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-38-19-77, -78) and Tank 43H (HTF-43-19-79, -80) Samples for Support of the Enrichment Control and Corrosion Control Programs

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September 2019

SRNL-STI-2019-00532, Rev. 0

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Printed in the United States of America

Prepared for
U.S. Department of Energy

SRNL-STI-2019-00532
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 indicate the concentrations of most species in the Tank 38H surface and sub-surface samples increased significantly from the previous sample results. The Tank 38H sub-surface sample is more concentrated than the surface sample indicating stratification within the tank. Both the Tank 43H surface and sub-surface samples are similar in composition to the previous samples for most species analyzed. The Tank 43H sub-surface sample remains slightly more concentrated than the surface sample indicating a small amount of stratification remains in the tank.

The total uranium concentrations for the two Tank 38H samples differ significantly due to the presence of sludge solids in the sub-surface sample that markedly increased the uranium concentration. Both the Tank 38H surface and sub-surface samples have higher uranium concentrations than the previous samples from the tank. The weight percent water insoluble solids on the Tank 38H sub-surface sample show the sample contained 0.30 wt% insoluble solids, well below the 1 wt% limit. The total uranium concentrations of the two Tank 43H samples differ by <10% and are similar to the previous sample results.

The sum of the major cations versus the sum of the major anions shows a difference of ~1% for the both the Tank 38H and Tank 43H surface samples providing an indication of good data quality for the non-radioactive analytes in the surface samples. The silicon concentrations measured in the Tank 38H surface and sub-surface samples are higher than the previous sample results. Due to the presence of sludge solids, the Tank 38H sub-surface sample show a silicon concentration an order of magnitude higher than the previous result. The Tank 43H surface and sub-surface samples have similar silicon concentrations and are similar to the previous Tank 43H samples. The silicon concentration was below detectable levels in the process blank. The five samples analyzed show silicon concentrations ranging from 88 to 710 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 July 27, 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 July 29, 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 surface samples from both tanks and the sub-surface sample from Tank 43H were mostly clear and showed no visible undissolved solids when poured into the plastic beakers. The sub-surface sample from Tank 38H contained dark solids suspended in the solution. The dark solids in the Tank 38H sub-surface sample settled overnight forming a thin layer on the bottom of the vessel.

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 (27 °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.

The weight percent water insoluble solids were determined on the Tank 38H sub-surface sample by filtering a known weight of the sample through a weighed 0.45 μm Nylon filter disk, washing the solids with de-ionized water (3 x 50 mL) to remove water soluble solids, drying any solids on the filter disk at ~105 °C for a minimum of 2 hours, and then reweighing the filter disk.

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 acceptance 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 Y7081-00081-33.

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-38-19-77	Surface	surface	97.8
HTF-38-19-78	Sub-surface	248”	104.8
HTF-43-19-79	Surface	surface	93.8
HTF-43-19-80	Sub-surface	161”	95.7

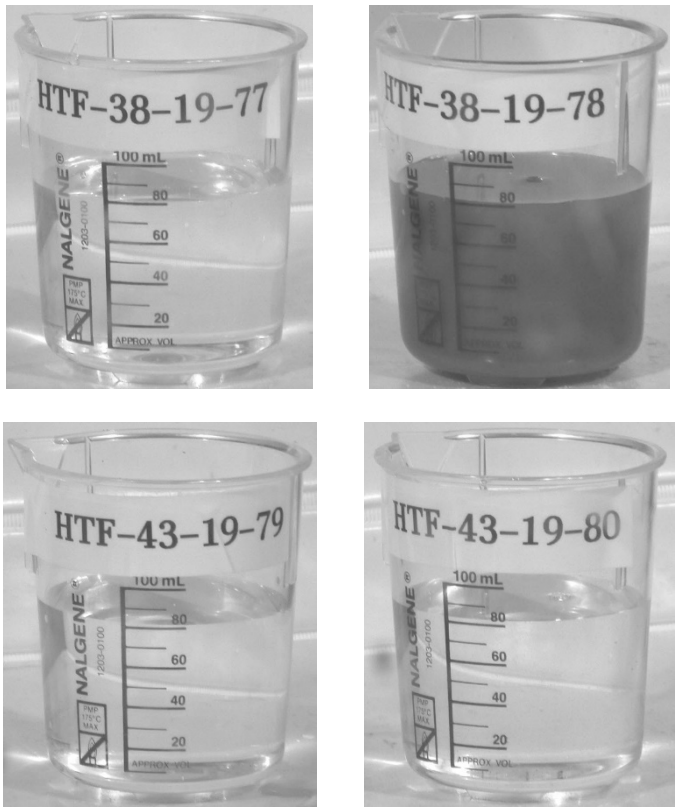

Figure 2-1. Samples from Tank 38H and 43H

3.0 Results and Discussion

The following tables contain the results from the analysis of the 2H-Evaporator samples. The tables show 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 tables 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 most species in the Tank 38H surface sample increased significantly from the previous surface sample with the sodium concentration increasing from 3.19 M in the previous sample to 5.71 M in the current sample.[10] The Tank 38H sub-surface sample also shows a marked increase in concentration for most species measured. The sodium concentration in the Tank 38H sub-surface sample increased from 3.73 M in the previous sample to 7.67 M in the current sample.[10] The Tank 38H sub-surface sample also shows large increases in concentration of uranium and plutonium likely due to the presence of sludge solids in the sample. The weight percent water insoluble solids on the Tank 38H sub-surface sample was measured and the results in Table 3-2 show the sample contained 0.30 wt% insoluble solids, well below the 1 wt% limit.

Both the Tank 43H surface and sub-surface samples are similar in composition to the previous samples for most species analyzed.[10] Most analytes for both samples are within 10-15% of the previous sample compositions. The Tank 43H sub-surface sample remains ~10-15% more concentrated than the surface sample indicating a small amount of stratification remains in the tank.

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, Pu-239, Pu-241). The sum of the major cations versus the sum of the major anions shows a difference of ~1% for the Tank 38H surface sample and ~1% for the Tank 43H surface sample 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 significantly due to the presence of sludge solids in the sub-surface sample that markedly increased the uranium concentration. Both the Tank 38H surface and sub-surface samples have higher uranium concentrations than the previous samples from the tank.[10] The total uranium concentrations of the two Tank 43H samples differ by <10% and are similar to the previous sample results. The U-235 weight fraction ranges from 0.59-0.60% in the four 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 higher concentrations of Pu-238 than the previous sample results.[10] The Pu-238 concentration in the Tank 38H sun-

surface sample is more than two orders of magnitude higher than the previous sample due to the presence of sludge solids in the sample.[10] The Pu-238 concentrations in both Tank 43H samples are similar to previous sample results.[10] The Pu-238 concentration in the Tank 43H sub-surface sample is ~20% higher than in the surface sample.

The Cs-137 results for the Tank 38H samples show similar concentrations between the current surface and sub-surface samples (~12% difference). Both the Tank 38H surface and sub-surface sample are ~2 times more concentrated in Cs-137 than the previous sample result.[10] The Tank 43H surface and sub-surface samples have similar Cs-137 concentrations and are similar to the previous Tank 43H samples.[10]

The silicon concentrations measured in the Tank 38H surface and sub-surface samples are higher than the previous sample results.[10] Due to the presence of sludge solids, the Tank 38H sub-surface sample shows a silicon concentration an order of magnitude higher than the previous result. The Tank 43H surface and sub-surface samples have similar silicon concentrations and are similar to the previous Tank 43H samples.[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 7-9%. The silicon concentration was below detectable levels in the process blank. The five samples analyzed show silicon concentrations ranging from 88 to 710 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-38-19-77		HTF-38-19-78		HTF-43-19-79		HTF-43-19-80	
				average	RSD	average	RSD	average	RSD	average	RSD
Sample Type	--	--	--	Surface		Sub-surface		Surface		Sub-surface	
density @ 27°C	grav.	g/mL	5%	1.23	0.6%	1.31	0.2%	1.16	0.6%	1.18	0.2%
U-233	ICP-MS	mg/L	10%	<1.03E-02	--	7.28E-02	5.4%	<1.01E-02	--	<1.01E-02	--
U-234	ICP-MS	mg/L	10%	<1.03E-02	--	3.89E-01	6.2%	<1.01E-02	--	<1.01E-02	--
U-235	ICP-MS	mg/L	10%	1.51E-01	1.8%	4.97E+00	6.5%	1.04E-01	0.5%	1.14E-01	1.0%
U-236	ICP-MS	mg/L	10%	<1.03E-02	--	3.63E-01	5.3%	<1.01E-02	--	<1.01E-02	--
U-238	ICP-MS	mg/L	10%	2.48E+01	1.8%	8.31E+02	5.5%	1.75E+01	0.5%	1.88E+01	0.7%
Total U	calc.	mg/L	--	2.49E+01	1.8%	8.37E+02	5.5%	1.76E+01	0.4%	1.89E+01	0.7%
U-235 / U	calc.	%	--	0.60%	0.3%	0.59%	1.2%	0.59%	0.7%	0.60%	0.3%
Pu-238	PuTTA	mg/L	10%	3.50E-04	4.0%	5.56E-02	2.0%	2.40E-04	7.1%	3.14E-04	9.3%
		dpm/mL		1.33E+04		2.11E+06		9.13E+03		1.19E+04	
Pu-239 [a]	PuTTA	mg/L	25%	3.87E-03	40%	3.20E-01	4.6%	5.13E-03	26%	6.63E-03	13%
Pu-239/240	PuTTA	dpm/mL		5.33E+02		4.41E+04		7.08E+02		9.15E+02	
Pu-241	Pu238/41	mg/L	20%	1.07E-05	6.3%	1.29E-03	2.9%	7.63E-06	19%	1.07E-05	7.2%
		dpm/mL		2.44E+03		2.95E+05		1.74E+03		2.45E+03	
Cs-137	gamma scan	dpm/mL	5%	2.16E+08	1.3%	2.42E+08	1.7%	1.55E+08	4.2%	1.66E+08	6.3%
Ba-137m				2.04E+08		2.29E+08		1.47E+08		1.57E+08	
OH^-_{free}	titration	M	10%	1.89E+00	0.6%	--	--	1.34E+00	0.8%	--	--
F^-	IC	M	10%	<1.10E-02	--	--	--	<1.06E-02	--	--	--
CHO_2^-	IC	M	10%	2.31E-02	0.6%	--	--	1.67E-02	0.7%	--	--
Cl^-	IC	M	10%	<5.90E-03	--	--	--	<5.66E-03	--	--	--
NO_2^-	IC	M	10%	1.70E+00	0.5%	--	--	1.21E+00	1.4%	--	--
NO_3^-	IC	M	10%	8.37E-01	0.2%	--	--	6.11E-01	2.8%	--	--
PO_4^{3-}	IC	M	10%	<2.20E-03	--	--	--	<2.11E-03	--	--	--
SO_4^{2-}	IC	M	10%	4.34E-02	0.4%	--	--	3.07E-02	0.8%	--	--
$C_2O_4^{2-}$	IC	M	10%	9.40E-03	0.6%	--	--	6.97E-03	0.8%	--	--
Br^-	IC	M	10%	<1.31E-02	--	--	--	<1.25E-02	--	--	--
CO_3^{2-}	TIC	M	10%	5.16E-01	1.8%	--	--	3.66E-01	0.4%	--	--
Al	ICP-ES	mg/L	10%	1.58E+03	0.4%	3.42E+03	0.8%	1.15E+03	0.8%	1.29E+03	1.1%
B	ICP-ES	mg/L	10%	1.23E+02	0.2%	1.50E+02	1.3%	8.92E+01	0.8%	9.87E+01	0.9%
Ca	ICP-ES	mg/L	10%	<1.08E+01	--	5.70E+01	8.4%	<1.06E+01	--	≤2.38E+01	--
Cr	ICP-ES	mg/L	10%	7.18E+01	0.5%	1.42E+02	1.4%	5.27E+01	1.0%	6.06E+01	0.3%
Fe	ICP-ES	mg/L	10%	8.70E+00	58%	1.58E+02	8.8%	5.85E+00	63%	5.62E+00	23%
K	ICP-ES	mg/L	10%	3.10E+02	1.0%	5.11E+02	0.6%	2.19E+02	2.9%	2.47E+02	2.6%
Li	ICP-ES	mg/L	10%	6.88E+01	0.6%	5.98E+01	1.2%	4.84E+01	0.5%	5.18E+01	0.9%
Na	ICP-ES	mg/L	10%	1.31E+05	0.5%	1.76E+05	0.4%	9.39E+04	2.1%	1.06E+05	1.0%
		M		5.71E+00		7.67E+00		4.08E+00		4.63E+00	
P	ICP-ES	mg/L	10%	1.21E+02	2.5%	3.34E+02	0.6%	8.57E+01	2.9%	1.00E+02	5.1%
Si	ICP-ES	mg/L	10%	1.20E+02	1.5%	7.10E+02	7.5%	8.82E+01	0.9%	8.76E+01	3.7%
Zn	ICP-ES	mg/L	10%	<3.92E+00	--	<6.96E+00	--	<3.85E+00	--	<3.84E+00	--
Anions	sum	M	--	5.65E+00	--	--	--	4.03E+00	--	--	--
Cations	sum	M	--	5.72E+00	--	7.68E+00	--	4.09E+00	--	4.64E+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

Table 3-2. Weight Percent Insoluble Measurement Results for Tank 38H Sub-Surface Sample HTF-38-19-78.

Tank	Sample ID	Wt% Insoluble Solids
Tank 38H	HTF-38-19-78	0.30 wt%

4.0 Conclusions

Overall, the results indicate the concentrations of most species in the Tank 38H surface and sub-surface samples increased significantly from the previous sample results. The Tank 38H sub-surface sample is more concentrated than the surface sample indicating stratification within the tank. Both the Tank 43H surface and sub-surface samples are similar in composition to the previous samples for most species analyzed. The Tank 43H sub-surface sample remains slightly more concentrated than the surface sample indicating a small amount of stratification remains in the tank.

The total uranium concentrations for the two Tank 38H samples differ significantly due to the presence of sludge solids in the sub-surface sample that markedly increased the uranium concentration. Both the Tank 38H surface and sub-surface samples have higher uranium concentrations than the previous samples from the tank. The weight percent water insoluble solids on the Tank 38H sub-surface sample show the sample contained 0.30 wt% insoluble solids, well below the 1 wt% limit. The total uranium concentrations of the two Tank 43H samples differ by <10% and are similar to the previous sample results.

The sum of the major cations versus the sum of the major anions shows a difference of ~1% for the both the Tank 38H and Tank 43H surface samples providing an indication of good data quality for the non-radioactive analytes in the surface samples. The silicon concentrations measured in the Tank 38H surface and sub-surface samples are higher than the previous sample results. Due to the presence of sludge solids, the Tank 38H sub-surface sample show a silicon concentration an order of magnitude higher than the previous result. The Tank 43H surface and sub-surface samples have similar silicon concentrations and are similar to the previous Tank 43H samples. The silicon concentration was below detectable levels in the process blank. The five samples analyzed show silicon concentrations ranging from 88 to 710 mg/L.

5.0 Acknowledgements

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

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