Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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Analysis of Tank 38H (HTF-38-19-18, -19) and Tank 43H (HTF-43-19-20, -21) Samples for Support of the Enrichment Control and Corrosion Control Programs

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May 2019

SRNL-STI-2019-00238, Rev. 0

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Prepared for
U.S. Department of Energy

SRNL-STI-2019-00238
Revision 0

Keywords: *2H Evaporator System Supernate Analysis, Radionuclides*

Retention: *Permanent*

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EXECUTIVE SUMMARY

SRNL analyzed samples from Tank 38H and Tank 43H to support ECP and CCP. Overall, the results of the analysis indicate the concentrations of species in the Tank 38H surface sample increased significantly from the previous sample with the sodium concentration increasing from 1.09 M in the previous sample to 3.19 M in the current sample. However, it should be noted that the previous Tank 38H surface sample was very dilute compared with typical Tank 38H samples of the recent past. The Tank 38H sub-surface sample shows a composition fairly similar to the previous sample's composition. Both the Tank 43H surface and sub-surface samples appear slightly more dilute than the previous samples based on the sodium concentrations. For both Tank 38H and Tank 43H, the subsurface samples appear slightly more concentrated for most species than the surface samples. These results indicate some success in increasing the salt solution concentration and reducing stratification in Tank 38H, but some stratification is still evident in both Tank 38H and Tank 43H.

The total uranium concentrations for the two Tank 38H samples differ by ~15% with the surface sample being less concentrated. The total uranium concentrations of the two Tank 43H samples differ by ~30% with the surface sample being less concentrated also. The U-235 weight fraction ranges from 0.65-0.68% in the Tank 38H samples and 0.62-0.63% in the Tank 43H samples. The plutonium results for both of the Tank 38H samples show much higher concentrations of Pu-238 than the previous sample results. However, this higher Pu-238 concentration is more in line with what has typically been measured in Tank 38H samples of the recent past. The Pu-238 concentrations in both Tank 43H samples are lower than the previous sample results by 30-65% and the current surface sample is also ~32% lower in concentration than the current sub-surface sample.

The sum of the major cations versus the sum of the major anions shows a difference of ~11% for the Tank 38H surface sample and ~2% for the Tank 43H surface sample providing an indication of good data quality for the non-radioactive analytes in the surface samples. The silicon concentrations measured in the surface and sub-surface samples from Tank 38H compare reasonably well with each other and both are ~30-60% lower than the previous sample result. The silicon concentrations measured in the Tank 43H surface and sub-surface samples also compare reasonably well with each other and with the previous Tank 43H sample results. The five samples analyzed show silicon concentrations ranging from 70 to 93 mg/L.

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LIST OF ABBREVIATIONS

AD	Analytical Development
DI	De-ionized
CCP	Corrosion Control Program
ECP	Enrichment Control Program
IC	Ion Chromatography
ICP-ES	Inductively Coupled Plasma Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
%RSD	Percent Relative Standard Deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TIC	Total Inorganic Carbon

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 Remediation (SRR) obtained samples from two different heights within each of the two waste tanks supporting the 2H-Evaporator operations on March 8, 2019. The Tank 38H (evaporator drop tank) and Tank 43H (evaporator feed tank) samples were received by the Savannah River National Laboratory (SRNL) Shielded Cells facility on March 8, 2019. Analysis of these samples provides information necessary for determining compliance with the ECP and CCP. The sample characterization was requested via a Technical Task Request[4] and conducted based on a Task Technical and Quality Assurance Plan.[5]

2.0 Experimental Procedure

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. Figure 2-1 shows a photograph of the samples in the clear beakers. The samples from both tanks were mostly clear and showed no visible undissolved solids when poured into the plastic beakers.

All four samples received the analyses required by the ECP that includes determination of uranium isotopes by inductively coupled plasma-mass spectrometry (ICP-MS) and determination of plutonium isotopes by radiochemical separation and counting methods. All four 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. The surface sample from both Tank 38H and Tank 43H received the analyses required by the CCP. The CCP analysis suite includes determination of free hydroxide, and ion chromatography (IC). The total inorganic carbon (TIC) was also determined on the surface samples to provide a concentration for the carbonate anion present in the samples.

Density measurements were made on well-mixed (unfiltered) aliquots of the samples using calibrated volumetric tubes at ambient cell temperature (20 °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 Analytical Development (AD) 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 AD for analysis by ICP-ES, ICP-MS for uranium isotopics, plutonium isotopics, and gamma spectroscopy.

Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Manual E7, Procedure 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. This review meets the acceptable criteria to comply with the TTR classification for this work as safety class. Data are recorded in the electronic laboratory notebook system as notebook/experiment number Y7081-00081-30.

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-38-19-18	Surface	surface	93.6
HTF-38-19-19	Sub-surface	258”	95.7
HTF-43-19-20	Surface	surface	60.4
HTF-43-19-21	Sub-surface	161”	96.9

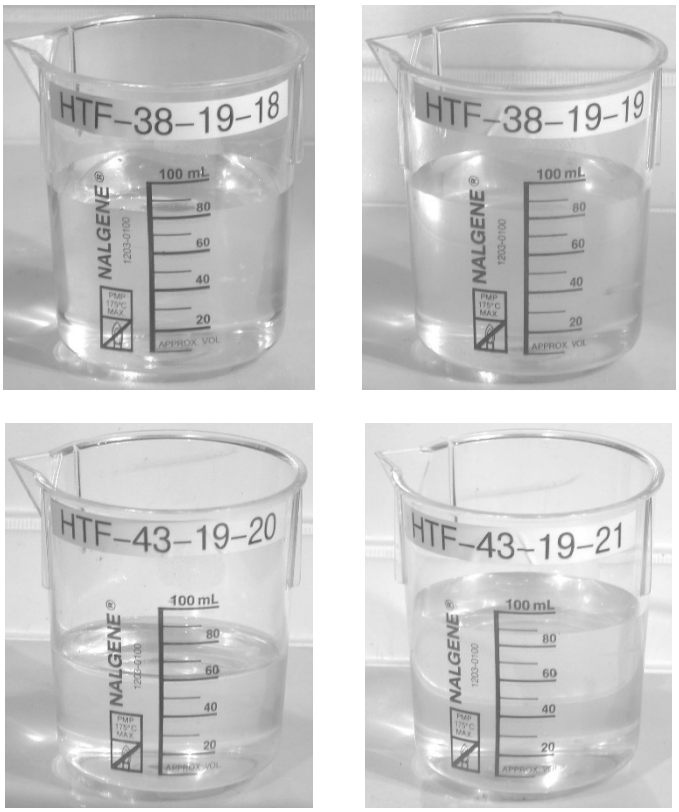

Figure 2-1. Samples from Tank 38H and 43H

3.0 Results and Discussion

The following table contains the results from the analysis of the 2H-Evaporator samples. The table shows the average concentrations and the percent relative standard deviations (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. Results preceded by "≤" indicate that at least one of the replicates for the sample was above the limits of quantification while one or more of the replicates analyzed were below detection. The percent 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 AD. 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.[7,8,9]

Overall, the results in Table 3-1 indicate the concentrations of species in the Tank 38H surface sample (HTF-38-19-18) increased significantly from the previous sample with the sodium concentration increasing from 1.09 M in the previous sample to 3.19 M in the current sample.[10] However, it should be noted that the previous Tank 38H surface sample was very dilute compared with typical Tank 38H samples of the recent past.[11,12] The Tank 38H sub-surface sample (HTF-38-19-19) shows a composition fairly similar to the previous sample's composition. Both the Tank 43H surface and sub-surface samples (HTF-43-19-20, -21) appear ~30-40% more dilute than the previous samples based on the sodium concentrations.[10] For both Tank 38H and Tank 43H, the subsurface samples appear slightly more concentrated for most species than the surface samples (~15-20% based on sodium). These results indicate some success in increasing the salt solution concentration and reducing stratification in Tank 38H, but some stratification is still evident in both Tank 38H and Tank 43H.

The results for the non-radioactive species in the table, along with the density measurement results, generally show small %RSD values for the replicate analyses with the exception of species close to the detection limit (e.g., Fe). The results for sample Tank 38H sub-surface sample (HTF-38-19-19) show slightly elevated %RSD values for all analytes due to one replicate's results being somewhat lower than the other two replicates. The inclusion of the lower concentration replicate in the calculation of the average has the effect of lowering the averages of most analytes for HTF-38-19-19 by ~6-12%. The sum of the major cations versus the sum of the major anions shows a difference of ~11% for the Tank 38H surface sample (HTF-38-19-18) and ~2% for the Tank 43H surface sample (HTF-43-19-20) providing an indication of good data quality for the non-radioactive analytes in the surface samples.

The total uranium concentrations in Table 3-1 for the two Tank 38H samples differ by ~15% with the surface sample (HTF-38-19-18) being less concentrated. The total uranium concentrations of the two Tank 43H samples differ by ~30% with the surface sample (HTF-43-19-20) also being less concentrated. The U-235 weight fraction ranges from 0.65-0.68% in the Tank 38H samples and 0.62-0.63% in the Tank 43H samples. These U-235 weight fraction ranges are similar to previous results on samples from these tanks.[10,11,12]

The plutonium results for both of the Tank 38H samples in Table 3-1 show much higher concentrations of Pu-238 than the previous sample results. The Pu-238 concentration in the Tank 38H surface sample is almost an order of magnitude higher than the previous sample.[10] However, this higher Pu-238 concentration is more in line with what has typically been measured in Tank

38H samples of the recent past.[11,12] The Pu-238 concentration in the Tank 38H sub-surface sample is about 2 times higher than measured in the Tank 38H surface sample. The Pu-238 concentrations in both Tank 43H samples are lower than the previous sample results by 30-65% and the current surface sample is also ~32% lower in concentration than the current sub-surface sample.[10] The Pu-239/240 and Pu-241 are difficult to interpret since the results are close to the detection limits and many past results were below detection for both tanks. However, it's reasonable to assume these align with the Pu-238 results.

The Cs-137 results for the Tank 38H samples show similar concentrations between the current surface and sub-surface samples. The Tank 38H surface sample (HTF-38-19-18) is ~3 times more concentrated in Cs-137 than the previous Tank 38H surface sample while the sub-surface sample Cs-137 concentration is similar to the previous sub-surface sample result.[10] The Tank 43H surface sample (HTF-43-19-20) shows a Cs-137 concentration ~20% lower than the sub-surface sample (HTF-43-19-21) and both samples show Cs-137 concentrations 20-30% lower than the previous Tank 43H samples.[10]

The silicon concentrations measured in the surface and sub-surface samples from Tank 38H compare reasonably well with each other and both are ~30-60% lower than the previous sample result.[10] The silicon concentrations measured in the Tank 43H surface and sub-surface samples also compare reasonably well (~15%) and both compare reasonably well (~15-25%) with the previous Tank 43H sample results.[10] 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 all close to the target concentrations with differences from the targeted concentrations of 6-11%. The silicon concentration was below detectable levels in the process blank. The five samples analyzed show silicon concentrations ranging from 70 to 93 mg/L.

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-38-19-18		HTF-38-19-19		HTF-43-19-20		HTF-43-19-21	
				average	RSD	average	RSD	average	RSD	average	RSD
Sample Type	--	--	--	Surface		Sub-surface		Surface		Sub-surface	
density @ 20°C	grav.	g/mL	5%	1.15	0.6%	1.18	0.2%	1.18	1.0%	1.23	0.4%
U-233	ICP-MS	mg/L	10%	<1.00E-02	--	<1.01E-02	--	<1.01E-02	--	<1.01E-02	--
U-234	ICP-MS	mg/L	10%	<1.00E-02	--	<1.01E-02	--	<1.01E-02	--	<1.01E-02	--
U-235	ICP-MS	mg/L	10%	1.06E-01	0.5%	1.20E-01	11%	1.08E-01	1.3%	1.40E-01	0.2%
U-236	ICP-MS	mg/L	10%	<1.00E-02	--	<1.01E-02	--	<1.01E-02	--	<1.01E-02	--
U-238	ICP-MS	mg/L	10%	1.56E+01	0.7%	1.81E+01	12%	1.71E+01	1.4%	2.23E+01	0.2%
Total U	calc.	mg/L	--	1.57E+01	0.7%	1.83E+01	12%	1.72E+01	1.4%	2.25E+01	0.2%
U-235 / U	calc.	%	--	0.68	0.5%	0.65	0.6%	0.63	1.6%	0.62	0.4%
Pu-238	PuTTA	mg/L	10%	2.07E-04	3.6%	3.99E-04	19%	2.59E-04[b]	4.7%	3.82E-04	18%
		dpm/mL		7.89E+03		1.52E+04		9.84E+03[b]		1.45E+04	
Pu-239 [a]	PuTTA	mg/L	30%	≤1.67E-03	--	3.09E-03	43%	4.21E-03	24%	1.81E-02	16%
Pu-239/240	PuTTA	dpm/mL		≤2.31E+02		4.26E+02		5.81E+02		2.50E+03	
Pu-241	Pu238/41	mg/L	25%	<4.09E-06	--	1.03E-05	26%	≤4.70E-06	--	1.87E-05	22%
		dpm/mL		<9.35E+02		2.36E+03		≤1.07E+03		4.27E+03	
Cs-137	gamma scan	dpm/mL	5%	1.46E+08	1.2%	1.49E+08	9.1%	1.57E+08	0.8%	1.98E+08	2.2%
Ba-137m				1.38E+08		1.41E+08		1.48E+08		1.87E+08	
OH^-_{free}	titration	M	10%	1.18E+00	0.9%	--	--	1.32E+00	2.7%	--	--
F^-	IC	M	10%	<1.01E-02	--	--	--	<1.03E-02	--	--	--
CHO_2^-	IC	M	10%	1.49E-02	0.6%	--	--	1.64E-02	0.3%	--	--
Cl^-	IC	M	10%	<5.42E-03	--	--	--	<5.51E-03	--	--	--
NO_2^-	IC	M	10%	1.02E+00	0.6%	--	--	1.11E+00	0.2%	--	--
NO_3^-	IC	M	10%	4.91E-01	1.0%	--	--	5.39E-01	0.3%	--	--
PO_4^{3-}	IC	M	10%	<2.03E-03	--	--	--	<2.06E-03	--	--	--
SO_4^{2-}	IC	M	10%	2.91E-02	1.0%	--	--	3.00E-02	0.2%	--	--
$C_2O_4^{2-}$	IC	M	10%	7.91E-03	0.8%	--	--	6.49E-03	1.8%	--	--
Br^-	IC	M	10%	<2.41E-03	--	--	--	<2.44E-03	--	--	--
CO_3^{2-}	TIC	M	10%	3.66E-01	0.8%	--	--	3.95E-01	1.2%	--	--
Al	ICP-ES	mg/L	10%	9.51E+02	0.3%	1.17E+03	12%	1.17E+03	0.4%	1.43E+03	0.1%
B	ICP-ES	mg/L	10%	7.65E+01	0.3%	8.43E+01	11%	9.02E+01	0.5%	1.21E+02	0.8%
Ca	ICP-ES	mg/L	10%	<1.30E+01	--	<1.32E+01	--	<1.32E+01	--	<1.33E+01	--
Cr	ICP-ES	mg/L	10%	4.17E+01	0.7%	4.85E+01	13%	5.11E+01	0.9%	6.77E+01	0.8%
Fe	ICP-ES	mg/L	10%	4.05E+00	1.5%	5.08E+00	26%	4.65E+00	28%	5.79E+00	25%
K	ICP-ES	mg/L	10%	1.82E+02	2.4%	2.22E+02	13%	2.22E+02	1.1%	2.64E+02	1.0%
Li	ICP-ES	mg/L	10%	3.92E+01	0.7%	4.34E+01	12%	4.74E+01	0.5%	5.62E+01	0.4%
Na	ICP-ES	mg/L	10%	7.33E+04	0.1%	8.57E+04	11%	9.13E+04	1.1%	1.12E+05	0.4%
		M		3.19E+00		3.73E+00		3.97E+00		4.89E+00	
P	ICP-ES	mg/L	10%	<7.43E+01	--	8.79E+01	10%	9.40E+01	3.7%	1.29E+02	1.5%
Si	ICP-ES	mg/L	10%	7.02E+01	0.3%	7.16E+01	14%	9.29E+01	1.2%	8.01E+01	1.1%
Zn	ICP-ES	mg/L	10%	<3.76E+00	--	<2.78E+00	--	<2.79E+00	--	<3.78E+00	--
Anions	sum	M	--	3.55E+00	--	--	--	3.89E+00	--	--	--
Cations	sum	M	--	3.19E+00	--	3.74E+00	--	3.98E+00	--	4.90E+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 [b] Average of only two values since the third was below detection

4.0 Conclusions

Overall, the results of the analysis indicate the concentrations of species in the Tank 38H surface sample increased significantly from the previous sample with the sodium concentration increasing from 1.09 M in the previous sample to 3.19 M in the current sample. However, it should be noted that the previous Tank 38H surface sample was very dilute compared with typical Tank 38H samples of the recent past. The Tank 38H sub-surface sample shows a composition fairly similar to the previous sample's composition. Both the Tank 43H surface and sub-surface samples appear slightly more dilute than the previous samples based on the sodium concentrations. For both Tank 38H and Tank 43H, the subsurface samples appear slightly more concentrated for most species than the surface samples. These results indicate some success in increasing the salt solution concentration and reducing stratification in Tank 38H, but some stratification is still evident in both Tank 38H and Tank 43H.

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5.0 Acknowledgements

The contributions of Rita Sullivan, in preparing the samples, and those of Amy Ekechukwu, Mark Jones, John Young, and Tom White, for providing analytical services, are appreciated and acknowledged.

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