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Analysis of Tank 38H (HTF-38-20-7, -8) and Tank 43H (HTF-43-20-9, -10) Samples for Support of the Enrichment Control and Corrosion Control Programs

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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 species in the Tank 38H surface sample decreased significantly from the previous surface sample. The Tank 38H sub-surface sample shows only small changes in concentration for most major species in the solution from the previous sample. The large differences in the concentrations of major components between the Tank 38H surface and sub-surface samples indicate significant stratification of solution species within the tank. The Tank 38H sub-surface sample contained a small amount of sludge solids (<1 wt%).

The Tank 43H surface sample is slightly more concentrated than the previous sample while the sub-surface sample is significantly more concentrated than the previous sub-surface sample. The Tank 43H sub-surface sample exhibits a composition significantly more concentrated than the surface sample indicating stratification within 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. Additionally, while the Tank 38H surface sample total uranium concentration is similar to the previous surface samples concentration, the Tank 38H sub-surface sample total uranium concentration decreased significantly from the previous sample due to there being less sludge solids in the current sample. The total uranium concentrations of the two Tank 43H samples differ by ~13% 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 ~1-3% 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 lower than the previous sample results. Due to the presence of sludge solids, the Tank 38H sub-surface sample shows a large %RSD for the silicon data likely due to difficulties reproducibly sub-sampling the solution and small amount of sludge solids present. The Tank 43H surface sample has a similar silicon concentration to the previous Tank 43H surface sample while the Tank 43H sub-surface sample silicon concentration decreased from the previous sample results. The silicon concentration was below detectable levels in the process blank. The five samples analyzed show silicon concentrations ranging from 44 to 94 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
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 Remediation (SRR) obtained samples from two different heights within each of the two waste tanks supporting the 2H-Evaporator operations on January 14, 2020. 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 January 14, 2020. 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] (TTR) and conducted based on a Task Technical and Quality Assurance Plan (TTQAP).[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. After settling overnight, the Tank 38H sub-surface sample bottle contained a clear solution with a small amount of dark solids sitting on the bottom. Based on experience with past samples, these solids were much less than 1 wt% insoluble solids.

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 (18 °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.[7] SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist.[8] This review meets the acceptable criteria to comply with the TTR[4] 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 Y7081-00081-36.[9]

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-38-20-7	Surface	surface	93.1
HTF-38-20-8	Sub-surface	224”	107.6
HTF-43-20-9	Surface	surface	98.4
HTF-43-20-10	Sub-surface	137”	105.1

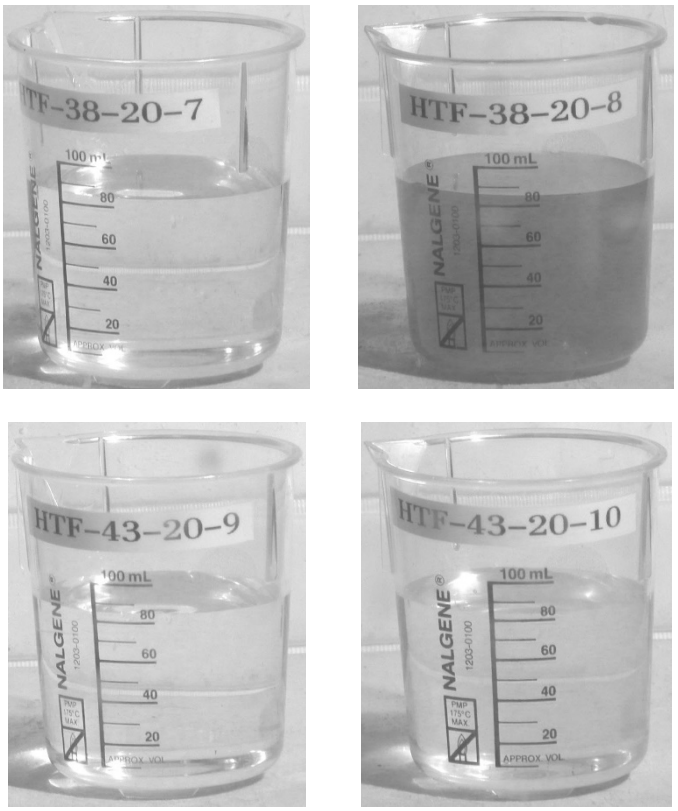

Figure 2-1. Samples from Tank 38H and 43H

3.0 Results and Discussion

Table 3-1 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. In this case, the less than value in the table results from averaging the three less than values for the replicates. 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 less than or equal to value in the table results from averaging all values for all three replicates. 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.[10,11,12]

The results in Table 3-1 indicate the concentrations of most species in the Tank 38H surface sample decreased significantly from the previous surface sample with the sodium concentration decreasing from 5.71 M in the previous sample to 3.08 M in the current sample.[13] The Tank 38H sub-surface sample shows only small changes in concentration for most major species in the solution. The sodium concentration in the Tank 38H sub-surface sample decreased only slightly from 7.67 M in the previous sample to 7.35 M in the current sample.[13] The large differences in the sodium and other major component concentrations between the Tank 38H surface and sub-surface samples indicate significant stratification of solution species within the tank.

The Tank 38H sub-surface sample shows large decreases in the concentrations of uranium and plutonium likely due to the presence of more sludge solids in the previous sample. The current Tank 38H sub-surface sample also contains a small amount of sludge solids (<1 wt%) that likely resulted in the large %RSD for species typically found in the sludge (Fe, Si, U, Pu).

The Tank 43H surface sample is slightly more concentrated than the previous sample as indicated by the increase in the sodium concentration from 4.08 M to 4.64 M.[13] The Tank 43H sub-surface sample exhibits a composition significantly more concentrated than the surface sample indicating some stratification within the tank. The Tank 43H sub-surface sample is also significantly more concentrated than the previous sub-surface sample with the sodium concentration increasing from 4.63 M to 6.96 M.[13]

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) and as mentioned above, sludge elements in the Tank 38H sub-surface sample. The sum of the major cations versus the sum of the major anions shows a difference of ~3% 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. Additionally, while the Tank 38H surface sample total uranium concentration is similar to the previous surface samples concentration, the Tank 38H sub-surface sample total uranium concentration decreased significantly from the previous sample due to there being less sludge solids in the current sample.[13] The total uranium concentrations of the two Tank 43H samples differ by ~13% and are similar to the previous sample results. The U-235 weight fraction ranges from 0.62-0.75% in the four samples. These U-235 weight fraction ranges are generally similar to previous results on samples from these tanks, however the Tank 38H surface sample of weight fraction of 0.75 is higher than typical. [13,14,15]

The results for all of the measured plutonium isotopes in the two Tank 38H samples differ significantly from each other with the sub-surface samples showing higher concentrations. Some of this difference in the Tank 38H samples can be attributed to the presence of sludge solids in the sub-surface sample. Both the Tank 38H surface and sub-surface samples show lower plutonium concentrations for all isotopes than measured in the previous samples.[13] The concentration of all plutonium isotopes measured in both Tank 43H samples increased from previous sample results except for Pu-239/240 and Pu-241 in the surface sample that remained about the same.[13] All plutonium isotope concentrations measured in the Tank 43H sub-surface sample are ~60-70% higher than in the surface sample.

The Cs-137 results for the Tank 38H surface sample decreased from the previous sample while the sub-surface sample remained relatively unchanged.[13] The Tank 38H sub-surface sample has an approximately 72% higher Cs-137 concentration than the surface sample. The Tank 43H surface and sub-surface samples have higher Cs-137 concentrations than the previous Tank 43H samples.[13] In Tank 43H, the sub-surface Cs-137 concentration is ~25% higher than the surface sample.

The silicon concentrations measured in the Tank 38H surface and sub-surface samples are lower than the previous sample results.[13] Due to the presence of sludge solids, the Tank 38H sub-surface sample shows a large %RSD likely due to difficulties reproducibly sub-sampling the solution and small amount of sludge solids present. The Tank 43H surface sample has a similar silicon concentration to the previous Tank 43H surface sample.[13] The Tank 43H sub-surface sample silicon concentration decreased from 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 all close to the target concentrations with differences from the targeted concentrations of 2-8%. The silicon concentration was below detectable levels in the process blank. The four samples analyzed show silicon concentrations ranging from 44 to 94 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-38-20-7		HTF-38-20-8		HTF-43-20-9		HTF-43-20-10	
				average	RSD	average	RSD	average	RSD	average	RSD
Sample Type	--	--	--	Surface		Sub-surface		Surface		Sub-surface	
density @ 18°C	grav.	g/mL	5%	1.14	0.6%	1.32	0.6%	1.20	0.5%	1.29	0.9%
U-233	ICP-MS	mg/L	10%	<1.02E-02	--	<1.02E-02	--	<1.02E-02	--	<1.05E-02	--
U-234	ICP-MS	mg/L	10%	<1.02E-02	--	3.92E-02[b]	47%	<1.02E-02	--	<1.05E-02	--
U-235	ICP-MS	mg/L	10%	1.59E-01	0.4%	2.69E-01	86%	1.67E-01	0.6%	1.33E-01	1.1%
U-236	ICP-MS	mg/L	10%	<1.02E-02	--	2.97E-02[b]	48%	1.07E-02	3.4%	<1.05E-02	--
U-238	ICP-MS	mg/L	10%	2.10E+01	0.4%	4.31E+01	86%	2.40E+01	1.1%	2.14E+01	0.6%
Total U	calc.	mg/L	--	2.12E+01	0.4%	4.34E+01	86%	2.42E+01	1.1%	2.15E+01	0.6%
U-235 / U	calc.	%	--	0.75	0.7%	0.62	0.5%	0.69	0.5%	0.62	1.2%
Pu-238	PuTTA	mg/L	10%	1.61E-04	6.8%	3.32E-03	95%	3.07E-04	3.8%	9.16E-04	5.6%
		dpm/mL		6.13E+03		1.26E+05		1.17E+04		3.48E+04	
Pu-239 [a]	PuTTA	mg/L	30%	2.55E-03[b]	11%	2.90E-02[b]	44%	5.05E-03	26%	1.65E-02	58%
Pu-239/240	PuTTA	dpm/mL		3.52E+02[b]		4.01E+03[b]		6.97E+02		2.28E+03	
Pu-241	Pu238/41	mg/L	20%	4.38E-06	6.8%	8.12E-05	95%	7.99E-06	19%	1.96E-05	3.8%
		dpm/mL		1.00E+03		1.86E+04		1.83E+03		4.48E+03	
Cs-137	gamma scan	dpm/mL	5%	1.41E+08	2.3%	2.43E+08	2.2%	1.87E+08	3.4%	2.33E+08	3.5%
Ba-137m				1.33E+08		2.30E+08		1.77E+08		2.21E+08	
OH^-_{free}	titration	M	10%	9.28E-01[b]	1.1%	--	--	1.56E+00	0.6%	--	--
F^-	IC	M	10%	<1.06E-02	--	--	--	<1.06E-02	--	--	--
CHO_2^-	IC	M	10%	<4.49E-03	--	--	--	<4.47E-03	--	--	--
Cl^-	IC	M	10%	<5.70E-03	--	--	--	<5.68E-03	--	--	--
NO_2^-	IC	M	10%	9.85E-01	0.2%	--	--	1.46E+00	1.4%	--	--
NO_3^-	IC	M	10%	5.27E-01	0.9%	--	--	7.34E-01	1.9%	--	--
PO_4^{3-}	IC	M	10%	≤2.47E-03	--	--	--	3.05E-03[b]	3.6%	--	--
SO_4^{2-}	IC	M	10%	2.45E-02	1.3%	--	--	3.76E-02	1.4%	--	--
$C_2O_4^{2-}$	IC	M	10%	6.10E-03	0.5%	--	--	8.29E-03	0.7%	--	--
Br^-	IC	M	10%	<1.26E-02	--	--	--	<1.26E-02	--	--	--
CO_3^{2-}	TIC	M	10%	3.28E-01	1.8%	--	--	3.99E-01	0.7%	--	--
Al	ICP-ES	mg/L	10%	7.78E+02	1.4%	3.04E+03	2.4%	1.28E+03	2.9%	1.93E+03	0.4%
B	ICP-ES	mg/L	10%	7.52E+01	0.6%	1.54E+02	0.5%	1.14E+02	3.1%	1.54E+02	0.5%
Ca	ICP-ES	mg/L	10%	<8.11E+00	--	<8.16E+00	--	<8.15E+00	--	<8.54E+00	--
Cr	ICP-ES	mg/L	10%	3.47E+01	2.1%	1.44E+02	1.4%	6.27E+01	3.6%	1.10E+02	0.7%
Fe	ICP-ES	mg/L	10%	2.57E+00	20%	1.49E+01	85%	3.70E+00	3.7%	5.44E+00	19%
K	ICP-ES	mg/L	10%	1.68E+02	1.4%	5.39E+02	0.6%	2.59E+02	3.2%	3.93E+02	1.0%
Li	ICP-ES	mg/L	10%	4.11E+01	1.8%	3.73E+01	1.0%	5.77E+01	2.7%	6.46E+01	0.6%
Na	ICP-ES	mg/L	10%	7.09E+04	1.1%	1.69E+05	0.8%	1.07E+05	0.6%	1.60E+05	0.8%
		M		3.08E+00		7.35E+00		4.64E+00		6.96E+00	
P	ICP-ES	mg/L	10%	6.22E+01	2.7%	4.19E+02	0.7%	1.14E+02	8.7%	1.86E+02	1.4%
Si	ICP-ES	mg/L	10%	7.50E+01	4.7%	7.48E+01[b]	44%	9.36E+01	4.7%	4.40E+01	1.4%
Zn	ICP-ES	mg/L	10%	2.59E+00	6.5%	1.50E+01	23%	3.70E+00	6.8%	4.91E+00	3.8%
Anions	sum	M	--	3.19E+00	--	--	--	4.70E+00	--	--	--
Cations	sum	M	--	3.08E+00	--	7.36E+00	--	4.65E+00	--	6.97E+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

The results indicate the concentrations of most species in the Tank 38H surface sample decreased significantly from the previous surface sample. The Tank 38H sub-surface sample shows only small changes in concentration for most major species in the solution from the previous sample. The large differences in the concentrations of major components between the Tank 38H surface and sub-surface samples indicate significant stratification of solution species within the tank. The Tank 38H sub-surface sample contained a small amount of sludge solids (<1 wt%).

The Tank 43H surface sample is slightly more concentrated than the previous sample while the sub-surface sample is significantly more concentrated than the previous sub-surface sample. The Tank 43H sub-surface sample exhibits a composition significantly more concentrated than the surface sample indicating stratification within 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. Additionally, while the Tank 38H surface sample total uranium concentration is similar to the previous surface samples concentration, the Tank 38H sub-surface sample total uranium concentration decreased significantly from the previous sample due to there being less sludge solids in the current sample than in the previous sample. The total uranium concentrations of the two Tank 43H samples differ by ~13% 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 ~1-3% 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 lower than the previous sample results. Due to the presence of sludge solids, the Tank 38H sub-surface sample shows a large %RSD for the silicon data likely due to difficulties reproducibly sub-sampling the solution and small amount of sludge solids present. The Tank 43H surface sample has a similar silicon concentration to the previous Tank 43H surface sample while the Tank 43H sub-surface sample silicon concentration decreased from the previous sample results. The silicon concentration was below detectable levels in the process blank. The five samples analyzed show silicon concentrations ranging from 44 to 94 mg/L.

5.0 Acknowledgements

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