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Analysis of Tank 35H Samples in Support of Salt Batch Planning

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April 2015

SRNL-STI-2015-00224, Rev. 0

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Prepared for
U.S. Department of Energy

SRNL-STI-2015-00224
Revision 0

Keywords: *Supernate Analysis, Radionuclides*

Retention: *Permanent*

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EXECUTIVE SUMMARY

Savannah River Remediation obtained three samples from different heights within Tank 35H. The samples were analyzed by Savannah River National Laboratory to support Salt Batch planning. The results from the analysis indicate the compositions of the three samples show increasing concentrations for most analytes going from HTF-35-15-17 through HTF-35-15-19 corresponding to successively deeper sampling locations. The data indicate some stratification within the tank. The plutonium and Sr-90 concentrations measured in the filtered samples were slightly lower than in the decanted (unfiltered) samples. The difference in the results for the filtered and unfiltered samples likely lies within the expected uncertainty for the measurement, but may indicate that filtration removed a small amount of suspended material from the samples.

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LIST OF ABBREVIATIONS

AD	Analytical Development
DI	De-ionized
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

1.0 Introduction

Savannah River Remediation (SRR) obtained three samples from different heights within Tank 35H. The samples were received by the Savannah River National Laboratory (SRNL) Shielded Cells on March 9, 2015. The analysis of these samples provides information necessary for salt batch planning. The sample characterization was requested via a Technical Task Request[1] and conducted based on a Task Technical and Quality Assurance Plan.[2]

2.0 Experimental Procedure

The three samples were opened in the SRNL Shielded Cells and poured into clear plastic beakers on March 10, 2015. The beakers were photographed and the mass of the samples determined. Table 2-1 provides the sampling height and mass of each sample. Figure 2-1 shows photographs of the samples. All three samples appeared cloudy but did not contain any visible solids when poured into the clear beakers. However, after sitting undisturbed overnight a fine layer of solids settled to the bottom of each poly bottle containing the samples. The solids were white in sample HTF-35-15-17 and progressively darker in the HTF-35-15-18 and HTF-35-15-19 samples.

Triplicate density measurements were made on decanted (unfiltered) aliquots of the samples using 2 mL volumetric tubes at room temperature (23 °C).

De-ionized (DI) water dilutions were made in triplicate from decanted (unfiltered) liquid from each 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. A special sample preparation was conducted in the Shielded Cells on triplicate, decanted (unfiltered) aliquots of the samples for I-129 analysis. Acid dilutions of filtered liquid from the samples were made in triplicate and submitted to AD for analysis by liquid scintillation for Sr-90, mercury analysis (CV Hg digested), and plutonium isotopics. A blank of the diluting acid (2 M HNO_3) was also prepared along with the samples.

Triplicate aliquots of decanted (unfiltered) liquid from each sample were also prepared for analysis using the warm acid strike method. 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 inductively coupled plasma-emission spectroscopy (ICP-ES), by inductively coupled plasma-mass spectrometry (ICP-MS), gamma spectroscopy, plutonium isotopics, Sr-90 liquid scintillation, and Tc-99 methods.

Table 2-1. Sampling Height in the Tank and Sample Mass of Tank 35H Samples

Sample ID	Sampling Height (inches from bottom)	Sample Mass (g)
HTF-35-15-17	335”	259.9
HTF-35-15-18	195”	280.5
HTF-35-15-19	130”	283.1

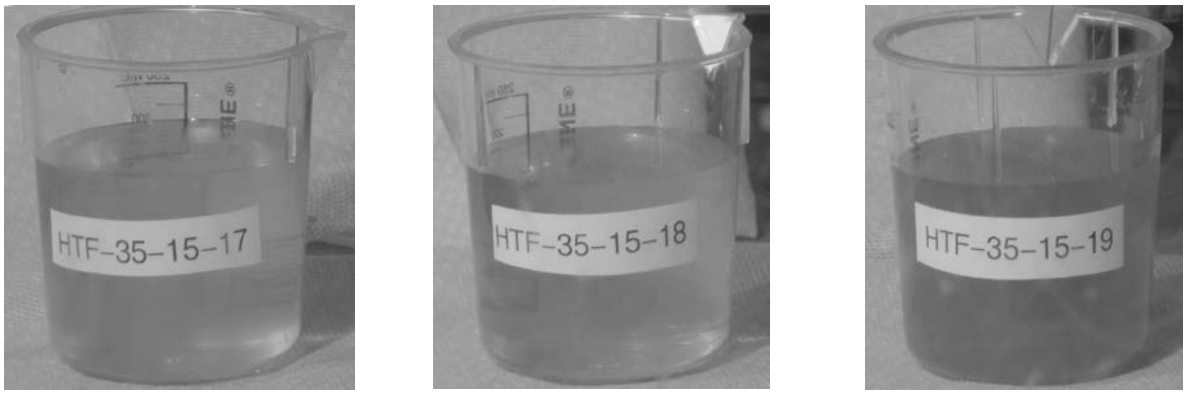

Figure 2-1. Samples from Tank 35H

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 Y7081-00081-03.

3.0 Results and Discussion

Tables 3-1 and 3-2 contain the results of the Tank 35H sample analyses. 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.

The results from the analysis indicate the compositions of the three samples show increasing concentrations for most analytes going from HTF-35-15-17 through HTF-35-15-19. However, sample HTF-35-15-19 has lower I-129, sulfate, and carbonate than the other two samples. The U-235 in the decanted (unfiltered) samples was below detection limits in the ICP-MS except for the HTF-35-15-19 sample. All of the plutonium isotope concentrations were below detection in HTF-35-15-17 decanted (unfiltered) and filtered samples. The plutonium concentrations measured in the filtered samples (Table 3-2) averaged ~23% lower than in the decanted (unfiltered) samples. The Sr-90 results from the filtered samples averaged ~27% lower than the unfiltered samples. The difference in the plutonium and strontium results for the filtered and unfiltered samples likely lies within the expected uncertainty for the measurement. However, the differences may indicate that filtration removed a small amount of suspended material from the samples.

The sodium concentration in the decanted (unfiltered) samples ranged from 4.85 M in HTF-35-15-17, to 8.11 M in HTF-35-15-18, to 8.53 M in HTF-35-15-19. The sum of the cations, primarily Na and K, compares well with the sum of the anions for each of the decanted (unfiltered) samples with differences of less than 10% except for sample HTF-35-15-18 that had a difference of 12%.

Table 3-1. Concentrations of Components of Tank 35H Decanted (Unfiltered) Liquid

		HTF-35-15-17		HTF-35-15-18		HTF-35-15-19	
Analyte	Units	Average	%RSD, n	Average	%RSD, n	Average	%RSD, n
F^-	M	<0.028	-	<0.028	-	<0.028	-
CH_2O^-	M	<0.018	-	<0.018	-	<0.018	-
Cl^-	M	<0.015	-	<0.015	-	<0.015	-
NO_2^-	M	0.884	3.0%, 3	1.16	0.1%, 3	1.36	0.8%, 3
Br^-	M	<0.033	-	<0.034	-	<0.034	-
NO_3^-	M	1.63	3.1%, 3	2.01	0.2%, 3	2.57	0.5%, 3
PO_4^{3-}	M	<0.006	-	<0.006	-	<0.006	-
SO_4^{2-}	M	0.068	3.4%, 3	0.063	1.4%, 3	0.033	1.9%, 3
$C_2O_4^{2-}$	M	<0.006	-	<0.006	-	<0.006	-
CO_3^{2-}	M	0.366	1.8%, 3	0.455	1.1%, 3	0.348	1.5%, 3
$OH^-_{(free)}$	M	1.45	2.3%, 3	2.86	4.2%, 3	3.17	1.1%, 3
Al	M	0.462	0.6%, 3	0.577	1.0%, 3	0.573	0.9%, 3
K	M	0.012	4.8%, 3	0.039	1.4%, 3	0.041	1.8%, 3
Na	M	4.85	0.6%, 3	8.11	0.8%, 3	8.53	0.8%, 3
P	M	0.004	2.7%, 3	0.010	2.1%, 3	0.012	1.1%, 3
Si	mg/L	22.0	11%, 3	25.3	55%, 3	32.6	55%, 2
Density	g/mL	1.22	0.2%, 3	1.34	0.3%, 3	1.37	0.6%, 3
Radioactive Species							
Sr-90	dpm/mL	9.82E+05	30%, 3	1.02E+06	5.7%, 3	2.50E+06	18%, 3
	µCi/mL	4.42E-01	-	4.58E-01	-	1.13E+00	-
Cs-137	dpm/mL	4.51E+08	2.3%, 3	1.48E+09	3.4%, 3	1.50E+09	6.4%, 3
	µCi/mL	2.03E+02	-	6.65E+02	-	6.78E+02	-
Tc-99	dpm/mL	8.94E+04	2.1%, 3	2.80E+05	2.0%, 3	3.26E+05	1.1%, 3
	µCi/mL	4.03E-02	-	1.26E-01	-	1.47E-01	-
I-129	dpm/mL	1.28E+02	22%, 3	3.63E+02	14%, 3	5.49E+01	22%, 3
	µCi/mL	5.78E-05	-	1.64E-04	-	2.47E-05	-
Pu-238	dpm/mL	<6.19E+02	-	1.39E+05	6.6%, 3	8.06E+05	7.6%, 3
	µCi/mL	<2.79E-04	-	6.27E-02	-	3.63E-01	-
Pu-239/240	dpm/mL	<3.12E+02	-	3.49E+03	0%, 1	1.61E+04	5.7%, 3
	µCi/mL	<1.41E-04	-	1.57E-03	-	7.25E-03	-
Pu-241	dpm/mL	<1.47E+03	-	8.51E+04	31%, 3	3.45E+05	6.4%, 3
	µCi/mL	<6.62E-04	-	3.83E-02	-	1.55E-01	-
Pu-Total	dpm/mL	<2.40E+03	-	2.24E+05	-	1.17E+06	-
	µCi/mL	<1.08E-03	-	1.01E-01	-	5.26E-01	-
U-235	mg/L	<5.91E-02	-	<6.40E-02	-	1.59E-01	1.6%, 3
U-238	mg/L	4.14E-01	0.7%, 3	1.22E+00	0.9%, 3	1.86E+00	1.6%, 3
U-total	mg/L	4.14E-01	-	1.22E+00	-	2.02E+00	-

Table 3-2. Concentrations of Components of Tank 35H Filtered Liquid

		HTF-35-15-17		HTF-35-15-18		HTF-35-15-19	
Analyte	Units	Average	%RSD, n	Average	%RSD, n	Average	%RSD, n
Hg	mg/L	<2.63E+00	-	9.22E+00	24%, 3	1.29E+01	16%, 3
Radioactive Species							
Sr-90	dpm/mL	5.16E+05	24%, 3	9.59E+05	27%, 3	1.77E+06	6.3%, 3
	µCi/mL	2.33E-01	-	4.32E-01	-	7.99E-01	-
Pu-238	dpm/mL	<5.10E+02	-	1.15E+05	7.6%, 3	6.79E+05	14%, 3
	µCi/mL	<2.30E-04	-	5.16E-02	-	3.06E-01	-
Pu-239/240	dpm/mL	<3.23E+02	-	<1.99E+03	-	1.24E+04	10%, 3
	µCi/mL	<1.45E-04	-	<8.95E-04	-	5.60E-03	-
Pu-241	dpm/mL	<8.03E+02	-	4.97E+04	13%, 3	2.88E+05	14%, 3
	µCi/mL	<3.62E-04	-	2.24E-02	-	1.30E-01	-

The free hydroxide and sodium show a larger change in going from the HTF-35-15-17 to HTF-35-15-18 than from HTF-35-15-18 to HTF-35-15-19 than the nitrate and nitrite concentrations. These results again indicate some stratification within the tank. The mercury concentrations measured in the filtered supernate were fairly low ranging from 12.9 to <2.63 mg/L.

The standards used for the silicon analysis (50 mg/L silicon in solution prepared by warm acid strike to final concentrations of 0.5, 1.0, and 2.0 mg/L) were all within 10% of the target concentration except for the 0.5 mg/L standard that was 11% high. The silicon concentration was below detectible levels in the process blank. The three decanted (unfiltered) samples show silicon concentrations of approximately 20-35 mg/L.

4.0 Conclusions

Three samples from different sampling heights within Tank 35H were analyzed by SRNL to support Salt Batch planning. The results from the analysis indicate the compositions of the three samples show increasing concentrations for most analytes going from HTF-35-15-17 through HTF-35-15-19 corresponding to successively deeper sampling locations. The data indicate some stratification within the tank. The plutonium and Sr-90 concentrations measured in the filtered samples were slightly lower than in the decanted (unfiltered) samples. The difference in the results for the filtered and unfiltered samples likely lies within the expected uncertainty for the measurement, but may indicate that filtration removed a very small amount of suspended material from the samples.

5.0 Acknowledgements

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

6.0 References

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2. Hay, M. S., *Task Technical and Quality Assurance Plan for Analysis of Samples from Salt Batch Source Tanks*, SRNL-RP-2013-00513, Rev. 2, March 2015.

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