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Analysis of Tank 38H (HTF-38-23-19, -20) and Tank 43H (HTF-43-23-21, -22) Samples for Support of the Enrichment Control and Corrosion Control Programs

C. L. Crawford

April 2023

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EXECUTIVE SUMMARY

SRNL analyzed samples from Tank 38H and Tank 43H to support ECP and CCP. The results indicate the concentrations of most soluble species in the Tank 38H surface sample decreased significantly from the previous surface sample. The Tank 38H sub-surface sample shows changes in concentration for soluble species in the solution with some increasing and some decreasing. The current Tank 38H sub-surface sample contains visible sludge solids similar to the previous sample, i.e., less than 1 %, based on visual appearance. The significant differences in the concentrations of major components between the Tank 38H surface and sub-surface samples indicate significant stratification of solution species between these two locations within the tank. Savannah River Mission Completion personnel indicate that ~ 150,000 gallons of Tank 22 supernate were received in Tank 38H since its last analysis during which time the 2H evaporator was not operated, so the observed stratification is expected.

The Tank 43H surface and sub-surface samples are similar in composition to the previous samples. The similar solution compositions measured in the Tank 43H surface and sub-surface samples indicate a minimal stratification within the tank.

The total uranium and plutonium in the current Tank 38H surface sample remains similar to the previous analysis. The Tank 38H sub-surface sample shows an increase in uranium and plutonium concentrations compared to the previous sample likely because of an increase in sludge solids in the current sample. The total uranium concentration in the two Tank 43H samples is essentially unchanged from previous sample results. The plutonium concentration in the Tank 43H surface sample is similar to the previous sample results while the plutonium in the Tank 43H sub-surface sample increased relative to the previous analysis.

The sum of the major cations versus the sum of the major anions shows a difference of <10% for both samples from Tank 38H and for both samples from Tank 43H providing an indication of good data quality for the non-radioactive analytes in the samples.

The silicon concentrations measured in the Tank 38H sub-surface sample (174 mg/L) increased compared with the previous sample. The Tank 38H surface sample silicon concentration (27.5 mg/L) is one-half of the previous sample results. The Tank 43H surface and sub-surface sample silicon concentrations compared to the previous sample results indicate Si in the concentration range of 60 to 83 mg/L. Thus, these current samples analyzed from Tanks 38H and 43H show overall silicon concentrations ranging from 27.5 to 174 mg/L.

TABLE OF CONTENTS

LIST OF TABLES	vii
LIST OF FIGURES	vii
LIST OF ABBREVIATIONS	viii
1.0 Introduction	1
2.0 Experimental	1
2.1 Technical	1
2.2 Quality Assurance	3
3.0 Results and Discussion	3
4.0 Conclusions	6
5.0 Acknowledgements	6
6.0 References	7

LIST OF TABLES

Table 2-1. Sampling Height and Sample Mass of the Tank 38H and 43H Samples.....	2
Table 3-1. ECP, CCP and other Analytical Data for Tank 38H and Tank 43H Samples. (Averages and %RSD values are of triplicate measurements).....	5

LIST OF FIGURES

Figure 2-1. Samples from Tank 38H and 43H.....	2
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LIST OF ABBREVIATIONS

ACSM	Analytical Characterization and Sample Management
DI	de-ionized
CCP	Corrosion Control Program
ECP	Enrichment Control Program
ICP-ES	inductively coupled plasma emission spectrometry
NM	Nuclear Measurements
%RSD	percent relative standard deviation
S/ICPMS	separation/ inductively coupled plasma mass spectrometry
SRNL	Savannah River National Laboratory
SRMC	Savannah River Mission Completion
TIC	total inorganic carbon
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request

1.0 Introduction

Feed limits have been established for the 2H-Evaporator system to ensure nuclear criticality is not possible and corrosion is minimized.¹ These limits are protected by the Enrichment Control Program (ECP) and the Corrosion Control Program (CCP) that require periodic sampling and analysis to confirm that the waste supernate composition stays within the limits.^{2,3}

Savannah River Mission Completion (SRMC) obtained samples from two different heights within each of the two waste tanks supporting the 2H-Evaporator operations on February 21, 2023 (Tank 38H) and February 22, 2023 (Tank 43H). The Tank 38H (evaporator drop tank) and Tank 43H (evaporator feed/vent tank) samples were received by the Savannah River National Laboratory (SRNL) Shielded Cells on February 21, 2023 (Tank 38H) and February 22, 2023 (Tank 43H). Analysis of these samples provides information necessary for determining compliance with the ECP and CCP. The sample characterization was requested via a Technical Task Request⁴ (TTR) and conducted based on a Task Technical and Quality Assurance Plan (TTQAP).⁵

2.0 Experimental

2.1 Technical

The samples from Tank 38H and 43H were opened in the SRNL Shielded Cells and poured into clear plastic beakers. The beakers were photographed and the masses of the samples determined. Table 2-1 provides the sampling height and mass of each sample. The original planned sample height for the Tank 38H sub-surface sample was 224" from the bottom. However, it was noted by the sampling team that the cord stopped eight inches short of the intended mark, so the actual sample height is shown as 232". Figure 2-1 shows a photograph of the samples in the clear beakers. The surface sample from Tank 38H (HTF-38-23-19) and both the surface and sub-surface samples from Tank 43H (HTF-43-23-21, -22, respectively) were mostly clear and showed no visible undissolved solids when poured into the plastic beakers. The sub-surface sample for Tank 38H (HTF-38-23-20) contained dark solids suspended in the solution. The amount of solids looked similar to the previous Tank 38H sub-surface sample which were estimated at < 1%.

All four samples received the analyses required by the ECP that includes determination of uranium isotopes by uranium separation/inductively coupled plasma-mass spectrometry (S/ICPMS) and determination of plutonium isotopes by radiochemical separation and counting methods. Uranium isotopes were also measured without separation by ICPMS. Additionally, the samples were also submitted for gamma spectroscopy to determine the Cs-137 concentration and inductively coupled plasma-emission spectroscopy (ICP-ES) to determine Na, Al, Si, and other metals. All four samples from both Tank 38H and Tank 43H also received the analyses required by the CCP. The CCP analysis suite includes determination of free hydroxide, and anions by ion chromatography (IC). The total inorganic carbon (TIC) and total organic carbon (TOC) were also determined on the samples to provide a concentration for the carbonate anion and total organic carbon, respectively, present in the samples.

Density measurements were made on well-mixed (unfiltered) aliquots of the samples using calibrated volumetric tubes at ambient cell temperature (21 °C).

For the samples receiving the CCP analysis suite, de-ionized (DI) water dilutions were made in triplicate from a well-mixed (unfiltered) sample and submitted to SRNL Analytical Characterization & Sample Management (AC&SM) for analysis. A blank of the DI water was also prepared along with the samples. The water dilutions were analyzed by ion chromatography, total inorganic carbon, and free hydroxide

methods. Triplicate aliquots of the well-mixed (unfiltered) sample from each sample receiving the ECP analysis suite were prepared for analysis using the warm acid strike method.⁶ A reagent blank and three silicon standard solutions were submitted for analysis with the samples. The samples prepared by warm acid strike were submitted to AC&SM and Nuclear Measurements (NM) for analysis by ICP-ES, uranium isotopics, plutonium isotopics, and gamma spectroscopy.

Table 2-1. Sampling Height and Sample Mass of the Tank 38H and 43H Samples

Sample ID	Sample Type	Sampling Height (inches from bottom)	Sample Mass (g)
HTF-38-23-19	Surface	surface	84.2
HTF-38-23-20	Sub-surface Variable Depth	232"	110.1
HTF-43-23-21	Surface	surface	101.3
HTF-43-23-22	Sub-surface Variable Depth	137"	102.2

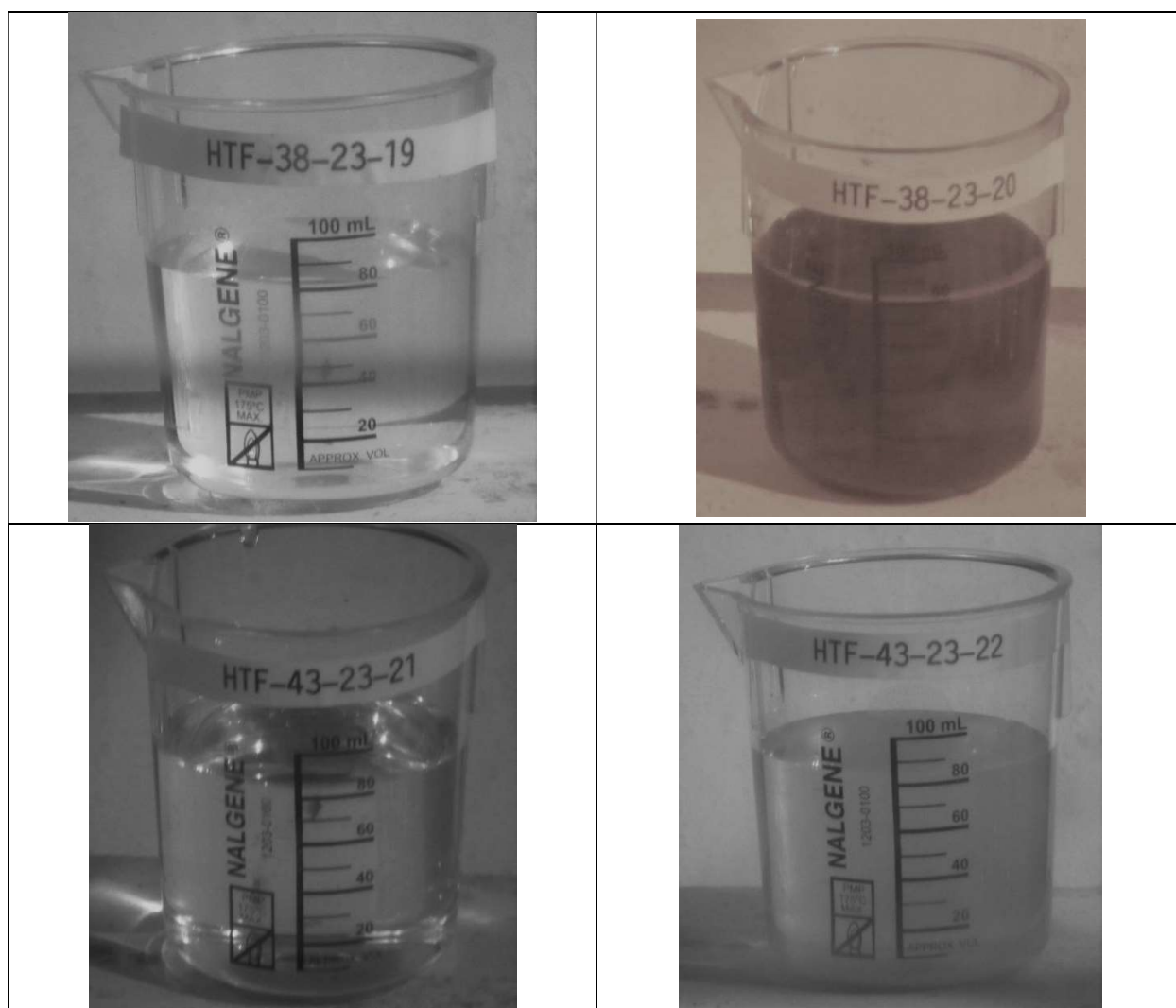


Figure 2-1. Samples from Tank 38H and 43H

2.2 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60.⁷ SRNL documents the extent of the Design Check using the SRNL Technical Report Design Checklist.⁸ This review meets the acceptable criteria to comply with the TTR⁴ requesting this work with a functional classification of Safety Class and per guidance in the TTQAP.⁵ Data are recorded in the electronic laboratory notebook system as notebook/experiment number B9108-00327-31.⁹

3.0 Results and Discussion

Table 3-1 contains the results from the analyses of the 2H-Evaporator system samples. The table shows the average concentrations and the percent relative standard deviation (%RSD) for the triplicate sample preparations unless otherwise noted. Results preceded by “<” indicate the analyte was below the limits of quantification for all three replicate aliquots of the sample. In this case, the less than value in the table results from averaging the three less than values for the replicates. Results with a grey-scale highlight preceded indicate that only two of the triplicate measurements were above the limits of quantitation while one of the replicates analyzed below detection. The average values and %RSD values shown are calculated with the two values that are above the quantitation limit. The %RSD presented in the table only includes the uncertainty associated with sub-sampling/sample preparation in the Shielded Cells and the analytical method. The estimated one sigma percent uncertainty provides an indication of the uncertainty associated with the analytical method as reported by AC&SM and NM. Neither of these measures of uncertainty includes the uncertainty associated with sampling a large waste tank. Previous investigations indicate the uncertainty from taking a small sample from a large waste tank can be significant.^{10,11,12}

The results in Table 3-1 indicate the concentrations of most soluble species in the Tank 38H surface sample decreased significantly from the previous surface sample with the sodium concentration decreasing from 6.71 M in the previous sample to 0.71 M in the current sample.¹³ Similar decreases in both nitrite and nitrate are observed with the current Tank 38H surface nitrite (0.29M) and nitrate (0.12M) being much lower than the previous nitrite (1.9M) and nitrate (0.97M). Free hydroxide concentration in the Tank 38H surface sample (<0.4M) is also significantly lower than the previous Tank 38 surface sample (1.7M). TOC in the Tank 38H surface sample was below detection at <236 mg C/L compared to the previous Tank 38 surface sample (574 mg C/L). The limit of quantification (LOQ) for TOC in these samples is reported as <11.8 mg C/L, which when multiplied by the nominal dilution factor used in the water dilutions (~ 20X) gives the reported <236 mg C/L. The Tank 38H sub-surface sample shows changes in concentration for soluble species in the solution with some increasing and some decreasing. The current Tank 38H sub-surface sample contains sludge solids like the previous sample based on visual appearance. The sodium concentration in the Tank 38H subsurface sample of 8.12 M is higher than the value of 7.77 M observed in the previous sample.¹³ The total uranium in the current Tank 38H surface sample is only about 12% of the total uranium from the previous analysis. The Tank 38H sub-surface sample shows an increase in uranium and plutonium concentrations compared to the previous sample that also contained visible sludge solids.¹³ The weight fraction of U-235 to U-total in the Tank 38H surface sample at 1.24% is higher than the previous sample at 0.80%. The U-235 to U-total weight fraction of 0.61% in the current Tank 38H sub-surface sample is similar to the 0.65% weight fraction in the previous sub-surface sample.¹³ Uranium isotopes are reported from the ICPMS analyses as the initial S/ICPMS results did not yield acceptable values. The S/ICPMS analysis will be repeated to optimize detection of the uranium isotopes and the S/ICPMS data can be provided in a future revision to this technical report. The significant differences in the sodium and other major component concentrations between the Tank 38H surface and sub-surface samples indicate obvious stratification of solution species within the tank where these two different samples were pulled. SRMC

personnel indicate that ~ 150,000 gallons of Tank 22 supernate were received in Tank 38H since its last analysis and the 2H evaporator has not been operated, so the observed stratification is expected.

The Tank 43H surface sample soluble species composition is essentially unchanged from the previous surface sample as indicated by minimal change in the sodium concentration from 6.15 M to 5.87 M in the current sample.¹³ The Tank 43H sub-surface sample is similar to the previous subsurface sample for most highly soluble species analyzed also. The sodium concentration in the current Tank 43H sub-surface sample of 5.81 M resembles the 6.02 M concentration measured in the previous sample. The total uranium concentration in the two Tank 43H samples is similar to the concentrations measured in the previous sample. The plutonium concentration in the Tank 43H surface sample is similar to the previous sample results while the plutonium in the Tank 43H subsurface sample increased relative to the previous analysis. The U-235 weight fraction is similar to the previous analysis in both the Tank 43H surface and sub-surface samples. The composition of the Tank 43H surface and sub-surface samples are similar indicating minimal stratification within the tank.

The results for most species in the table, along with the density measurement results, generally show small %RSD values for the replicate analyses. The sum of the major detectable cations versus the sum of the major detectable anions shows a difference of <10% for the Tank 38H subsurface and Tank 43H samples providing an indication of good data quality for the non-radioactive analytes in the samples.

The Cs-137 results for the Tank 38H subsurface sample and both the Tank 43H samples are similar to the previous samples.¹³ The Tank 38H surface sample Cs-137 result (1.3E+08 dpm/mL) is about 63% lower than the previous Tank 38H surface sample (3.6E+08 dpm/mL) indicating a more dilute Tank 38H surface sample. The Tank 38H surface sample silicon concentration decreased relative to the previous sample.¹³ The silicon concentrations measured in the Tank 38H sub-surface sample increased compared with the previous sample results. The Tank 43H surface and sub-surface samples silicon concentrations are of similar magnitude (~ 60 mg/L to 80 mg/L) compared to the previous sample results.¹³ The standards used for the silicon analysis (50 mg/L silicon in the solution prepared by warm acid strike diluted to final concentrations of 0.5, 1.0, and 2.0 mg/L) were close to the target concentrations with differences from the targeted concentrations of ≤10%. The silicon concentration was below detectable levels in the process blank. Silicon concentrations range from 27.5 to 174 mg/L in all four of the samples analyzed.

Table 3-1. ECP, CCP and other Analytical Data for Tank 38H and Tank 43H Samples. (Averages and %RSD values are of triplicate measurements)

analyte	method	units	est. 1σ	HTF-38-23-19		HTF-38-23-20		HTF-43-23-21		HTF-43-23-22	
				average	%RSD	average	%RSD	average	%RSD	average	%RSD
Sample Type	--	--	--	Surface		Sub-surface		Surface		Sub-surface	
density @ 21°C	grav.	g/mL	5%	1.03	0.9%	1.36	1.3%	1.27	0.1%	1.25	0.2%
U-233	ICPMS	mg/L	20%	<1.27E-03	--	7.00E-03	7.4%	4.02E-03	2.3%	3.75E-03	1.1%
U-234	ICPMS	mg/L	20%	<1.27E-02	--	1.13E-01	7.6%	<1.26E-02	--	<1.24E-02	--
U-235	ICPMS	mg/L	20%	5.80E-02	4.6%	7.64E-01	6.8%	2.70E-01	0.7%	2.59E-01	1.1%
U-236	ICPMS	mg/L	20%	<1.27E-02	--	6.40E-02	6.5%	1.66E-02	2.0%	1.58E-02	2.1%
U-238	ICPMS	mg/L	20%	4.59E+00	1.8%	1.23E+02	6.6%	3.38E+01	1.5%	3.23E+01	0.8%
Total U	calc.	mg/L	--	4.68E+00	1.8%	1.24E+02	6.6%	3.41E+01	1.4%	3.26E+01	0.8%
U-235 / U	calc.	%	--	1.24%	3.5%	0.61%	1.5%	0.79%	1.1%	0.79%	0.3%
Pu-238	PuTTA	mg/L	10%	5.88E-05	17%	2.00E-02	12.3%	3.98E-04	4.0%	2.44E-04	7%
		dpm/mL		7.60E+05		1.51E+04		9.28E+03			
Pu-239 ^a	PuTTA	mg/L	30%	<5.89E-03	--	1.54E-01	3%	<4.90E-03	--	<4.21E-03	--
Pu-239/240	PuTTA	dpm/mL		<8.12E+02		2.13E+04		<6.76E+02		<5.81E+02	
Pu-241	Pu238/41	mg/L	20%	<7.92E-06	--	6.40E-04	10.0%	1.30E-05	5%	9.53E-06	9.1%
		dpm/mL		1.46E+05		2.98E+03		2.18E+03			
Cs-137	gamma scan	dpm/mL	5%	1.31E+08	1.3%	2.88E+08	1.8%	2.81E+08	2.5%	2.78E+08	2.5%
Ba-137m				1.24E+08		2.73E+08		2.66E+08		2.63E+08	
OH ⁻ _{free}	titration	M	10%	<3.99E-01	--	2.47E+00	2.0%	1.72E+00	7.2%	1.74E+00	6.9%
F ⁻	IC	M	10%	<1.05E-02	--	<1.01E-02	--	<1.03E-02	--	<1.01E-02	--
CHO ₂ ⁻	IC	M	10%	<4.44E-03	--	2.09E-02	11.2%	1.96E-02	6.7%	1.80E-02	4.3%
Cl ⁻	IC	M	10%	<5.63E-03	--	8.42E-03	1.2%	6.13E-03	12.1%	5.26E-03	4.1%
NO ₂ ⁻	IC	M	10%	2.91E-01	0.4%	2.06E+00	0.9%	1.79E+00	4.9%	1.83E+00	3.2%
NO ₃ ⁻	IC	M	10%	1.24E-01	5.8%	2.09E+00	2.5%	9.73E-01	8.6%	8.54E-01	4.4%
PO ₄ ³⁻	IC	M	10%	<2.10E-03	--	1.39E-02	0.7%	3.35E-03	20.5%	2.23E-03	6.1%
SO ₄ ²⁻	IC	M	10%	3.30E-03	0.4%	7.18E-02	0.4%	4.66E-02	6.6%	4.56E-02	4.8%
C ₂ O ₄ ²⁻	IC	M	10%	<2.27E-03	--	8.65E-03	8.1%	5.91E-03	5.8%	5.04E-03	8.4%
Br ⁻	IC	M	10%	<1.25E-02	--	<1.20E-02	--	<1.22E-02	--	<1.20E-02	--
CO ₃ ²⁻	TIC	M	14%	2.03E-01	81%	7.73E-01	0.8%	6.07E-01	5.7%	5.86E-01	4.9%
Al	ICP-ES	mg/L	10%	1.41E+02	8.4%	4.28E+03	0.1%	1.73E+03	0.9%	1.81E+03	0.6%
B	ICP-ES	mg/L	10%	2.20E+01	2.1%	1.60E+02	0.4%	1.50E+02	1.0%	1.48E+02	0.7%
Ca	ICP-ES	mg/L	10%	<1.01E+01	--	4.88E+01	4.7%	<1.01E+01	--	<9.88E+00	--
Cr	ICP-ES	mg/L	10%	5.12E+00	4.0%	1.53E+02	0.4%	7.04E+01	1.0%	6.82E+01	0.6%
Fe	ICP-ES	mg/L	10%	<1.27E+01	--	9.65E+01	10.0%	<1.26E+01	--	<1.25E+01	--
K	ICP-ES	mg/L	10%	<7.05E+01	--	6.44E+02	2.2%	3.07E+02	2.0%	3.09E+02	1.9%
Li	ICP-ES	mg/L	10%	<1.08E+02	--	<1.08E+02	--	<1.08E+02	--	<1.06E+02	--
Na	ICP-ES	mg/L	10%	1.78E+04	1.0%	1.87E+05	0.8%	1.35E+05	1.5%	1.34E+05	0.9%
		M		8.12E+00		5.87E+00		5.81E+00			
P	ICP-ES	mg/L	10%	<1.25E+01	--	4.55E+02	0.2%	1.29E+02	1.2%	1.27E+02	0.9%
Si	ICP-ES	mg/L	10%	2.75E+01	4.6%	1.74E+02	8.6%	6.47E+01	1.4%	6.40E+01	1%
Zn	ICP-ES	mg/L	10%	<1.23E+00	--	8.02E+00	0.9%	3.72E+00	1.5%	1.75E+01	0.9%
TOC	TOC	mg C/L	14%	<2.36E+02	--	5.74E+02	2.0%	3.58E+02	7.0%	3.40E+02	4.3%
Anions	sum	M	--	8.32E-01	--	8.46E+00	--	5.86E+00	--	5.75E+00	--
Cations	sum	M	--	7.74E-01	--	8.14E+00	--	5.88E+00	--	5.82E+00	--

calc. = calculation; est.1σ = estimated one sigma percent uncertainty as reported by AD.

^a Pu-239 mass assumes entire Pu-239/240 activity is Pu-239

4.0 Conclusions

The results indicate the concentrations of most soluble species in the Tank 38H surface sample decreased significantly from the previous surface sample. The Tank 38H sub-surface sample shows changes in concentration for soluble species in the solution with some increasing and some decreasing. The current Tank 38H sub-surface sample contains visible sludge solids similar to the previous sample, i.e., less than 1 %, based on visual appearance. The significant differences in the concentrations of major components between the Tank 38H surface and sub-surface samples indicate significant stratification of solution species between these two locations within the tank. Savannah River Mission Completion personnel indicate that ~ 150,000 gallons of Tank 22 supernate were received in Tank 38H since its last analysis during which time the 2H evaporator was not operated, so the observed stratification is expected.

The Tank 43H surface and sub-surface samples are similar in composition to the previous samples. The similar solution compositions measured in the Tank 43H surface and sub-surface samples indicate a minimal stratification within the tank.

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The sum of the major cations versus the sum of the major anions shows a difference of <10% for both samples from Tank 38H and for both samples from Tank 43H providing an indication of good data quality for the non-radioactive analytes in the samples.

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5.0 Acknowledgements

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