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Results for the Fourth Quarter Calendar Year 2022 Salt Solution Sample for Performance Assessment Analyses

C. L. Crawford

June 2023

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EXECUTIVE SUMMARY

In this Technical Report, the chemical and radionuclide contaminant results from the fourth quarter calendar year 2022 salt solution sample for Performance Assessment (PA) analyses are presented in tabulated form. The information from this characterization will be used by Savannah River Mission Completion (SRMC) for the transfer of aqueous waste to Tank 50 and 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 salt solution sample was obtained.¹ The chemical and radionuclide contaminant results from the characterization of the fourth quarter calendar year 2022 sampling of salt solution sent to Z area were requested by SRMC personnel via a Technical Task Request (TTR).² 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 the TTR deliverable relating Salt Solution Analysis from the SRMC request.²

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

- WAC LIMITS and TARGETS were met for all analyzed chemical and radioactive contaminants for which the detection limits are below the WAC LIMITS and TARGETS.¹
- Measured average concentrations of nitrate, nitrite and total mercury are approximately 24%, 9% and 17% of the WAC LIMITS, respectively.
- Measured average concentrations of Tc-99 and I-129 are approximately 22% and 24% of the WAC LIMITS, respectively.
- All other radionuclide average concentrations are at 3% or less of the WAC LIMITS and TARGETS.

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	4
3.0 Results and Discussion	4
4.0 Conclusions	6
5.0 References	7

LIST OF TABLES

Table 2-1. WDA Samples for Salt Solution Composite Sample	3
Table 3-1. Chemical Contaminants from Fourth Quarter CY22 Salt Solution Sample and SPF WAC, Attachment 8.1 LIMITS	5
Table 3-2. Radionuclide Contaminants from Fourth Quarter CY22 Salt Solution Sample and SPF WAC, Attachment 8.3 LIMITS	5
Table 3-3. Radionuclide Contaminants from Fourth Quarter CY22 Salt Solution Sample and SPF WAC, Attachment 8.4 TARGETS.....	6

LIST OF ABBREVIATIONS

ACSM	Analytical Characterization and Sample Management
DMA	Direct Mercury Analyzer
HDPE	High-Density Polyethylene
IC	Ion Chromatography
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
LSC	Liquid Scintillation Counting
MRL	Minimum Reporting Limit
Pu alpha PHA	Plutonium alpha Pulse Height Analysis
SDI	Salt Disposition Integration
SDU	Saltstone Disposal Unit
SPF	Saltstone Production Facility
SRNL	Savannah River National Laboratory
SRMC	Savannah River Mission Completion
SWPF	Salt Waste Processing Facility
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
VDS	Variable Depth Sample
WAC	Waste Acceptance Criteria
WDA	Waste Disposal Authority

1.0 Introduction

Salt Solution that is sent to Z area 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).^{1,2} This quarterly analysis is in addition to the full suite analyses of Tank 50 salt solution conducted semiannually per X-TTR-Z-00025.⁴ The information from this characterization will be used by Savannah River Mission Completion (SRMC) for the transfer of aqueous waste to Z Area 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 Chemical and radionuclide analyses of salt solution are required to demonstrate that the composition of feed received into Z-Area is in compliance with the Saltstone Performance Assessment.⁵

2.0 Experimental

2.1 Technical

The fourth quarter Calendar Year (CY22) salt solution sample was received in the SRNL on February 7, 2023. The 200-mL sample was composited from various decontaminated salt solution samples from the Salt Waste Processing Facility (SWPF) by SWPF personnel using proceduralized request forms originated by Waste Disposal Authority (WDA) personnel.⁶ Table 2-1 shows a list of the various SWPF tanks sampled (Tk-207, Tk-220, Tk-221) and the sampling dates that comprised the WDA composite sample. These forty parent samples of ~50 mL each were collected from October 1, 2022 through December 30, 2022. Approximately 20 mL from each parent sample was then composited into a 1-L plastic bottle to create ~800 mL of composite sample at the SWPF. From that composite sample, three equivalent 200-mL aliquots were obtained and placed in 250-mL plastic bottles. One of these 200-mL aliquot samples was then transferred from SWPF to SRNL for analysis.

The salt solution sample was received into the SRNL Shielded Cells Facility and then transferred into a radiochemical hood for handling. The contents of the 200-mL salt solution in a plastic bottle were initially mixed by swirling the container before opening. No visible insoluble solids were observed in the salt solution sample. After initial mixing, an aliquot of the salt solution sample was poured into a 30-mL Teflon vial with near zero headspace. This sample was used for Hg analysis. The zero-headspace vial for Hg testing was transferred to storage in a refrigerator.⁷ Aliquots of the salt solution sample were promptly collected with slurry pipettes to minimize settling effects and placed into High-Density Polyethylene (HDPE) bottles.

Unless otherwise stated, all concentrations presented in the tables (except upper limits) are averages based on analyses of triplicate aliquots of the January quarterly CY23 salt solution 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.

Data reported for inductively coupled plasma mass spectrometry (ICP-MS) are derived from the digested salt solution sample (1 mL supernate diluted to 50 mL total volume) by the aqua regia method.⁸ The aqua regia method heats the salt solution sample 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 analyses are determined from Ion Chromatography (IC) analysis of the as-submitted salt solution sample. Approximately 150 mL of the sample was 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.⁹ The Cs-137 radionuclide is determined from gamma spectroscopy. Plutonium isotopes (Pu-238, Pu-239/Pu-240) are determined from a Plutonium alpha Pulse Height Analysis (Pu alpha PHA) method. The plutonium isotopes Pu-239 and Pu-240 cannot be separated using this method.

Mercury analyses performed at SRNL by Analytical Characterization and Sample Management (ACSM) included Total Mercury using the Direct Mercury Analyzer (DMA) method.¹⁰ As discussed above, the salt solution sample for mercury analysis was obtained in a near zero-headspace container that was immediately refrigerated after removal from the radiochemical hood on the same day of preparation.⁷

Table 2-1. WDA Samples for Salt Solution Composite Sample

WDA Sample Request List - 2022.Q4				Corresponding WAC Request
Sample	Request	Tank	Collection Date	
1 S-220930-00022	R-20220930-00002	TK-221	10/01/2022 03:42	R-20220930-00001
2 S-221001-00236	R-20221001-00002	TK-220	10/02/2022 07:34	R-20221001-00001
3 S-221003-00022	R-20221003-00002	TK-221	10/03/2022 13:35	R-20221003-00001
4 S-221007-00022	R-20221007-00002	TK-220	10/07/2022 21:54	R-20221007-00001
5 S-221008-00029	R-20221008-00002	TK-221	10/09/2022 02:14	R-20221008-00001
6 S-221009-00085	R-20221009-00002	TK-220	10/10/2022 12:09	R-20221009-00001
7 S-221013-00022	R-20221013-00002	TK-221	10/14/2022 11:10	R-20221013-00001
8 S-221015-00031	R-20221015-00003	TK-220	10/15/2022 16:00	R-20221015-00002
9 S-221016-00022	R-20221016-00002	TK-221	10/16/2022 21:44	R-20221016-00001
10 S-221017-00022	R-20221017-00002	TK-220	10/18/2022 04:05	R-20221017-00001
11 S-221020-00115	R-20221020-00002	TK-221	10/21/2022 02:00	R-20221020-00001
12 S-221021-00298	R-20221021-00002	TK-220	10/22/2022 04:24	R-20221021-00001
13 S-221023-00022	R-20221023-00002	TK-221	10/23/2022 08:30	R-20221023-00001
14 S-221024-00024	R-20221024-00002	TK-220	10/24/2022 16:20	R-20221024-00001
15 S-221026-00022	R-20221026-00002	TK-221	10/26/2022 07:57	R-20221026-00001
16 S-221028-00026	R-20221028-00002	TK-220	10/29/2022 00:09	R-20221028-00001
17 S-221029-00188	R-20221029-00002	TK-221	10/30/2022 07:46	R-20221029-00001
18 S-221031-00037	R-20221031-00002	TK-220	11/01/2022 02:38	R-20221031-00001
19 S-221105-00022	R-20221105-00002	TK-221	11/05/2022 19:56	R-20221105-00001
20 S-221106-00128	R-20221106-00003	TK-220	11/06/2022 21:45	R-20221106-00004
21 S-221107-00265	R-20221107-00002	TK-221	11/08/2022 13:16	R-20221107-00001
22 S-221117-00115	R-20221117-00002	TK-220	11/18/2022 01:57	R-20221117-00001
23 S-221118-00188	R-20221118-00002	TK-221	11/19/2022 09:40	R-20221118-00001
24 S-221123-00145	R-20221123-00002	TK-221	11/23/2022 19:59	R-20221123-00001
25 S-221124-00022	R-20221124-00002	TK-220	11/24/2022 22:26	R-20221124-00001
26 S-221126-00022	R-20221126-00003	TK-221	11/26/2022 11:14	R-20221126-00001
27 S-221127-00116	R-20221127-00002	TK-220	11/28/2022 09:42	R-20221127-00001
28 S-221201-00116	R-20221201-00002	TK-221	12/02/2022 19:30	R-20221201-00001
29 S-221204-00125	R-20221204-00003	TK-220	12/04/2022 15:15	R-20221204-00002
30 S-221207-00170	R-20221207-00003	TK-221	12/08/2022 01:42	R-20221207-00002
31 S-221209-00154	R-20221209-00003	TK-220	12/09/2022 15:53	R-20221209-00002
32 S-221212-00146	R-20221212-00003	TK-221	12/13/2022 08:03	R-20221212-00002
33 S-221214-00033	R-20221214-00003	TK-220	12/14/2022 16:52	R-20221214-00002
34 S-221216-00171	R-20221216-00003	TK-221	12/16/2022 15:56	R-20221216-00002
35 S-221217-00129	R-20221217-00003	TK-207	12/18/2022 19:34	R-20221217-00002
36 S-221219-00139	R-20221219-00003	TK-220	12/20/2022 14:39	R-20221219-00002
37 S-221222-00045	R-20221222-00003	TK-221	12/22/2022 09:52	R-20221222-00002
38 S-221223-00039	R-20221223-00003	TK-220	12/24/2022 05:21	R-20221223-00002
39 S-221228-00028	R-20221228-00003	TK-221	12/29/2022 02:08	R-20221228-00002
40 S-221230-00117	R-20221230-00002	TK-220	12/30/2022 17:32	R-20221230-00001

2.2 Quality Assurance

Quality Assurance requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60.¹¹ SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.¹² 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

Average analyzed nonradionuclide chemical concentrations, their 1-sigma standard deviations calculated from the triplicate measured 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 after accounting for the analytical uncertainty in each measured concentration.¹ The reported one sigma (1σ) method uncertainty for both IC and DMA is 10%. This method uncertainty is not incorporated into the reported standard deviations in Table 3-1. Comparison of the average analyzed detectable values shown in Table 3-1 to the WAC LIMITS indicates that average nitrate and nitrite anions and average total mercury are present relative to the WAC LIMITS at 24%, 9% and 17%, respectively. The average nitrate, nitrite and total mercury data for this quarterly salt solution sample relative to the WAC LIMITS are similar to the previous reported data for the September 2022 bimonthly Tank 50 WAC variable depth sample of 24%, 9% and 12%, respectively.¹³ Since comparison of the average values for nitrate, nitrite and total mercury to the WAC LIMITS shows these ratios in the range of 9% to 24% and the method uncertainty is only 10%, it is likely that these measurements are well within the WAC LIMITS.

Average analyzed radionuclide concentrations and the respective radiochemical analysis methods, their standard deviations and their corresponding WAC¹ LIMITS are shown in Table 3-2. Similar data and WAC¹ TARGETS are shown in Table 3-3. Per the WAC, the TARGETS shown shall not be exceeded after 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 1σ values that are calculated from the normal ‘standard deviation’ function for either duplicate or triplicate values from within Excel[®] spreadsheets. The reported 1σ method uncertainties are also included in Table 3-2 and Table 3-3. These uncertainties for the radiochemical analytes range from 5% to 20% for Table 3-2 and for Table 3-3 and 10% for ICP-MS. Method uncertainties are not reported for radiochemical counting analytes that are reported as ‘less than’ values. An indication of ‘minimum detectable activity’ is given for these analytes in Table 3-3.

Comparison of the average analyzed detectable values to the WAC LIMITS¹ (Table 3-2) indicates that Tc-99 and I-129 are the highest analytes relative to the WAC LIMITS at 22% and 24%, respectively. Comparison of the average analyzed detectable values to the WAC TARGETS (Table 3-3) indicates that none of the radionuclides are higher than 3% of the WAC TARGETS.¹ Since comparison of the average values for the analytes in Table 3-2 and Table 3-3 to the WAC LIMITS or TARGETS shows these ratios in the range of 3% to 24%, and the method uncertainties range from 5% to 20% for radiochemical analytes and ICP-MS data, it is likely these measurements are well within the WAC LIMITS or TARGETS.

Table 3-1. Chemical Contaminants from Fourth Quarter CY22 Salt Solution Sample and SPF WAC, Attachment 8.1 LIMITS

<u>Chemical Name</u> <u>(Formula)</u>	<u>Method /</u> <u>1σ</u> <u>Uncertainty</u>	<u>Average Concentration</u> <u>(mg/L)</u>	<u>Std. Dev.</u>	<u>WAC LIMIT</u> <u>(mg/L)</u>
Nitrate (NO ₃ ⁻)	IC / 10%	1.04E+05	1.82E+02	4.37E+05
Nitrite (NO ₂ ⁻)	IC / 10%	1.99E+04	1.54E+01	2.14E+05
Total Mercury (Hg)	DMA / 10%	5.43E+01	3.61E-01	3.25E+02

Table 3-2. Radionuclide Contaminants from Fourth Quarter CY22 Salt Solution Sample and SPF WAC, Attachment 8.3 LIMITS

<u>Radionuclide</u>	<u>Method / 1σ Uncertainty</u>	<u>Average</u> <u>Concentration</u> <u>(pCi/mL)</u>	<u>Std. Dev.</u>	<u>WAC LIMIT</u> <u>(pCi/mL)</u>
Carbon-14 (¹⁴ C)	C-14 Liquid Scintillation / 20%	7.54E+02	3.75E+01	1.13E+05
Strontium-90 (⁹⁰ Sr)	Sr-90 Liquid Scintillation / 16.9% - 18.9%	6.77E+02	2.11E+02	2.62E+06
Technetium-99 (⁹⁹ Tc)	Tc-99 Liquid Scintillation / 8.34% - 8.74%	4.68E+04	4.17E+03	2.11E+05
Iodine-129 (¹²⁹ I)	I-129 (w/ separation) Liquid Scintillation / 4.92% - 4.95%	2.42E+01	3.04E+00	1.00E+02
Cesium-137 (¹³⁷ Cs)	Gamma Scan / 5%	8.17E+03	5.20E+01	1.29E+06
Plutonium-241 (²⁴¹ Pu)	Pu238/241 Liquid Scintillation / 15.3%	4.65E+02	2.74E+01	8.38E+05

Table 3-3. Radionuclide Contaminants from Fourth Quarter CY22 Salt Solution Sample and SPF WAC, Attachment 8.4 TARGETS

Radionuclide	Method / 1 σ Uncertainty	Average Concentration (pCi/mL)	Std. Dev.	WAC TARGET (pCi/mL)
Yttrium-90 (⁹⁰Y)	Secular Equilibrium w/ 100% of Sr-90 / 16.9% - 18.9%	6.77E+02	2.11E+02	2.62E+06
Cesium-135 (¹³⁵Cs)	Cs-135 / 20%	2.92E+00	1.47E-01	2.50E+02
Barium-137m (^{137m}Ba)	Calculation (Secular Equilibrium w/ 94.6% of Cs-137) / 5%	7.73E+03	4.92E+01	1.22E+06
Thorium-230 (²³⁰Th)	Th-229/230 / minimum detectable activity	<2.78E-02	NA	6.26E+03
Uranium-234 (²³⁴U)	ICP-MS / 10%	7.24E+01	1.45E+00	3.12E+03
Uranium-238 (²³⁸U)	ICP-MS / 10%	7.45E-01	1.26E-02	3.12E+03
Plutonium-238 (²³⁸Pu)	Pu238/241 Pu alpha PHA / 5.43% - 5.54%	1.04E+03	6.31E+01	6.67E+04
Plutonium-239/240 (²³⁹Pu/²⁴⁰Pu)	Pu238/241 Pu alpha PHA / 12.3% - 14.2%	2.42E+01	1.87E+00	6.67E+04
Plutonium-242 (²⁴²Pu)	ICP-MS / not applicable	<1.24E+01	NA	6.67E+04
Americium-241 (²⁴¹Am)	Am/Cm / minimum detectable activity	<4.55E-01	NA	6.67E+04
Americium-242m (^{242m}Am)	Am/Cm / minimum detectable activity	<6.17E-02	NA	4.50E+05
Americium-243 (²⁴³Am)	Am/Cm / minimum detectable activity	<2.25E-01	NA	6.67E+04

4.0 Conclusions

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

- WAC LIMITS and TARGETS were met for all analyzed chemical and radioactive contaminants for which the detection limits are below the WAC LIMITS and TARGETS.¹
- Measured average concentrations of nitrate, nitrite and total mercury are approximately 24%, 9% and 17% of the WAC LIMITS, respectively.
- Measured average concentrations of Tc-99 and I-129 are approximately 22% and 24% of the WAC LIMITS, respectively.
- All other radionuclide average concentrations are at 3% or less of the WAC LIMITS and TARGETS.

5.0 References

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