1.00E+00	NA	**3.73E+02**
Selenium (Se)	ICP-MS	<2.74E-01	NA	**3.75E+02**
Silver (Ag)	ICP-ES	<3.28E-01	NA	**6.19E+02**
Aluminum (Al)	ICP-ES	5.15E+03	3.74E+01	**1.16E+05**
Potassium (K)	ICP-ES	4.19E+02	1.63E+01	**3.03E+04**
Butanol (C_4H_9OH)	VOA	<5.00E-01[e]	NA	**7.73E+00**
Propanol (C_3H_7OH)	VOA	<2.50E-01[e]	NA	**1.88E+00**
Phenol (C_6H_5OH)	SVOA	<1.00E+01[e]	NA	**7.50E+02**
Tetraphenylborate [TPB] ($B(C_6H_5)_4^-$)	HPLC	<5.00E+00	NA	**5.00E+00**
Total Organic Carbon (----)	TOC	2.20E+02[b]	1.53E+00	**4.50E+03**
Isopar L (----)	SVOA	<3.30E+01[e]	NA	**8.75E+01**

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; only two detectable values.
d. Measurement performed on single sample rather than triplicate samples.
e. Measurement performed on duplicate samples rather than triplicate samples.

Table 3-2. Chemical Contaminants from Second Quarter CY21 Tank 50 Samples and SPF WAC, Attachment 8.2 Targets

Chemical Name (Formula)	Method	Average Concentration (mg/L)	Std. Dev.	WAC Target (mg/L)
Aluminum (Al)	ICP-ES	5.15E+03	3.74E+01	**7.00E+03**[e]
Boron (B)	ICP-ES	4.32E+01	8.03E-01	**7.43E+02**
Cobalt (Co)	ICP-MS[a]	1.22E-01[a]	4.36E-02	**1.45E+02**
Copper (Cu)	ICP-ES	<4.94E+00	NA	**7.43E+02**
Iron (Fe)	ICP-ES	1.15E+01	6.68E+00	**4.95E+03**
Lithium (Li)	ICP-ES	< 2.09E+00	NA	**7.43E+02**
Manganese (Mn)	ICP-ES	<7.66E-01	NA	**7.43E+02**
Molybdenum (Mo)	ICP-ES	1.56E+01	6.04E-01	**7.43E+02**
Nickel (Ni)	ICP-ES	<1.07E+00	NA	**7.43E+02**
Silicon (Si)	ICP-ES	3.16E+01	6.69E+00	**1.07E+04**
Strontium (Sr)	ICP-ES	<1.56E-01	NA	**7.43E+02**
Zinc (Zn)	ICP-ES	4.84E+00	3.75E-01	**8.03E+02**
Dimethyl Mercury ($(CH_3)_2Hg$)	VOA[b]	<1.00E-01	NA	**1.00E+00**
Benzene (C_6H_6)	VOA	<1.50E-01[c]	NA	**3.10E+02**
Methanol (CH_3OH)	VOA	d	NA	**1.88E+00**
Toluene ($C_6H_5CH_3$)	VOA	<1.50E-01[c]	NA	**3.10E+02**
Dibutylphosphate [DBP] ($C_8H_{19}O_4P$)	IC	<2.50E+02	NA	**3.47E+02**
Tributylphosphate [TBP] ($(C_4H_9O)_3PO$)	SVOA	<7.50E-01[c]	NA	**7.50E+00**
Total Organic Carbon (TOC)	TOC	2.20E+02	1.53E+00	**7.50E+02**[e]
EDTA ($C_{10}H_{12}N_2O_8^{4-}$)	HPLC	<1.00E+02	NA	**3.10E+02**
NORPAR 13 ($C_nH_{2\cdot n}$)	SVOA	<7.50E-01[c]	NA	**7.50E-01**
Formate ($CHOO^-$)	IC	<1.00E+02	NA	**6.38E+03**

a. Cobalt based on the stable Co-59 isotope.
b. This method is a semi-quantitative method due to lack of a dimethyl mercury standard.
c. Measurement performed on duplicate samples rather than triplicate samples.
d. Currently, a routine method for detecting this species does not exist in SaM.
e. 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.[1]

Table 3-3. Radionuclide Contaminants from Second Quarter CY21 Tank 50 Samples and SPF WAC, Attachment 8.3 Limits

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Limit (pCi/mL)
Tritium (^{3}H)	Tritium Counting	1.02E+03	5.71E+01	**5.63E+05**
Carbon-14 (^{14}C)	C-14 Liquid Scintillation	3.90E+02	3.55E+01	**1.13E+05**
Nickel-63 (^{63}Ni)	Ni-59/63	<6.04E+00	NA	**1.13E+05**
Strontium-90 (^{90}Sr)	Sr-90 Liquid Scintillation	1.07E+05	1.08E+04	**2.62E+06**
Technetium-99 (^{99}Tc)	Tc-99 Liquid Scintillation	3.25E+04	1.08E+03	**2.11E+05**
Iodine-129 (^{129}I)	I-129 (w/ separation) Liquid Scintillation	2.30E+01	1.04E+00	**1.00E+02**
Cesium-137 (^{137}Cs)	Gamma Scan	1.20E+05	6.59E+03	**1.29E+06**
Uranium-233 (^{233}U)	ICP-MS	<2.65E+02	NA	**1.13E+04**
Uranium-235 (^{235}U)	ICP-MS	2.60E-01	2.93E-03	**1.13E+02**
Plutonium-241 (^{241}Pu)	Pu238/241 Liquid Scintillation	1.27E+03	1.56E+02	**8.38E+05**
Total Alpha	Liquid Scintillation Counting (Cs removed)	<3.87E+03	NA	**2.13E+05**

Table 3-4. Radionuclide Contaminants from Second Quarter CY21 Tank 50 Samples and SPF WAC, Attachment 8.4 Targets

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Target (pCi/mL)
Aluminum-26 (^{26}Al)	Gamma Scan (Cs removed)	<4.73E-02	N/A	**2.88E+03**
Potassium-40 (^{40}K)	Gamma Scan (Cs removed)	<2.06E+00	NA	**1.00E+02**
Cobalt-60 (^{60}Co)	Gamma Scan (Cs removed)	<8.69E-02	NA	**9.75E+02**
Nickel-59 (^{59}Ni)	Ni-59/63	<2.65E+00	NA	**1.13E+03**
Selenium-79 (^{79}Se)	Se-79	<1.43E+01	NA	**1.90E+04**
Yttrium-90 (^{90}Y)	Secular Equilibrium w/ 100% of Sr-90	1.07E+05	1.08E+04	**2.62E+06**
Zirconium-93 (^{93}Zr)	ICP-MS	9.01E+01	5.48E+00	**1.00E+05**
Niobium-94 (^{94}Nb)	Nb-94	<2.68E-01	NA	**1.53E+02**
Rhodium-106 (^{106}Rh)	Secular Equilibrium w/ 100% of Ru-106	<1.95E+00	NA	**3.12E+05**
Ruthenium-106 (^{106}Ru)	Gamma Scan (Cs removed)	<1.95E+00	NA	**3.12E+05**
Antimony-125 (^{125}Sb)	Gamma Scan (Cs removed)	7.48E+01	4.50E-01	**7.99E+03**
Tellurium-125m (^{125m}Te)	Secular Equilibrium w/ 100% of Sb-125	7.48E+01	4.50E-01	**1.83E+03**
Tin-126 (^{126}Sn)	Gamma Scan (Cs removed)	2.98E+02	1.57E+01	**1.80E+04**
Cesium-134 (^{134}Cs)	Gamma Scan	<8.69E+01	NA	**5.93E+03**
Cesium-135 (^{135}Cs)	Cs-135	9.68E-01	2.05E-01	**2.50E+02**
Barium-137m (^{137m}Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137)	1.16E+05	4.07E+03	**1.22E+06**
Cerium-144 (^{144}Ce)	Gamma Scan (Cs removed)	<4.27E+00	NA	**3.12E+04**
Praseodymium-144 (^{144}Pr)	Secular Equilibrium w/ 100% of Ce-144	<4.27E+00	NA	**3.12E+04**
Promethium-147 (^{147}Pm)	Pm-147/Sm-151 Liquid Scintillation	<1.45E+01	NA	**1.57E+06**
Samarium-151 (^{151}Sm)	Pm-147/Sm-151 Liquid Scintillation	<4.82E+01	NA	**2.25E+04**
Europium-154 (^{154}Eu)	Gamma Scan (Cs removed)	<3.06E-01	NA	**1.62E+03**
Radium-226 (^{226}Ra)	Ra-226	<3.74E+00	NA	**1.00E+03**
Radium-228 (^{228}Ra)	Gamma Scan (Cs removed)	<8.29E-01	NA	**1.00E+04**
Actinium-227 (^{227}Ac)	Th-229/230	<1.47E-01	NA	**1.00E+04**
Thorium-229 (^{229}Th)	Th-229/230	<7.12E-02	NA	**1.63E+05**
Thorium-230 (^{230}Th)	Th-229/230	<8.02E-02	NA	**6.26E+03**
Thorium-232 (^{232}Th)	ICP-MS	<3.00E-03	NA	**2.88E+03**
Protactinium-231 (^{231}Pa)	Pa-231	<2.61E+00	NA	**1.00E+03**
Uranium-232 (^{232}U)	U-232	2.14E+00	6.68E-01	**2.27E+03**
Uranium-233 (^{233}U)	ICP-MS	<2.65E+02	NA	**3.12E+03**
Uranium-234 (^{234}U)	ICP-MS	<1.71E+02	NA	**3.12E+03**
Uranium-236 (^{236}U)	ICP-MS	<1.77E+00	NA	**3.12E+03**
Uranium-238 (^{238}U)	ICP-MS	2.32E+00	4.89E-02	**3.12E+03**

Table 3-4. Radionuclide Contaminants from Second Quarter CY21 Tank 50 Samples and SPF WAC, Attachment 8.4 Targets, continued

Radionuclide	Method	Average Concentration (pCi/mL)	Std. Dev.	WAC Target (pCi/mL)
Neptunium-237 (^{237}Np)	ICP-MS	<1.93E+01	NA	**1.00E+04**
Plutonium-238 (^{238}Pu)	Pu238/241 Pu alpha PHA	3.42E+03	4.17E+02	**6.67E+04**
Plutonium-239 (^{239}Pu)	Pu238/241 Pu alpha PHA	1.31E+02	2.00E+01	**6.67E+04**
Plutonium-240 (^{240}Pu)	Pu238/241 Pu alpha PHA	1.31E+02	2.00E+01	**6.67E+04**
Plutonium-242 (^{242}Pu)	ICP-MS	<1.05E+02	NA	**6.67E+04**
Plutonium-244 (^{244}Pu)	ICP-MS	<4.86E-01	NA	**7.02E+04**
Americium-241 (^{241}Am)	Am/Cm	1.77E+00	2.30E-01	**6.67E+04**
Americium-242m (^{242m}Am)	Am/Cm	<1.17E-01	NA	**4.50E+05**
Americium-243 (^{243}Am)	Am/Cm	<8.29E-01	NA	**6.67E+04**
Curium-242 (^{242}Cm)	Am/Cm	<9.64E-02	NA	**1.13E+04**
Curium-244 (^{244}Cm)	Am/Cm	1.32E+00	2.55E-01	**6.67E+04**
Curium-245 (^{245}Cm)	Am/Cm	<3.46E+00	NA	**2.25E+05**
Total Alpha	Liquid Scintillation Counting (Cs removed)	<3.87E+03	NA	**6.67E+04**

The following tables show various chemical contaminants (Table 3-5), organic species (Table 3-6) and processing constituents (Table 3-7) related to the Salt Disposal Unit (SDU) that are referred to in the WAC per Tables 2, 4 and 5, respectively.[1] The reported detection limit for Isopar L of <2.69E+01 ppm in Table 3-5 is lower than the current Isopar limit of 87.5 ppm associated with SDU flammability for the Salt Disposition Integration (SDI) WAC.[1] The pH value shown in Table 3-7 is calculated from the pH equation for water (pH + pOH = 14) with the measured [OH^-] from Table 3-1 used in the calculation.

Table 3-5. Chemical Contaminants Impacting Saltstone Disposal Unit (SDU) Flammability from Second Quarter CY21 Tank 50 Samples and WAC Table 2 Limits and Targets

Chemical Name (Formula)	Method	Average Concentration (mg/L)	Std. Dev.	WAC Limit/Target
Isopar L (----)	SVOA	<2.69E+01 ppm[a,b]	NA	**8.75E+01 ppm (Limit)**
Tetraphenylborate [TPB] ($B(C_6H_5)_4^-$)	HPLC	<5.00E+00	NA	**5.00E+00 mg/L (Limit)**
Ammonium (NH_4^+)	IC	<5.00E+00	NA	**2.12E+02 mg/L (Limit)**
Total Mercury (Hg)	DMA	6.38E+01	5.29E-01	**3.25E+02 mg/L (Limit)**
Monomethyl Mercury (CH_3Hg)	CVAFS	2.22E+01	7.00E-01	**3.50E+02 mg/L (Limit)**
Dimethyl Mercury ($(CH_3)_2Hg$)	VOA[c]	<1.00E-01	NA	**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. This method is a semi-quantitative method due to lack of a dimethyl mercury standard.

Table 3-6. Other Organics Impacting SDU Flammability from Second Quarter CY21 Tank 50 Samples and WAC Table 4 Concentrations

Chemical Name (Formula)	Method	Average Concentration (mg/L)	Std. Dev.	WAC Concentrations (mg/L)
Butanol (C_4H_9OH)[a]	VOA	<5.00E-01	NA	**0.75**
Tributylphosphate[TBP] ($(C_4H_9O)_3PO$)[a]	SVOA	<7.50E-01	NA	**1.0**
Propanol (C_3H_7OH)[a]	VOA	<2.50E-01	NA	**0.25**
Methanol (CH_3OH)	b	NA	NA	**0.05**
NORPAR 13 ($C_nH_{2.n}$)[a]	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 Second Quarter CY21 Tank 50 Samples and WAC Table 5 Limits

Processing Constituents	Method	Value	Std. Dev.	WAC Limit
pH	Calculated	>13	NA	**> 10**
Sodium Concentration	ICP-ES	4.74 M	2.46E-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] There were no detectable nitrosamine species in the Tank 50 surface sample via the SVOA analyses shown in Table 3-8.

Table 3-8. Additional Measured Constituents

Constituent	Method	Average Value	Std. Dev.
Density (slurry)	Measured (25.4ºC)	1.225 g/mL	<0.001 g/mL
Specific Gravity	a	1.229	<0.001
Total Solids	Measured	26.58 wt %	0.18 wt %
Total Beta	LSC	3.91E+05 pCi/mL	5.12E+03 pCi/mL
Total Gamma	b	1.16E+05 pCi/mL	3.12E+02 pCi/mL[c]
Beryllium (Be)	ICP-ES	<1.56E-01 mg/L	NA
N-Nitrosodimethylamine ($C_2H_6N_2O$)	SVOA[d]	<1 mg/L	NA
N-Dioctylnitrosamine ($C_{16}H_{34}N_2O$)	SVOA[d]	<1 mg/L	NA

a. Calculated from the measured density of slurry and density of water at 25.4 °C.[20]
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. Measurement performed on duplicate samples rather than triplicate samples

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.[1]

- Nitrosamines were not detected in the Tank 50 salt solution sample above the instrument detection limits of <1 mg/L.

- The minimum detection limit (<2.68E-01 pCi/mL) is reported for ^{94}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.[4] However, the minimum detection limit reported for the Second Quarter CY21 Tank 50 sample for ^{94}Nb is lower than the estimated detection limit of 4.38E-01 pCi/mL initially established by SRNL in 2009.[5] Thus per guidance from SRR,[4] SRNL continues to achieve as low as practical detection limits for this radionuclide.

5.0 References

[1] Harrington, S. J., "Waste Acceptance Criteria for Transfers to the Z-Area Saltstone Production Facility During Salt Disposition Integration (SDI) (U)", Savannah River Remediation, X-SD-Z-00004, Rev. 4, March 2021.

[2] Condon, W. A., "Routine Saltstone Support for Salt Solution and Grout Analyses – FY2021", Savannah River Remediation, X-TTR-Z-00023, Rev. 0, October 2020.

[3] Crawford, C. L., Hill, K. A., "Task Technical and Quality Assurance Plan for Salt Solution and Grout Sample Preparation and Analyses FY2021 ", Savannah River National Laboratory, SRNL-RP-2020-00602, Rev. 0, October 2020.

[4] Dixon, D. B., "Minimum Detection Limits for Saltstone Quarterly WAC Analyses", Savannah River Remediation, SRR-WSE-2013-00005, Rev. 1, January 2013.

[5] 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.

[6] Crawford, C. L., "Results for the Second Quarter Calendar Year 2021 Tank 50 Salt Solution Sample: Cs-137", Savannah River National Laboratory, SRNL-L3100-2021-00014, Rev.0, May 2021.

[7] Crawford, C. L., "2Q CY21 Tank 50 WAC Characterization", B9108-00327-14, SRNL E-Notebook (Production), Savannah River National Laboratory, April 2021.

[8] "Gas Chromatography/Mass Spectrometry for Volatile Organics: Contract Laboratory Program Methods", Manual L16.1, Procedure ADS-2656, Rev. 9, October 2016.

[9] "Gas Chromatography/Mass Spectrometry for Semivolatile Organics Including Polychlorinated Biphenyls", Manual L16.1, Procedure ADS-2657, Rev. 7, May 2016.

[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 July 9, 2021).

[13] White, T. L., Brown, L. W., Looney, B. B and Jones, M. A., "Total Mercury Analysis Comparison Deployment of Analytical Method for the Savannah River Site Liquid Waste System", Savannah River National Laboratory, SRNL-STI-2019-00056, Rev. 0, July 2019.

[14] Boggess, A. J., Bannochie, C. J., White, T. L., Jones, M. A. and Edwards, T. B., "Methylmercury and Ethylmercury Analytical Performance in SRR Samples Measured by SRNL and Eurofins Frontier Global Sciences", Savannah River National Laboratory, SRNL-STI-2018-00250, Rev. 0, July 2019.

[15] Boggess, A. J., White, T. L., Jones, M. A., Edwards, T. B. and Harris, S. P., "Development and Comparison of Purgeable Mercury Values in SRR Samples Measured by SRNL and Eurofins FGS", Savannah River National Laboratory, SRNL-STI-2019-00300, Rev. 0, September 2019.

[16] Boggess, A. J., Jones, M. A., White, T. L., "Analysis of Ionic Mercury Species in SRR Samples Measured by SRNL and Eurofins FGS", Savannah River National Laboratory, SRNL-STI-2020-00081, Rev. 0, May 2020.

[17] "Best Handling Practices for Elemental Mercury, Organo-Mercury Compounds, and Inorganic Mercury Compounds", SRNL-TR-2019-00243, Rev. 1, June 2020.

[18] "Technical Reviews", Savannah River Site, Manual E7, Procedure 2.60, Latest Revision.

[19] "Savannah River National Laboratory Technical Report Design Check Guidelines", Westinghouse Savannah River Company, WSRC-IM-2002-00011, Rev. 2, August 2004.

[20] *CRC Handbook of Chemistry and Physics,* 102nd Print Edition, Internet Version 2021; Section 6: Fluid Properties. Edited by Rumble, J. R., CRC Press Taylor and Francis Group, Boca Raton, FL, Internet Version 2021. http://hbcponline.com/faces/contents/ContentsSearch.xhtml (accessed July 9, 2021).

Distribution:

Name:	
F. L. Armstead	D. E. Kucab
J. P. Arnold	C. A. Langton
M. J. Barnes	J. D. Ledbetter
E. P. Barrowclough	B. Lee
J. M. Benedict	K. R. Liner
M. N. Borders	K. S. Lott
J. M. Bricker	J. Manna
M. A. Broome	K. B. Martin
K. M. Brotherton	D. J. McCabe
N. F. Chapman	G. A. Morgan
J. H. Christian	P. W. Norris
W. A. Condon	J. E. Occhipinti
J. M. Conley	F. M. Pennebaker
A. D. Cozzi	M. M. Potvin
C. L. Crawford	J. D. Rahming
W. B. Dean	W. G. Ramsey
D. P. Diprete	J. W. Ray
K. D. Dixon	C. Ridgeway
R. E. Edwards	L. B. Romanowski
C. M. Gregory	K. H. Rosenberger
S. J. Harrington	E. T. Sadowski
E. W. Harrison	A. Samadi-Dezfouli
C. C. Herman	F. M. Smith
K. A. Hill	A. V. Staub
P. J. Hill	M. E. Stone
A. T. Hooker	K. R. Wells
T. H. Huff	B. J. Wiedenman
H. M. Hunter	T. L. White
J. F. Iaukea	M. L. Whitehead
V. Jain	A. W. Wiggins
R. C. Jolly	Records Administration (EDWS)
A. W. Jung	