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Results for the Fourth Quarter Calendar Year 2020 Tank 50 Salt Solution Sample

C. L. Crawford

March 2021

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REVIEWS AND APPROVALS

AUTHOR:

CHARLES CRAWFORD (Affiliate) Digitally signed by CHARLES CRAWFORD (Affiliate)
Date: 2021.03.01 16:54:03 -05'00'

C. L. Crawford, Author, Separation Sciences & Engineering

TECHNICAL REVIEW:

JONATHAN CHRISTIAN (Affiliate) Digitally signed by JONATHAN CHRISTIAN (Affiliate)
Date: 2021.03.01 17:26:46 -05'00'

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

APPROVAL:

BOYD WIEDENMAN (Affiliate) Digitally signed by BOYD WIEDENMAN (Affiliate)
Date: 2021.03.01 17:54:00 -05'00'

B. J. Wiedenman, Manager
Separation Sciences & Engineering

FRANK PENNEBAKER (Affiliate) Digitally signed by FRANK PENNEBAKER (Affiliate)
Date: 2021.03.02 06:39:44 -05'00'

F. M. Pennebaker, Program Manager
Chemical Processing Sciences

SAMUEL FINK (Affiliate) Digitally signed by SAMUEL FINK (Affiliate)
Date: 2021.03.02 07:36:29 -05'00'

S. D. Fink, Director
Chemical Processing Sciences

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 Fourth Quarter Calendar Year 2020 (CY20) 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.¹ The chemical and radionuclide contaminant results from the characterization of the Fourth Quarter CY20 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. However, data pertaining to the regulatory limits for Resource Conservation and Recovery Act (RCRA) metals for this current 4QCY20 Tank 50 sample will be obtained and documented per the special analysis associated with the vault classification sample as described in the associated TTQAP.⁴

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 Analyzed average values for Cs-137 and Ba-137m are higher than the WAC Target and Limit, respectively, for Salt Disposition Integration (SDI).¹ This Tank 50 sample was obtained in late October of 2020 with a minimum processing of salt batch solution through the Salt Waste Processing Facility (SWPF). As more salt batch processing occurs through SWPF, it is expected that the Cs-137 concentrations in Tank 50 will decrease in the future.
- Nitrosamines were not detected in the Tank 50 salt solution sample above the instrument detection limits of <1 mg/L.
- The minimum detection limit (<2.82E-01 pCi/mL) 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.⁵ However, the minimum detection limit reported for the Fourth Quarter CY20 Tank 50 sample for ⁹⁴Nb is lower than the estimated detection limit of 4.38E-01 pCi/mL initially established by SRNL in 2009.⁶ Thus per guidance from SRR,⁵ SRNL continues to achieve as low as practical detection limits for this radionuclide.

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LIST OF ABBREVIATIONS

AR&D	Analytical Research and 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
LSC	Liquid Scintillation Counting
MRL	Minimum Reporting Limit
PHA	Pulse Height Analysis (alpha PHA)
RCRA	Resource Conservation and Recovery Act
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
TCCR	Tank Closure Cesium Removal
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.⁷

2.0 Experimental

2.1 Technical

The Fourth Quarter CY20 Tank 50 samples [a 200-mL sample obtained 6” below the surface (HTF-50-20-96) and a 3-L variable depth sample (VDS) obtained 66” from the tank bottom (HTF-50-20-97)] were obtained on October 27, 2020, and received at Savannah River National Laboratory (SRNL) on October 27, 2020.⁸

Appendix A shows all the transfers associated with Tank 50 dating from 8/7/19 when the previous 3Q19 Tank 50 WAC sample was obtained⁹ through 10/27/19 when the 4Q20 Tank 50 WAC sample was obtained.⁸ These data indicate 5,862 gallons of ‘internal additions’ such as bearing water and other material occurred from August 2019 through March of 2020. A total of 71,709 gallons of treated salt solution from SWPF was received into Tank 50 from 10/21/20 through 10/27/20. Other transfers into Tank 50 from the ETF and H-area totaling 43,944 gallons occurred during September 2019 through April 2020. A total of 256,054 gallons of Tank 50 material was transferred out to Z area during the month of October 2020.

The contents of the 3-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 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 1-L sample within the variable depth sampler inside the SRNL Shielded Cells Facility. 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 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 Fourth Quarter CY20 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 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 Analytical Research and Development (AR&D) Procedure L16.1, ADS-2656.¹⁰ 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 AR&D Procedure L16.1, ADS-2657.¹¹ Both of these methods use discrete standards as detailed in the procedures.^{10,11} The SVOA method uses organic solvents to extract SVOA analytes that are analyzed by GC/MS. A 3 mL dichloromethane (also known as methylene chloride, CH₂Cl₂) 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 (C₆H₁₄) 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.¹² 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 °C. 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 free hydroxide concentration [OH⁻] is measured via a wet chemical ‘Total Base’ software-controlled auto titrator technique that measures total base, free hydroxide and other base excluding carbonate. Carbonate is excluded by the addition of 0.5 M BaCl₂ solution prior to the titration. A standard consisting of Al (NO₃)₃ • 9H₂O (aluminum nitrate nonahydrate), NaOH (sodium hydroxide), and Na₂CO₃ (sodium carbonate, anhydrous) is used as reference in this measurement. 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. Densities were measured on triplicate samples of the Tank 50 slurry by SRNL AR&D. 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.¹³

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.¹⁴ The Cs-137 and C-134 radionuclides are determined from gamma spectroscopy. Total beta is measured from a radscreen method using Liquid Scintillation Counting

^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.

(LSC). The total alpha is measured from the same method after removal of Cs-137 from the sample using ammonium phosphomolybdate.

Mercury analyses performed at SRNL AR&D included total mercury using the Direct Mercury Analyzer (DMA) method and monomethyl mercury and ethyl mercury by Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS). Other mercury (Hg) species shown in Table 3-1, Table 3-2 and Table 3-5 will be calculated from separate aliquots analyzed by Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS) by the Eurofins Frontier Global Sciences, Inc. laboratory in Seattle, WA, and reported in a separate memorandum at a later time. 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. 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.¹⁵ Monomethyl and ethyl mercury 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. All samples sent to Eurofins Frontier Global Sciences, Inc., for analysis are diluted by ~ 2,500X by adding a 0.1 mL aliquot of Tank 50 sample to a total of 250 mL reagent water supplied by Eurofins. These diluted samples for monomethyl and ethyl Hg also contain ~ 0.4% high purity HCl acid. The Hg species routinely reported from Eurofins include total mercury, monomethyl mercury, elemental mercury (Hg (0)), ethyl mercury, ionic mercury and dimethyl mercury. Monomethyl, ethyl, and dimethyl mercury are organomercury species. Samples of Tank 50 submitted to SRNL AR&D for total Hg and methyl Hg analysis were submitted without dilution. These samples were diluted within the AR&D laboratories to meet the targeted calibration range of either the DMA instrument for total Hg or the CVAFS instrument for methyl Hg.

2.2 Quality Assurance

Quality Assurance requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60.¹⁶ SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.¹⁷ 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 calculated from either triplicate or duplicate values 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 1-sigma standard deviations calculated from either triplicate or duplicate values 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.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 18%, 22% and 11%, 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 59%, with average TOC at a lower ratio to WAC Target of 28%. 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 WAC¹ Limits and Targets are shown in Table 3-3 and Table 3-4, respectively. The minimum detection limit reported for Nb-94 of $<2.82\text{E-}01$ pCi/mL in Table 3-4 is above the 2013-requested SRR target minimum detection limit of $2.0\text{E-}03$ pCi/mL “to meet future inventory reporting requirements”⁵ but is lower than the estimated detection limit initially established by SRNL of $4.38\text{E-}01$ pCi/mL in 2009.⁶ 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 $1.52\text{E+}02$ pCi/mL shown in Table 3-4.

Comparison of the average analyzed detectable values shown in Table 3-3 to the WAC Limits¹ indicates that Tc-99 and I-129 are the highest analytes relative to the WAC Limits at 13% and 16%, respectively. The Cs-137 value is 1.19X times the SDI WAC Limit¹ as previously reported.⁷ Comparison of the average analyzed detectable values shown in Table 3-4 to the WAC Targets indicates that Pu-238 is the highest analyte relative to the WAC Targets¹ at 18%. The Ba-137m value is 1.19X times the SDI WAC Limit.¹ Table 3-4 indicates that the upper limit determined in the triplicate samples analyzed for total alpha for the 4Q20 sample is $1.28\text{E+}04$ pCi/mL.

Table 3-1. Chemical Contaminants from Fourth Quarter CY20 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	1.44E+04 ^a	2.48E+01	4.08E+05
Ammonium (NH ₄ ⁺)	IC	<1.00E+01	NA	2.12E+02
Carbonate (CO ₃ ²⁻)	TIC	1.64E+04 ^b	7.63E+01	1.20E+05
Chloride (Cl ⁻)	IC	5.36E+02	3.06E+00	7.95E+03
Fluoride (F ⁻)	IC	<1.00E+01	NA	4.07E+03
Free Hydroxide (OH ⁻)	Total Base	2.83E+04 ^b	1.57E+03	1.58E+05
Nitrate (NO ₃ ⁻)	IC	9.64E+04	9.50E+02	4.37E+05
Nitrite (NO ₂ ⁻)	IC	1.98E+04	5.77E+01	2.14E+05
Oxalate (C ₂ O ₄ ²⁻)	IC	6.61E+02	1.42E+01	2.72E+04
Phosphate (PO ₄ ³⁻)	IC	1.96E+02	2.89E+00	3.14E+04
Sulfate (SO ₄ ²⁻)	IC	6.37E+03	1.25E+02	5.69E+04
Arsenic (As)	ICP-MS	<2.80E-01	NA	1.97E+02
Barium (Ba)	ICP-ES	1.90E+01 ^c	7.94E-01	6.19E+02
Cadmium (Cd)	ICP-ES	<4.04E+00	NA	3.10E+02
Chromium (Cr)	ICP-ES	3.56E+01	3.92E-01	1.50E+03
Lead (Pb)	ICP-MS	3.58E-01	1.49E-02	7.50E+02
Total Mercury (Hg)	DMA	3.60E+01	1.54E+00	3.25E+02
Elemental Mercury (Hg(0))	CVAFS	d	--	3.25E+02
Monomethyl Mercury (CH ₃ Hg)	CVAFS	2.24E+01	2.26E+00	3.50E+02
Ethyl Mercury (C ₂ H ₅ Hg)	CVAFS w/ Distillation	<1	NA	3.73E+02
Selenium (Se)	ICP-MS	<3.65E-01	NA	3.75E+02
Silver (Ag)	ICP-ES	<4.84E+00	NA	6.19E+02
Aluminum (Al)	ICP-ES	4.10E+03	7.04E+00	1.16E+05
Potassium (K)	ICP-ES	3.99E+02	1.37E+01	3.03E+04
Butanol (C ₄ H ₉ OH)	VOA	<2.00E-01 ^e	NA	7.73E+00
Propanol (C ₃ H ₇ OH)	VOA	<2.00E-01 ^e	NA	1.88E+00
Phenol (C ₆ H ₅ OH)	SVOA	<1.00E+00 ^e	NA	7.50E+02
Tetraphenylborate [TPB] (B(C ₆ H ₅) ₄ ⁻)	HPLC	<5.00E+00	NA	5.00E+00
Total Organic Carbon (----)	TOC	2.08E+02 ^b	1.53E+00	4.50E+03
Isopar L (----)	SVOA	<3.30E+01 ^f	NA	8.75E+01

- Result is calculated from the measured Al concentration assuming all the Al is present as the aluminate species.
- Measurement performed on filtered supernate samples.
- A similar value was also determined for the blank, so the average value reported is likely high biased.
- This Hg species will be reported in a separate memorandum.
- Measurement performed on duplicate samples rather than triplicate samples.
- Measurement performed on single sample rather than triplicate samples.

Table 3-2. Chemical Contaminants from Fourth Quarter CY20 Tank 50 Samples and SPF WAC, Attachment 8.2 Targets

<u>Chemical Name (Formula)</u>	<u>Method</u>	<u>Average Concentration (mg/L)</u>	<u>Std. Dev.</u>	<u>WAC Target (mg/L)</u>
Aluminum (Al)	ICP-ES	4.10E+03	7.04E+00	7.00E+03^f
Boron (B)	ICP-ES	3.12E+01	1.61E+00	7.43E+02
Cobalt (Co)	ICP-MS ^a	<2.80E-02 ^a	NA	1.45E+02
Copper (Cu)	ICP-ES	<3.11E+00	NA	7.43E+02
Iron (Fe)	ICP-ES	3.16E+01 ^b	2.69E+01	4.95E+03
Lithium (Li)	ICP-ES	<5.82E+00	NA	7.43E+02
Manganese (Mn)	ICP-ES	<1.78E+00	NA	7.43E+02
Molybdenum (Mo)	ICP-ES	<1.60E+01	NA	7.43E+02
Nickel (Ni)	ICP-ES	<6.95E+00	NA	7.43E+02
Silicon (Si)	ICP-ES	<3.72E+01	NA	1.07E+04
Strontium (Sr)	ICP-ES	<3.92E-01	NA	7.43E+02
Zinc (Zn)	ICP-ES	1.08E+01	3.08E-01	8.03E+02
Dimethyl Mercury ((CH₃)₂Hg)	CVAFS	c	--	1.00E+00
Benzene (C₆H₆)	VOA	<2.00E-01 ^d	NA	3.10E+02
Methanol (CH₃OH)	VOA	e	NA	1.88E+00
Toluene (C₆H₅CH₃)	VOA	<2.00E-01 ^d	NA	3.10E+02
Dibutylphosphate [DBP] (C₈H₁₉O₄P)	IC	<2.50E+02	NA	3.47E+02
Tributylphosphate [TBP] ((C₄H₉O)₃PO)	SVOA	<7.50E-01 ^d	NA	7.50E+00
Total Organic Carbon (TOC)	TOC	2.08E+02	1.53E+00	7.50E+02^f
EDTA (C₁₀H₁₂N₂O₈⁴⁻)	HPLC	<1.00E+02	NA	3.10E+02
NORPAR 13 (C_nH_{2-n})	SVOA	<7.50E-01 ^g	NA	7.50E-01
Formate (CHOO⁻)	IC	<1.00E+01	NA	6.38E+03

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

b. Only two detectable values were determined from the triplicate sample set.

c. Dimethyl mercury will be reported in a separate memorandum.

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

e. Currently, a routine method for detecting this species does not exist in AR&D.

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.¹

g. Measurement performed on a single sample rather than triplicate samples.

Table 3-3. Radionuclide Contaminants from Fourth Quarter CY20 Tank 50 Samples and SPF WAC, Attachment 8.3 Limits

<u>Radionuclide</u>	<u>Method</u>	<u>Average Concentration (pCi/mL)</u>	<u>Std. Dev.</u>	<u>WAC Limit (pCi/mL)</u>
Tritium (³H)	Tritium Counting	6.42E+02	4.75E+01	5.63E+05
Carbon-14 (¹⁴C)	C-14 Liquid Scintillation	5.93E+02	5.74E+01	1.13E+05
Nickel-63 (⁶³Ni)	Ni-59/63	<6.44E+00	NA	1.13E+05
Strontium-90 (⁹⁰Sr)	Sr-90 Liquid Scintillation	6.55E+04	6.00E+03	2.62E+06
Technetium-99 (⁹⁹Tc)	Tc-99 Liquid Scintillation	2.66E+04	1.62E+03	2.11E+05
Iodine-129 (¹²⁹I)	I-129 (w/ separation) Liquid Scintillation	1.64E+01	1.20E+00	1.00E+02
Cesium-137 (¹³⁷Cs)	Gamma Scan	1.53E+06	1.04E+04	1.29E+06
Uranium-233 (²³³U)	ICP-MS	<2.71E+02	NA	1.13E+04
Uranium-235 (²³⁵U)	ICP-MS	1.86E-01	4.39E-03	1.13E+02
Plutonium-241 (²⁴¹Pu)	Pu238/241 Liquid Scintillation	2.61E+03	3.78E+02	8.38E+05
Total Alpha	Liquid Scintillation Counting (Cs removed)	<1.28E+04	NA	2.13E+05

Table 3-4. Radionuclide Contaminants from Fourth Quarter CY20 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)	<9.41E-02	N/A	2.88E+03
Potassium-40 (⁴⁰ K)	Gamma Scan (Cs removed)	<2.91E+00	NA	1.00E+02
Cobalt-60 (⁶⁰ Co)	Gamma Scan (Cs removed)	<2.72E-01	NA	9.75E+02
Nickel-59 (⁵⁹ Ni)	Ni-59/63	<1.99E+00	NA	1.13E+03
Selenium-79 (⁷⁹ Se)	Se-79	1.93E+01 ^a	NA	1.90E+04
Yttrium-90 (⁹⁰ Y)	Secular Equilibrium w/ 100% of Sr-90	6.55E+04	6.00E+03	2.62E+06
Zirconium-93 (⁹³ Zr)	ICP-MS	<7.05E+01	NA	1.00E+05
Niobium-94 (⁹⁴ Nb)	Nb-94	<2.82E-01	NA	1.53E+02
Rhodium-106 (¹⁰⁶ Rh)	Secular Equilibrium w/ 100% of Ru-106	<4.59E+00	NA	3.12E+05
Ruthenium-106 (¹⁰⁶ Ru)	Gamma Scan (Cs removed)	<4.59E+00	NA	3.12E+05
Antimony-125 (¹²⁵ Sb)	Gamma Scan (Cs removed)	1.74E+01	5.41E-01	7.99E+03
Tellurium-125m (^{125m} Te)	Secular Equilibrium w/ 100% of Sb-125	1.74E+01	5.41E-01	1.83E+03
Tin-126 (¹²⁶ Sn)	Gamma Scan (Cs removed)	2.45E+02	1.42E+01	1.80E+04
Cesium-134 (¹³⁴ Cs)	Gamma Scan	<1.16E+03	NA	5.93E+03 ^a
Cesium-135 (¹³⁵ Cs)	Cs-135	8.69E+00	3.52E-01	2.50E+02
Barium-137m (^{137m} Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137)	1.45E+06	9.84E+03	1.22E+06
Cerium-144 (¹⁴⁴ Ce)	Gamma Scan (Cs removed)	<8.69E+00	NA	3.12E+04
Praseodymium-144 (¹⁴⁴ Pr)	Secular Equilibrium w/ 100% of Ce-144	<8.69E+00	NA	3.12E+04
Promethium-147 (¹⁴⁷ Pm)	Pm-147/Sm-151 Liquid Scintillation	<4.21E+01	NA	1.57E+06
Samarium-151 (¹⁵¹ Sm)	Pm-147/Sm-151 Liquid Scintillation	<4.11E+01	NA	2.25E+04
Europium-154 (¹⁵⁴ Eu)	Gamma Scan (Cs removed)	<8.56E-01	NA	1.62E+03
Radium-226 (²²⁶ Ra)	Ra-226	<4.07E+00	NA	1.00E+03
Radium-228 (²²⁸ Ra)	Gamma Scan (Cs removed)	<1.94E+00	NA	1.00E+04
Actinium-227 (²²⁷ Ac)	Th-229/230	<2.32E-02	NA	1.00E+04
Thorium-229 (²²⁹ Th)	Th-229/230	<3.85E-02	NA	1.63E+05
Thorium-230 (²³⁰ Th)	Th-229/230	<8.78E-02	NA	6.26E+03
Thorium-232 (²³² Th)	ICP-MS	<3.08E-03	NA	2.88E+03
Protactinium-231 (²³¹ Pa)	Pa-231	<1.18E+01	NA	1.00E+03
Uranium-232 (²³² U)	U-232	7.70E+00 ^a	NA	2.27E+03
Uranium-233 (²³³ U)	ICP-MS	<2.71E+02	NA	3.12E+03
Uranium-234 (²³⁴ U)	ICP-MS	<1.75E+02	NA	3.12E+03
Uranium-236 (²³⁶ U)	ICP-MS	<1.82E+00	NA	3.12E+03
Uranium-238 (²³⁸ U)	ICP-MS	2.00E+00	5.13E-03	3.12E+03

Table 3-4. Radionuclide Contaminants from Fourth Quarter CY20 Tank 50 Samples and SPF WAC, Attachment 8.4 Targets, 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	<1.98E+01	NA	1.00E+04
Plutonium-238 (²³⁸ Pu)	Pu238/241 Pu alpha PHA	1.18E+04	9.24E+02	6.67E+04
Plutonium-239 (²³⁹ Pu)	Pu238/241 Pu alpha PHA	3.79E+02	3.07E+01	6.67E+04
Plutonium-240 (²⁴⁰ Pu)	Pu238/241 Pu alpha PHA	3.79E+02	3.07E+01	6.67E+04
Plutonium-242 (²⁴² Pu)	ICP-MS	< 1.07E+02	NA	6.67E+04
Plutonium-244 (²⁴⁴ Pu)	ICP-MS	< 4.97E-01	NA	7.02E+04
Americium-241 (²⁴¹ Am)	Am/Cm	3.11E+00	1.70E+00	6.67E+04
Americium-242m (^{242m} Am)	Am/Cm	<1.09E+00	NA	4.50E+05
Americium-243 (²⁴³ Am)	Am/Cm	<5.90E-01	NA	6.67E+04
Curium-242 (²⁴² Cm)	Am/Cm	<8.96E-01	NA	1.13E+04
Curium-244 (²⁴⁴ Cm)	Am/Cm	2.11E+00	1.76E+00	6.67E+04
Curium-245 (²⁴⁵ Cm)	Am/Cm	<5.81E+00	NA	2.25E+05
Total Alpha	Liquid Scintillation Counting (Cs removed)	<1.28E+04	NA	6.67E+04

a. Only a single detectable value was reported for these radionuclides.

The following tables show various chemical contaminants (Table 3-5), organic species (Table 3-6) and processing constituents (Table 3-7) related to the SDU that are referred to in the WAC per Tables 2, 4 and 5, respectively.¹ The reported detection limit for Isopar L of <2.70E+01 ppm in Table 3-5 is lower than the current Isopar limit of 87.5 ppm associated with SDU flammability for the 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 Fourth Quarter CY20 Tank 50 Samples and WAC Table 2 Limits and Targets

<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	< 2.70E+01 ppm ^{a,b}	NA	8.75E+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+01	NA	2.12E+02 mg/L (Limit)
Total Mercury (Hg)	DMA	3.60E+01	1.54E+00	3.25E+02 mg/L (Limit)
Monomethyl Mercury (CH ₃ Hg)	CVAFS	2.24E+01	2.26E+00	3.50E+02 mg/L (Limit)
Dimethyl Mercury ((CH ₃) ₂ Hg)	CVAFS	c	--	1.00E+00 mg/L (Target)

a. Measurement performed on a single sample 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. Monomethyl mercury and Dimethyl mercury will be reported at a later time.

Table 3-6. Other Organics Impacting SDU Flammability from Fourth Quarter CY20 Tank 50 Samples and WAC Table 4 Concentrations

<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) ^a	VOA	<2.00E-01	NA	0.75
Tributylphosphate[TBP] ((C ₄ H ₉ O) ₃ PO) ^a	SVOA	<7.50E-01	NA	1.0
Propanol (C ₃ H ₇ OH) ^a	VOA	<2.00E-01	NA	0.25
Methanol (CH ₃ OH)	b	NA	NA	0.05
NORPAR 13 (C _n H _{2.n}) ^c	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 AR&D.
c. Measurement performed on a single sample rather than triplicate samples.

Table 3-7. Processing Constituents from Fourth Quarter CY20 Tank 50 Samples and WAC Table 5 Limits

<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	4.83 M	3.06E-02	2.5 M < [Na ⁺] < 7.0 M
Total Insoluble Solids	Calculated	~0 wt %	NA	< 15 wt %

*pH estimated from pH equation (pH + pOH = 14) with measured [OH⁻] from Table 3-1.

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

<u>Constituent</u>	<u>Method</u>	<u>Average Value</u>	<u>Std. Dev.</u>
Density (slurry)	Measured (21.1°C)	1.220 g/mL	0.000 g/mL ^b
Specific Gravity	a	1.222	0.000 ^b
Total Solids	Measured	24.92 wt %	0.20 wt %
Total Beta	LSC	2.14E+06 pCi/mL	1.62E+04 pCi/mL
Total Gamma	c	1.45E+06 pCi/mL	5.57E+03 pCi/mL ^d
Beryllium (Be)	ICP-ES	<1.45E-01 mg/L	NA
N-Nitrosodimethylamine (C ₂ H ₆ N ₂ O)	SVOA	<1 mg/L	NA
N-Dioctylnitrosamine (C ₁₆ H ₃₄ N ₂ O)	SVOA	<1 mg/L	NA

a. Calculated from the measured density of slurry and density of water at 21.1 °C.¹⁸

b. All triplicate density for slurry were equivalent as well as all triplicate density for water were equivalent.

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

d. 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.¹ Analyzed average values for Cs-137 and Ba-137m are higher than the WAC Target and Limit, respectively, for Salt Disposition Integration (SDI).¹ This Tank 50 sample was obtained in late October of 2020 with a minimum processing of salt batch solution through the Salt Waste Processing Facility (SWPF). As more salt batch processing occurs through SWPF, it is expected that the Cs-137 concentrations in Tank 50 will decrease in the future.
- Nitrosamines were not detected in the Tank 50 salt solution sample above the instrument detection limits of <1 mg/L.
- The minimum detection limit (<2.06E-01 pCi/mL) 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 from 2013.⁵ However, the minimum detection limit reported for the Fourth Quarter CY20 Tank 50 sample for ⁹⁴Nb is lower than the estimated detection limit of 4.38E-01 pCi/mL initially established by SRNL in 2009.⁶ Thus per guidance from SRR,⁵ SRNL continues to achieve as low as practical detection limits for this radionuclide.

Appendix A
Tank 50 Transfers After 3Q19 WAC Sample (8/7/19) Through 4Q20 WAC Sample
(10/27/20)

Tank:		50	From:	8/17/19	To:	10/27/20
	Sending Location	Receiving Location	Amount	Comments		
Internal Addition						
03/18/2020	Bearing Water	Tank 50	20	TK 50 PUMP RECIRC		
11/03/2019	Flush Water	Tank 50	296	DEMISTER FLUSH.		
08/23/2019	Other	Tank 50	3,510	DRAINING TO TK 50 FR		
08/22/2019	Other	Tank 50	2,036	PUMPED BRENNER TK		
Receipt						
10/27/2020	SWPF	Tank 50	29,905	DSSHT to Tk-50 Transfer		
10/23/2020	SWPF	Tank 50	28,150	DSST to tank 50		
10/21/2020	SWPF	Tank 50	13,654	DSSHT to Tk 50 Transfer		
04/30/2020	ETF	Tank 50	19,094	--		
01/08/2020	ETF	Tank 50	22,640	ETP TO TK 50		
10/08/2019	211H	Tank 50	105	710 vessel to tk 50		
09/28/2019	211H	Tank 50	456	7.10 vessel transfer to Tk 50		
09/15/2019	211H	Tank 50	526	HCAN 7.10 TO TANK 50		
09/08/2019	211H	Tank 50	456	7.10 vessel to Tank 50		
09/02/2019	211H	Tank 50	667	710 transfer to tk 50		
Transfer						
10/20/2020	Tank 50	Z Area	36,434	Tk 50 to Z Area Transfer		
10/19/2020	Tank 50	Z Area	30,151	--		
10/15/2020	Tank 50	Z Area	35,486	TANK 50 TO Z AREA		
10/14/2020	Tank 50	Z Area	28,361	TANK 50 TO Z-AREA TR		
10/13/2020	Tank 50	Z Area	34,644	TANK 50 TO Z-AREA TR		
10/12/2020	Tank 50	Z Area	19,094	--		
10/11/2020	Tank 50	Z Area	26,711	--		
10/10/2020	Tank 50	Z Area	28,220	--		
10/07/2020	Tank 50	Z Area	5,405	TK 50 TO Z		
10/06/2020	Tank 50	Z Area	11,548	--		

5.0 Reference

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