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Analysis of Tank 38H (HTF-38-18-78, -79) and Tank 43H (HTF-43-18-80, -81, -83) 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. Compared with recent samples, the surface and sub-surface samples from Tank 38H show much lower concentrations of most species analyzed except for silicon. The Tank 38H samples show stratification in the tank with the concentration of species in the surface sample being much lower than in the sub-surface sample and both samples being much lower than recent results. The Tank 43H surface and two sub-surface samples generally showed consistent results across the three samples and compare well with recent analyses. The lowest sub-surface sample from Tank 43H contained slightly higher concentrations of uranium and plutonium likely due to the presence of a small amount of dark sludge solids in the sample.

The total uranium in the Tank 38H surface sample was 8.71 mg/L while the sub-surface sample was 17.0 mg/L. The Tank 43H samples contained total uranium concentrations of 28.5 mg/L in the surface sample, 31.4 mg/L in the sub-surface sample from 161 inches and 35.3 mg/L for the sub-surface sample from 90 inches. The U-235 weight fraction ranged from 0.62% to 0.63% for the Tank 38H and Tank 43H samples.

The sum of the major cations versus the sum of the major anions shows a difference of <5% for the three Tank 43H samples and ~8% for the Tank 38H surface sample providing an indication of good data quality for the non-radioactive analytes reported. The silicon concentrations measured in the surface and sub-surface samples from Tank 38H and Tank 43H compare reasonably well with recent analyses. The five samples analyzed show silicon concentrations ranging from 104 to 181 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 October 5, 2018. The Tank 38H (evaporator drop tank) and Tank 43H (evaporator feed tank) samples were received by the Savannah River National Laboratory (SRNL) Shielded Cells on October 8, 2018. Analysis of these samples provides information necessary for determining compliance with the ECP and CCP. An additional sample from just above the solids layer was obtained from Tank 43H to determine whether suspended solids could have contributed to solids buildup in the gravity drain line. 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 were mostly clear and showed no visible undissolved solids when poured into the plastic beakers. The sub-surface sample from Tank 38H was also mostly clear with no solids visible. The Tank 43H sub-surface sample from 161 inches in the tank was cloudy and a light dusting of fine particles settled out of the liquid after sitting overnight. The Tank 43H sub-surface sample from 90 inches in the tank contained significantly more solids than the sample from 161 inches. However, the weight percent insoluble solids were estimated to be significantly less than 1 wt% based on visual observation.

All five 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 and inductively coupled plasma-emission spectroscopy (ICP-ES) to determine Na, Al, Si, and other metals. The surface sample from Tank 38H and all three samples from 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 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 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. Data are recorded in the electronic laboratory notebook system as notebook/experiment number Y7081-00081-27.

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-38-18-78	Surface	surface	84.13
HTF-38-18-79	Sub-surface	270”	101.0
HTF-43-18-80	Surface	surface	100.3
HTF-43-18-81	Sub-surface	161”	102.5
HTF-43-18-83	Sub-surface	90”	101.9

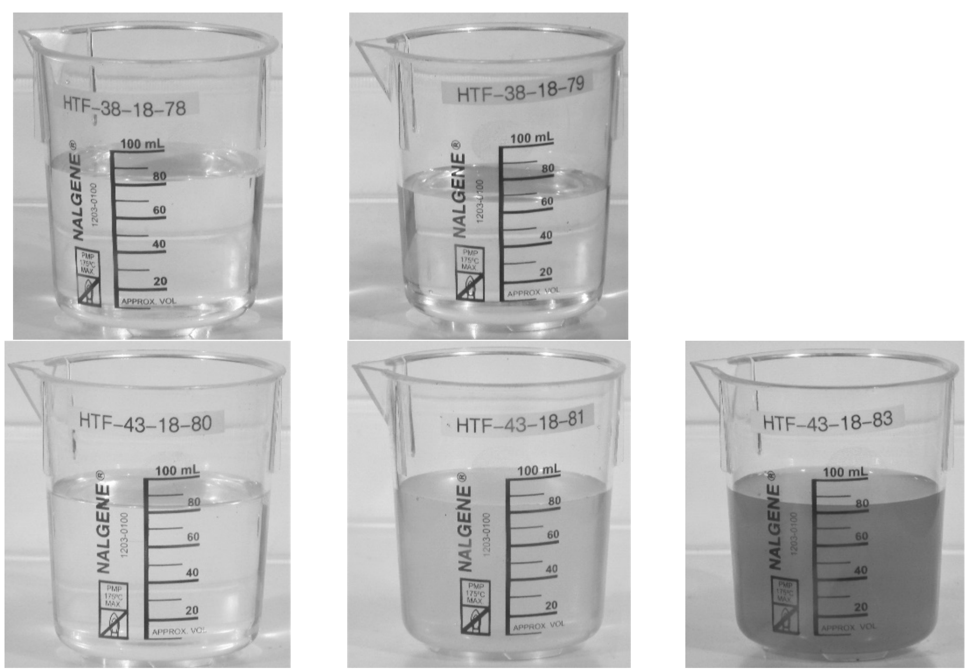

Figure 2-1. Samples from Tank 38H and 43H

3.0 Results and Discussion

The following tables contain the results from the analysis of the Tank 38H and Tank 43H samples. The tables show the average concentration and the percent relative standard deviations (%RSD) for the triplicate sample preparations. 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 %RSD presented in the table only includes the uncertainty associated with sub-sampling and sample preparation in the Shielded Cells. The %RSD does not include tank sampling uncertainty. 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]

The uranium concentrations in Table 3-1 for the two Tank 38H samples differ by a factor of two and both are much lower than concentrations in recent samples. This finding is consistent with the fact that both Tank 38H samples appear more dilute than recent samples as evidenced by the sodium concentrations of 1.09 M for the Tank 38H surface sample and 3.55 M for the Tank 38H sub-surface sample. The most recent samples from Tank 38H showed sodium concentrations of 7.70 M for the surface sample and 7.48 M for the sub-surface sample.[10] In Table 3-2, the three Tank 43H samples show slightly increasing uranium concentrations from the surface sample down to the sample 90 inches from the bottom. The Tank 43H uranium concentrations are similar to the most recent previous analysis of this tank. The total uranium in the Tank 38H surface sample was 8.71 mg/L while the sub-surface sample was 17.0 mg/L. The Tank 43H samples contained total uranium concentrations of 28.5 mg/L in the surface sample, 31.4 mg/L in the sub-surface sample from 161 inches and 35.3 mg/L for the sub-surface sample from 90 inches. The U-235 weight fraction ranged from 0.62% to 0.63% for the Tank 38H and Tank 43H samples.

The plutonium results for the Tank 38H samples in Table 3-1 show much lower concentrations than recent samples results and the surface sample is much lower than the sub-surface sample. The Tank 43H plutonium concentrations in Table 3-2 are consistent with the range of concentrations measured in recent samples. The Tank 43H sub-surface sample from 90 inches shows the highest plutonium concentration likely due to the small amount of solids present in that sample. The Pu-239/240 results were generally below detection except in the Tank 43H sub-surface sample from 90 inches.

The Cs-137 results for the Tank 38H samples show the same trend with the concentration in the surface sample being much lower than the sub-surface sample and both samples being much lower than recent results. The Cs-137 concentrations in the Tank 43H samples show consistency across all three samples. The Tank 43H concentrations are slightly higher than recent results.

The non-radioactive components of the samples such as the metals from the ICP-ES analysis and anions from the IC analysis appear consistent for all five samples. The sum of the major cations versus the sum of the major anions shows a difference of <5% for the three Tank 43H samples and ~8% for the Tank 38H surface sample providing an indication of good data quality for the non-radioactive analytes in the table. The Tank 38H samples show the same trend seen in the radioactive components with the concentration in the surface sample being much lower than the sub-surface sample and both samples being much lower than recent results. The concentrations of metals and anions in the surface and sub-surface samples from Tank 43H show reasonable agreement.

The silicon concentrations measured in the surface and sub-surface samples from Tank 38H compare reasonably well with recent Tank 38H analyses in contrast to most of the other species measured on these samples. The silicon concentrations measured in the Tank 43H surface and sub-surface samples also compare well with recent Tank 43H analyses although slightly higher. The standards used for the silicon analysis (50 mg/L silicon in the solution prepared by warm acid strike 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 8-12%. The silicon concentration was below detectable levels in the process blank. The five samples analyzed show silicon concentrations ranging from 104 to 181 mg/L.

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

analyte	method	units	est. 1σ	HTF-38-18-78 average	HTF-38-18-78 RSD	HTF-38-18-79 average	HTF-38-18-79 RSD
density @ 23°C	grav.	g/mL	5%	1.05	1.3%	1.17	0.5%
U-233	ICP-MS	mg/L	10%	<1.01E-02	--	<1.02E-02	--
U-234	ICP-MS	mg/L	10%	<1.01E-02	--	<1.02E-02	--
U-235	ICP-MS	mg/L	10%	5.51E-02	1.1%	1.07E-01	13%
U-236	ICP-MS	mg/L	10%	<1.01E-02	--	<1.02E-02	--
U-238	ICP-MS	mg/L	10%	8.66E+00	0.6%	1.69E+01	13%
Total U	calc.	mg/L	--	8.71E+00	0.6%	1.70E+01	13%
U-235 / U	calc.	%	--	0.63%	0.6%	0.63%	0.6%
Pu-238	PuTTA	mg/L	10%	2.43E-05	22%	1.62E-04	19%
		dpm/mL		9.25E+02		6.16E+03	
Pu-239 [a]	PuTTA	mg/L	30%	<2.25E-03	--	≤1.16E-03	--
Pu-239/240	PuTTA	dpm/mL		<3.10E+02		≤1.60E+02	
Pu-241	Pu238/41	mg/L	15%	<1.19E-06	--	3.74E-06	40%
		dpm/mL		<2.71E+02		8.55E+02	
Cs-137	gamma scan	dpm/mL	5%	4.56E+07	4.3%	1.51E+08	13%
Ba-137m				4.31E+07		1.43E+08	
OH^-	titration	M	10%	3.11E-01	3.7%	--	--
F^-	IC	M	10%	<1.09E-02	--	--	--
CHO_2^-	IC	M	10%	<4.60E-03	--	--	--
Cl^-	IC	M	10%	<5.84E-03	--	--	--
NO_2^-	IC	M	10%	4.04E-01	1.3%	--	--
Br^-	IC	M	10%	<2.59E-03	--	--	--
NO_3^-	IC	M	10%	1.54E-01	1.3%	--	--
PO_4^{3-}	IC	M	10%	<2.18E-03	--	--	--
SO_4^{2-}	IC	M	10%	9.05E-03	0.1%	--	--
$C_2O_4^{2-}$	IC	M	10%	<2.35E-03	--	--	--
CO_3^{2-}	TIC	M	10%	1.39E-01	1.3%	--	--
Al	ICP-ES	mg/L	10%	2.27E+02	0.5%	1.42E+03	13%
B	ICP-ES	mg/L	10%	4.18E+01	0.4%	8.04E+01	13%
Ca	ICP-ES	mg/L	10%	<4.36E-01	--	<5.79E-01	--
Cr	ICP-ES	mg/L	10%	7.76E+00	6.4%	4.37E+01	12%
Fe	ICP-ES	mg/L	10%	7.14E+00	60%	3.33E+00	18%
K	ICP-ES	mg/L	10%	<2.72E+02	--	1.95E+02	16%
Li	ICP-ES	mg/L	10%	3.90E+01	0.3%	4.86E+01	13%
Na	ICP-ES	mg/L	10%	2.51E+04	0.2%	8.17E+04	13%
		M		1.09E+00		3.55E+00	
P	ICP-ES	mg/L	10%	<2.18E+02	--	7.69E+01	8.9%
Si	ICP-ES	mg/L	10%	1.81E+02	0.3%	1.07E+02	13%
Zn	ICP-ES	mg/L	10%	9.52E-01	37%	<9.65E+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. ECP, CCP, and other Analytical Data for Tank 43H Samples. (Averages and %RSD values are of triplicate measurements)

analyte	method	units	est. 1σ	HTF-43-18-80 average	HTF-43-18-80 RSD	HTF-43-18-81 average	HTF-43-18-81 RSD	HTF-43-18-83 average	HTF-43-18-83 RSD
density @ 23°C	grav.	g/mL	5%	1.27	0.7%	1.30	0.8%	1.29	0.6%
U-233	ICP-MS	mg/L	10%	<1.03E-02	--	<1.03E-02	--	<1.02E-02	--
U-234	ICP-MS	mg/L	10%	<1.03E-02	--	<1.03E-02	--	1.21E-02	2.0%
U-235	ICP-MS	mg/L	10%	1.78E-01	0.3%	1.96E-01	0.2%	2.18E-01	1.6%
U-236	ICP-MS	mg/L	10%	1.10E-02	2.9%	1.26E-02	3.1%	1.55E-02	5.4%
U-238	ICP-MS	mg/L	10%	2.83E+01	1.0%	3.12E+01	0.5%	3.50E+01	0.7%
Total U	calc.	mg/L	--	2.85E+01	1.0%	3.14E+01	0.5%	3.52E+01	0.7%
U-235 / U	calc.	%	--	0.62%	0.7%	0.62%	0.4%	0.62%	0.9%
Pu-238	PuTTA	mg/L	10%	3.71E-04	3.2%	1.10E-03	10%	4.20E-03	2.0%
		dpm/mL		1.41E+04		4.16E+04		1.60E+05	
Pu-239 [a]	PuTTA	mg/L	30%	<1.54E-03	--	<2.76E-03	--	1.12E-02	14%
Pu-239/240	PuTTA	dpm/mL		<2.13E+02		<3.81E+02		1.55E+03	
Pu-241	Pu238/41	mg/L	15%	5.75E-06	50%	9.75E-06	56%	5.70E-05	2.6%
		dpm/mL		1.32E+03		2.23E+03		1.30E+04	
Cs-137	gamma scan	dpm/mL	5%	2.42E+08	1.3%	2.51E+08	2.4%	2.40E+08	1.9%
Ba-137m				2.29E+08		2.37E+08		2.27E+08	
OH^-	titration	M	10%	2.29E+00	2.7%	2.63E+00	8.8%	2.32E+00	3.3%
F^-	IC	M	10%	<1.07E-02	--	<1.12E-02	--	<1.10E-02	--
CHO_2^-	IC	M	10%	2.97E-02	3.6%	3.19E-02	0.6%	3.10E-02	1.3%
Cl^-	IC	M	10%	<5.74E-03	--	<5.98E-03	--	<5.89E-03	--
NO_2^-	IC	M	10%	1.87E+00	3.8%	2.01E+00	1.7%	2.09E+00	11%
Br^-	IC	M	10%	<2.55E-03	--	<2.65E-03	--	<2.61E-03	--
NO_3^-	IC	M	10%	1.03E+00	4.4%	1.13E+00	3.7%	1.16E+00	5.7%
PO_4^{3-}	IC	M	10%	<2.14E-03	--	<2.23E-03	--	<2.20E-03	--
SO_4^{2-}	IC	M	10%	5.11E-02	4.5%	5.74E-02	2.4%	6.08E-02	10%
$C_2O_4^{2-}$	IC	M	10%	4.32E-03	0.3%	4.42E-03	1.5%	4.23E-03	2.4%
CO_3^{2-}	TIC	M	10%	5.29E-01	0.1%	5.65E-01	0.4%	5.50E-01	0.6%
Al	ICP-ES	mg/L	10%	1.91E+03	0.7%	2.16E+03	1.2%	2.12E+03	0.6%
B	ICP-ES	mg/L	10%	1.38E+02	0.5%	1.53E+02	1.3%	1.48E+02	0.9%
Ca	ICP-ES	mg/L	10%	4.46E+00[b]	45%	8.55E+00	44%	1.17E+01	6.9%
Cr	ICP-ES	mg/L	10%	8.12E+01	0.9%	9.36E+01	1.1%	9.18E+01	0.6%
Fe	ICP-ES	mg/L	10%	4.09E+00	7.8%	9.63E+00	12%	2.47E+01	6.8%
K	ICP-ES	mg/L	10%	3.48E+02	1.7%	3.96E+02	1.1%	3.89E+02	2.7%
Li	ICP-ES	mg/L	10%	7.05E+01	0.8%	7.54E+01	1.3%	7.37E+01	0.9%
Na	ICP-ES	mg/L	10%	1.44E+05	1.8%	1.64E+05	0.8%	1.59E+05	0.5%
		M		6.28E+00		7.12E+00		6.92E+00	
P	ICP-ES	mg/L	10%	1.48E+02	2%	1.73E+02	1.1%	1.68E+02	1.5%
Si	ICP-ES	mg/L	10%	1.08E+02	0.3%	1.04E+02	1.4%	1.18E+02	1.2%
Zn	ICP-ES	mg/L	10%	<1.05E+01	--	<4.12E+01	--	<4.54E+01	--

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

4.0 Conclusions

Compared with recent samples, the surface and sub-surface samples from Tank 38H show much lower concentrations of most species analyzed except for silicon. The Tank 38H samples show stratification in the tank with the concentration of species in the surface sample being much lower than in the sub-surface sample and both samples being much lower than recent results. The Tank 43H surface and two sub-surface samples generally showed consistent results across the three samples and compare well with recent analyses. The lowest sub-surface sample from Tank 43H contained slightly higher concentrations of uranium and plutonium likely due to the presence of a small amount of dark sludge solids in the sample.

The total uranium in the Tank 38H surface sample was 8.71 mg/L while the sub-surface sample was 17.0 mg/L. The Tank 43H samples contained total uranium concentrations of 28.5 mg/L in the surface sample, 31.4 mg/L in the sub-surface sample from 161 inches and 35.3 mg/L for the sub-surface sample from 90 inches. The U-235 weight fraction ranged from 0.62% to 0.63% for the Tank 38H and Tank 43H samples.

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5.0 Acknowledgements

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