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Characterization of Tank 9H Dissolution Batches in Support of Tank Closure Cesium Removal (TCCR) 1A Batch 1 Preparations

K. M. L. Taylor-Pashow

August 2021

SRNL-STI-2021-00385, Revision 0

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EXECUTIVE SUMMARY

Savannah River Remediation (SRR) is currently preparing the first batch of material to be processed through the Tank Closure Cesium Removal (TCCR) 1A system. The feed for TCCR 1A will consist of dissolved saltcake from Tank 9H. Two batches of salt (Batch 1A and Batch 1B) have been dissolved in Tank 9H and subsequently transferred to Tank 10H to prepare Batch 1 for TCCR 1A. Savannah River National Laboratory (SRNL) received samples from each batch of dissolved salt prior to transfer for characterization.

SRNL received both a surface and a variable depth sample from Batches 1A and 1B. In both cases no solids were observed in the surface sample, but were observed in the depth sample. For Batch 1A the variable depth sample was only slightly cloudy, while for Batch 1B the variable depth sample contained a significant amount (10.14 wt%) of solids. The solids were determined to be primarily aluminum containing phases, with only a small fraction (0.22 wt%) being sludge solids. In general, the samples from Batch 1A were more concentrated salt solutions than Batch 1B, with sodium concentrations of 8.53 and 8.57 M for the surface and filtered depth samples in Batch 1A, respectively. The sodium concentrations in Batch 1B samples ranged from 4.27 M for the surface sample to 7.57 M for the depth sample filtrate, indicating some stratification within the tank. The ^{137}Cs activity as well as the total Cs concentration in the filtered Batch 1A depth sample were approximately double the activity and concentration measured in the filtrate from the Batch 1B depth sample. The total Cs concentration in the Batch 1A depth sample filtrate was 22.4 mg/L, while for the Batch 1B depth sample filtrate the total Cs concentration was calculated to be 12.0 mg/L. These Cs concentrations are significantly higher than was measured in Batches 1-3 from Tank 10H dissolved saltcake which was previously processed through the original TCCR unit.

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LIST OF ABBREVIATIONS

ELN	electronic laboratory notebook
IC	ion chromatography
ICP-ES	inductively coupled plasma – emission spectroscopy
ICP-MS	inductively coupled plasma – mass spectrometry
IDCF	inhalation dose conversion factor
IDP	inhalation dose potential
LSC	liquid scintillation counting
M&TE	Measurement & Test Equipment
PMP	polymethylpentene
RSD	relative standard deviation
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TCCR	Tank Closure Cesium Removal
TIC/TOC	total inorganic carbon/total organic carbon
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
VDS	variable depth sample
WCS	Waste Characterization System

1.0 Introduction

In support of the Tank Closure Cesium Removal (TCCR) 1A program, SRNL analyzed several samples from Tank 9H. Salt in Tank 9H has been dissolved in two separate batches, referred to as Batches 1A and 1B, and subsequently transferred to Tank 10H to prepare the first batch of salt solution to be processed through TCCR 1A. In the TCCR unit the waste is filtered and transferred through columns containing crystalline silicotitanate ion exchange media. Samples were collected from Tank 9H after each dissolution campaign and transferred to SRNL for characterization before the transfers to Tank 10H were initiated. Prior samples from Tank 9H were collected and fully characterized in July 2019 and these results are documented in a previous report.[1] The July 2019 samples were collected after hydrolancing activities had been performed in Tank 9H, during which approximately 4200 gallons of water were added. Since that characterization, several additional hydrolancing activities as well as water additions were performed in Tank 9H, totaling the addition of approximately 27,000 gallons representing Batch 1A. Prior to collection of samples for characterization the contents of Tank 9H were recirculated for 72 hours. After recirculation was stopped, two samples, a surface sample (HTF-09-21-32) and a variable depth sample (HTF-09-21-33), were collected from the tank on April 15, 2021 and delivered to SRNL the following day for characterization as requested by SRR.[2] The Batch 1A material (28,726 gallons) was transferred from Tank 9H to Tank 10H on May 2-3, 2021. Following the transfer, 48,439 gallons of water were added to Tank 9H to prepare Batch 1B. The contents of the tank were recirculated for approximately 10 days prior to collecting two samples from the tank on May 17, 2021. A surface sample (HTF-09-21-41) and a variable depth sample (HTF-09-21-42) were collected from the tank and delivered to SRNL the same day for characterization as requested by SRR.[3] The Batch 1B material (67,479 gallons) was transferred to Tank 10H on June 23-25, 2021. Analytical results from characterization of these two sets of samples are summarized below.

2.0 Experimental Procedure

2.1 Tank 9H Batch 1A Samples (HTF-09-21-32 and HTF-09-21-33)

Two 200-mL dip samples were received from Tank 9H on April 16, 2021, one surface and one variable depth sample (VDS). The samples were placed into the Shielded Cells the same day. The samples were then opened and transferred to clear polymethylpentene (PMP) beakers for observation. The surface sample (HTF-09-21-32) was colorless and did not contain visible solids, while the VDS (HTF-09-21-33) appeared slightly cloudy. Photographs of the samples are provided below in Figure 3-1. The samples were not combined and were analyzed individually. A portion of the VDS (HTF-09-21-33) was filtered through a 0.2-µm cellulose nitrate filter and that filtered portion was used for completion of the corrosion control analyses. Both the unfiltered surface and the filtered variable depth samples were characterized for the normal corrosion control suite of analyses. Additional analyses requested in support of the TCCR program were performed on the VDS (unfiltered). Samples submitted for the corrosion control suite of analyses (gamma spectroscopy, inductively coupled plasma – emission spectroscopy (ICP-ES), total inorganic and total organic carbon (TIC/TOC), ion chromatography (IC) for anions, and titration for free hydroxide) were diluted by a factor of approximately 10 with distilled deionized water prior to analysis. Samples from the unfiltered portion of HTF-09-21-33 were diluted by a factor of approximately 5 with 3 M nitric acid prior to submission for the following analyses: inductively coupled plasma – mass spectrometry (ICP-MS), liquid scintillation counting (LSC) with and without Cs removal, gamma spectroscopy after Cs removal, and special radiochemistry methods for the following isotopes: ^{3}H, ^{94}Nb, ^{63}Ni, ^{151}Sm, and ^{232}U. Undiluted

and unfiltered samples from HTF-09-21-33 were submitted for ^{14}C and ^{79}Se analyses. The density of the unfiltered HTF-09-21-32 and filtered HTF-09-21-33 samples were measured using a Measuring & Test Equipment (M&TE) balance in duplicate using volumetric flasks. Samples used for density measurements were returned to the sample bottle.

2.2 Tank 9H Batch 1B Samples (HTF-09-21-41 and HTF-09-21-42)

Two 200-mL dip samples were received from Tank 9H on May 17, 2021, one surface and one VDS. The samples were placed into the Shielded Cells the following day. The samples were then opened and transferred to clear PMP beakers for observation after shaking by hand (manipulator) to mix. The surface sample (HTF-09-21-41) was colorless and did not contain visible solids, while the VDS (HTF-09-21-42) contained a significant amount of solids. Photographs of the samples are provided below in Figure 3-2. The samples were not combined and were analyzed individually. Approximately half of the VDS (HTF-09-21-42) was filtered through a 0.2-µm cellulose nitrate filter to give a filtrate sample that was used for some analyses. Both the unfiltered surface sample and variable depth filtrate sample were characterized for the normal corrosion control suite of analyses in addition to LSC with and without Cs removal. Samples submitted for the corrosion control suite of analyses (gamma spectroscopy, ICP-ES, TIC/TOC, IC for anions, and titration for free hydroxide) were diluted by a factor of approximately 10 with distilled deionized water prior to analysis. Samples from the unfiltered surface sample (HTF-09-21-41) and the depth sample filtrate (HTF-09-21-42) were diluted by a factor of approximately 5 with 3 M nitric acid prior to submission for LSC analysis. Samples of the HTF-09-21-42 original slurry were digested with aqua regia and then diluted with distilled deionized water prior to analysis by ICP-ES, gamma spectroscopy, and LSC. In addition, a sample of the unwashed solids collected on the filter were analyzed by X-ray diffraction. The densities of the samples (unfiltered surface sample and filtered and unfiltered VDS) were measured using a M&TE balance in duplicate using volumetric flasks or pipette tips (slurry sample). Samples used for density measurements were returned to the sample bottles.

In addition to the analyses performed, a set of dissolution experiments were performed to determine if the solids present in the sample were water soluble. For these experiments aliquots of both the original slurry from HTF-09-21-42 as well as the solids collected on the filter after filtration of HTF-09-21-42 were placed into vials and attempts were made to dissolve the solids in distilled deionized water. For the slurry sample, 0.668 g of slurry was transferred to a vial. Water was added dropwise, with intermittent mixing (shaking using manipulator), up until a maximum of a 15:1 mass ratio of water to sample had been added. A total of 10.025 g of water was added to the slurry sample. A similar process was performed with a sample of the solids collected from the filter. In the case of the solids, 0.614 g of sample was transferred to the vial and a total of 9.246 g of water was added.

2.3 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 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 work was performed following the applicable Task Technical and Quality Assurance Plan (TTQAP).[4] The Task Technical Request (TTR) associated with this work[5] requested a functional classification of Safety Class (see section 9.5 of the TTQAP entitled "Clarification of Safety Significant Functional Classification"). To match the requested functional

classification, this report and calculations within received a technical review by design verification.[6] Data are recorded in the Electronic Laboratory Notebook (ELN) system.[7]

3.0 Results and Discussion

3.1 Tank 9H TCCR Batch 1A Samples (HTF-09-21-32 and HTF-09-21-33)

Photographs of the Tank 9H Batch 1A samples are provided in Figure 3-1. The surface sample (HTF-09-21-32) appeared clear and colorless, with no evidence of significant solids, while the variable depth sample (HTF-09-21-33) appeared slightly cloudy.

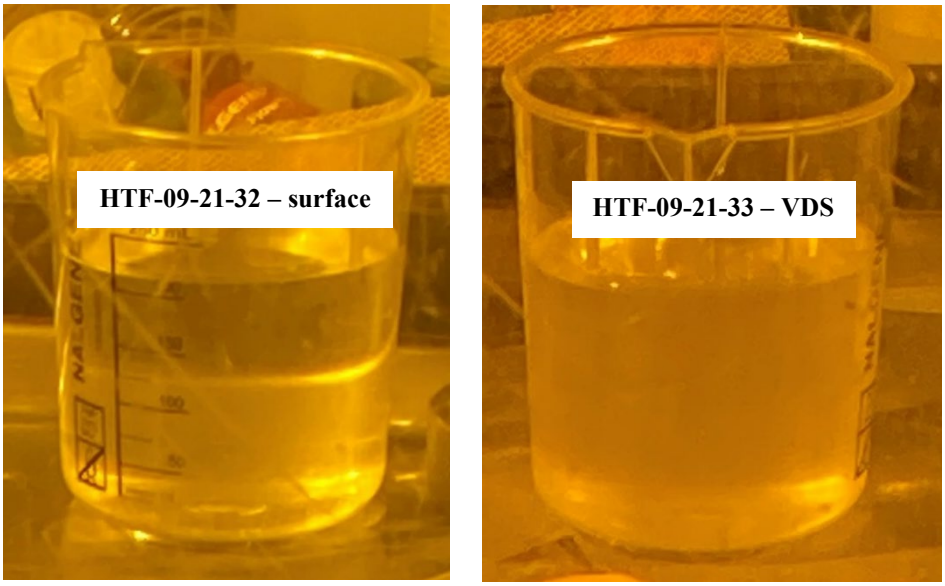

Figure 3-1. Photographs of Tank 9H Batch 1A samples HTF-09-21-32 (left) and HTF-09-21-33 (right).

The densities of the Tank 9H Batch 1A samples are reported in Table 3-1. The density of the surface sample was slightly higher than the VDS; although the percent difference between the two samples is only 1.3%.

Table 3-1. Density Measurements of Tank 9H Batch 1A Samples[8]

Sample	Sample Location	Avg. Density (g/mL)	% RSD
HTF-09-21-32	surface	1.437	0.043
HTF-09-21-33 (filtered)	variable depth	1.418	0.189

The ICP-ES results of the Tank 9H Batch 1A samples are shown in Table 3-2. The only elements above the detection limit in both samples were Al, Cr, K, Mo, Na, P, and S. In general, the concentrations were slightly higher for all of these elements in the VDS; however, most were within the analytical uncertainty. The sodium concentrations (8.53 M and 8.57 M) were slightly lower than what is predicted based on the measured densities.[9]

Table 3-2. ICP-ES Results for the Tank 9H Batch 1A Samples

Element	HTF-09-21-32 (M)	Uncertainty[a]	HTF-09-21-33 (M)	Uncertainty[a]	% Difference[b]
Ag	< 1.13E-05	n/a	< 1.13E-05	n/a	n/a
Al	0.153	10%	0.176	10%	14.0%
B	< 9.99E-03	n/a	< 9.99E-03	n/a	n/a
Ba	< 2.00E-05	n/a	< 2.00E-05	n/a	n/a
Be	< 6.44E-05	n/a	< 6.42E-05	n/a	n/a
Ca	< 5.71E-04	n/a	< 5.69E-04	n/a	n/a
Cd	< 1.34E-05	n/a	< 1.35E-05	n/a	n/a
Ce	< 1.16E-04	n/a	< 1.16E-04	n/a	n/a
Co	< 2.73E-05	n/a	< 2.73E-05	n/a	n/a
Cr	1.20E-03	10%	1.30E-03	10%	8.0%
Cu	< 2.66E-04	n/a	< 2.66E-04	n/a	n/a
Fe	< 1.01E-04	n/a	< 1.01E-04	n/a	n/a
Gd	< 4.72E-05	n/a	< 4.71E-05	n/a	n/a
K	1.70E-02	10.8%	2.00E-02	11%	16.2%
La	< 1.60E-05	n/a	< 1.60E-05	n/a	n/a
Li	< 2.01E-03	n/a	< 2.00E-03	n/a	n/a
Mg	< 6.71E-05	n/a	< 6.71E-05	n/a	n/a
Mn	< 1.00E-04	n/a	< 1.00E-04	n/a	n/a
Mo	3.00E-04	10.1%	4.00E-04	12%	28.6%
Na	8.53	10%	8.57	10%	0.47%
Ni	< 6.83E-05	n/a	< 6.82E-05	n/a	n/a
P	7.50E-03	10.2%	8.20E-03	10%	8.9%
Pb	< 4.20E-04	n/a	< 4.19E-04	n/a	n/a
S	3.50E-02	10.2%	3.90E-02	10%	10.8%
Sb	< 3.61E-04	n/a	< 3.60E-04	n/a	n/a
Si	< 2.33E-03	n/a	< 2.33E-03	n/a	n/a
Sn	< 9.52E-04	n/a	< 9.52E-04	n/a	n/a
Sr	< 6.62E-06	n/a	< 6.61E-06	n/a	n/a
Th	< 7.07E-05	n/a	< 7.02E-05	n/a	n/a
Ti	< 3.97E-04	n/a	< 3.97E-04	n/a	n/a
U	< 1.47E-04	n/a	< 1.46E-04	n/a	n/a
V	< 1.80E-04	n/a	< 1.79E-04	n/a	n/a
Zn	< 8.08E-05	n/a	< 8.06E-05	n/a	n/a
Zr	< 2.07E-05	n/a	< 2.07E-05	n/a	n/a

[a]The uncertainty is the reported analytical method uncertainty at two sigma. [b]The percent difference between the measured values for the surface and variable depth samples.

Table 3-3 provides the anion and carbon results for the Tank 9H Batch 1A samples. For the majority of the anions the VDS had a higher concentration, the exceptions were oxalate and carbonate where the surface sample was higher in concentration. The higher carbonate concentration near the surface would be expected, and the oxalate concentrations in both samples were relatively low compared to the other anions. The differences between the surface and the variable depth samples ranged from 8.3% different for nitrate to 23% different for the free hydroxide. The total organic carbon (TOC) content in the two samples was similar (within analytical error). The anion and cation balance showed a percent difference of 4.4% for the surface sample and 6.3% for the variable depth sample, with the anions being higher in both cases.

Table 3-3. Anion and Carbon Results for the Tank 9H Batch 1A Sample[8]

Analyte	HTF-09-21-32	Uncertainty[a]	HTF-09-21-33	Uncertainty[a]	% Difference[b]
Free OH^- (M)	0.420	10%	0.529	10%	23.0%
NO_3^- (M)	4.82	10%	5.24	10%	8.3%
SO_4^{2-} (M)	2.51E-02	10%	2.95E-02	10%	16.1%
NO_2^- (M)	0.979	10%	1.140	10%	15.2%
$C_2O_4^{2-}$ (M)	3.02E-03	10%	2.55E-03	10%	16.9%
F^- (M)	< 5.26E-03	n/a	< 5.26E-03	n/a	n/a
Cl^- (M)	1.92E-03	10%	< 2.82E-03	n/a	n/a
CO_3^{2-} (M)	1.25	10%	1.00	10%	22.2%
$Al(OH)_4^-$ (M)[c]	0.153	10%	0.176	10%	14.0%
TOC (mg/L)	305	10%	284	10%	7.1%

[a]The uncertainty is the reported analytical method uncertainty at one sigma for all anions except Free OH^-, CO_3^{2-}, and $Al(OH)_4$- where the method uncertainty is 2 sigma. Method uncertainty for TOC is also 2 sigma. [b]The percent difference between the measured values for the surface and variable depth samples. [c]Based on Al concentration measured by ICP-ES (see Table 3-2).

Gamma spectroscopy was performed for both samples as part of the corrosion control analysis. The results from these analyses are provided in Table 3-4. As can be seen, the ^{137}Cs activity was approximately 14% higher in the VDS compared to the surface sample. Based on these results, the remaining required analyses for the TCCR program were performed on the unfiltered VDS.

Table 3-4. Cs Activity from Gamma Counting for Tank 9H Batch 1A Samples[8]

Isotope	HTF-09-21-32 (dpm/mL)	Uncertainty[a]	HTF-09-21-33 (dpm/mL)	Uncertainty[a]	% Difference[b]
^{134}Cs	< 2.05E+05	MDA	< 2.41E+05	MDA	n/a
^{137}Cs	6.92E+08	5.0%	7.98E+08	5.0%	14.2%

[a]The uncertainty is the reported analytical method uncertainty at one sigma. MDA = minimum detectable activity.
[b]The percent difference between the measured values for surface and variable depth samples.

The isotopic distribution of Cs based on the mass spectrometry results is provided in Table 3-5. The total Cs calculated using the isotopic ratios from the ICP-MS data and the ^{137}Cs amount from the gamma data is 22.4 mg/L, which is in good agreement with the sum of the Cs isotope masses reported by ICP-MS (22.3 mg/L). The full suite of ICP-MS results is provided in Table 3-6.

Table 3-5. Cs Isotopes from ICP-MS for the Tank 9H Batch 1A VDS (HTF-09-21-33)

Isotope	Mean Concentration (mg/L)	%RSD[a]	Isotopic Distribution, Mass %	Mole Fraction
Cs-133	16.6	2.59	74.5	0.750
Cs-134	5.20E-03	1.81	0.02	0.0002
Cs-135	1.57	1.77	7.04	0.070
Cs-137	4.10	0.74	18.4	0.180

[a]The %RSD is based on the standard deviation of duplicate samples. The reported method uncertainty is 20% at two sigma.

Table 3-6. ICP-MS Results (including As and Se) for Tank 9H Batch 1A VDS (HTF-09-21-33)

m/z	Avg. Conc. (µg/L)	% RSD*	m/z	Avg. Conc. (µg/L)	% RSD*	m/z	Avg. Conc. (µg/L)	% RSD*
59	< 5.00E+00	n/a	**128**	< 5.00E+00	n/a	**174**	< 5.00E+00	n/a
84	< 5.00E+00	n/a	**130**	< 5.00E+00	n/a	**175**	< 5.00E+00	n/a
85	2.03E+03	1.75%	**133**	1.66E+04	2.59%	**176**	< 5.00E+00	n/a
86	< 5.00E+00	n/a	**134**	5.20E+00	1.81%	**177**	< 5.00E+00	n/a
87	4.30E+03	1.85%	**135**	1.57E+03	1.77%	**178**	< 5.00E+00	n/a
88	6.38E+00	5.14%	**136**	< 5.00E+00	n/a	**179**	< 5.00E+00	n/a
89	< 5.00E+00	n/a	**137**	4.10E+03	0.74%	**180**	< 5.00E+00	n/a
90	8.93E+00	9.35%	**138**	2.07E+01	17.18%	**181**	< 5.00E+00	n/a
91	9.25E+00	0.96%	**139**	< 5.00E+00	n/a	**182**	1.29E+02	4.49%
92	1.93E+02	0.58%	**140**	< 5.00E+00	n/a	**183**	7.04E+01	1.86%
93	1.07E+01	2.39%	**141**	< 5.00E+00	n/a	**184**	1.51E+02	0.92%
94	1.25E+02	0.16%	**142**	< 5.00E+00	n/a	**185**	< 5.00E+00	n/a
95	1.13E+04	0.88%	**143**	< 5.00E+00	n/a	**186**	1.37E+02	1.30%
96	2.22E+02	2.26%	**144**	< 5.00E+00	n/a	**187**	5.71E+00	6.54%
97	1.03E+04	1.07%	**145**	< 5.00E+00	n/a	**191**	< 5.00E+00	n/a
98	1.00E+04	0.22%	**146**	< 5.00E+00	n/a	**193**	< 5.00E+00	n/a
99	6.61E+03	0.23%	**147**	< 5.00E+00	n/a	**194**	< 5.00E+00	n/a
100	1.03E+04	0.37%	**148**	< 5.00E+00	n/a	**195**	< 5.00E+00	n/a
101	5.13E+02	0.88%	**149**	< 5.00E+00	n/a	**196**	6.24E+00	5.00%
102	4.48E+02	0.05%	**150**	< 5.00E+00	n/a	**198**	3.05E+02	0.26%
103	1.35E+03	0.56%	**151**	< 5.00E+00	n/a	**203**	< 5.00E+00	n/a
104	2.32E+02	1.71%	**152**	< 5.00E+00	n/a	**204**	2.24E+02	0.12%
105	< 1.50E+01	n/a	**153**	< 5.00E+00	n/a	**205**	7.15E+00	1.02%
106	< 1.00E+01	n/a	**154**	< 5.00E+00	n/a	**206**	8.80E+02	0.86%
107	< 1.00E+01	n/a	**155**	< 5.00E+00	n/a	**207**	7.47E+02	0.80%
108	< 1.00E+01	n/a	**156**	< 5.00E+00	n/a	**208**	1.83E+03	1.52%
109	< 5.00E+00	n/a	**157**	< 5.00E+00	n/a	**229**	< 5.00E+00	n/a
110	< 5.00E+00	n/a	**158**	< 5.00E+00	n/a	**230**	< 5.00E+00	n/a
111	5.98E+00	5.51%	**159**	< 5.00E+00	n/a	**232**	< 5.00E+00	n/a
112	2.07E+01	0.81%	**160**	< 5.00E+00	n/a	**233**	< 5.00E+00	n/a
113	8.58E+01	0.89%	**161**	< 5.00E+00	n/a	**234**	< 5.00E+00	n/a
114	1.91E+01	0.16%	**162**	< 5.00E+00	n/a	**235**	2.09E+01	2.92%
116	2.97E+02	1.21%	**163**	< 5.00E+00	n/a	**236**	5.13E+00	n/a
117	2.02E+02	2.87%	**164**	< 5.00E+00	n/a	**237**	< 5.00E+00	n/a
118	5.77E+02	0.68%	**165**	< 5.00E+00	n/a	**238**	5.10E+02	1.59%
119	1.89E+02	0.60%	**166**	< 5.00E+00	n/a	**239**	< 5.00E+00	n/a
120	7.72E+02	2.11%	**167**	< 5.00E+00	n/a	**240**	< 5.00E+00	n/a
121	< 5.00E+00	n/a	**168**	< 5.00E+00	n/a	**241**	< 5.00E+00	n/a
122	2.06E+02	0.49%	**169**	< 5.00E+00	n/a	**242**	< 5.00E+00	n/a
123	< 5.00E+00	n/a	**170**	< 5.00E+00	n/a	**243**	< 5.00E+00	n/a
124	3.13E+02	1.56%	**171**	< 5.00E+00	n/a	**244**	< 5.00E+00	n/a
125	< 5.00E+00	n/a	**172**	< 5.00E+00	n/a	**As**	1.83E+02	2.72%
126	1.01E+03	0.83%	**173**	< 5.00E+00	n/a	**Se**	< 2.50E+02	n/a

*The %RSD is based on the standard deviation of duplicate samples. The reported method uncertainty is 20% at two sigma.

Total alpha and beta activities in the Tank 9H VDS (HTF-09-21-33) as measured by LSC are shown in Table 3-7. The LSC counting was performed both with and without Cs removal to obtain a lower detection limit for the alpha. Both sets of values are reported below.

Table 3-7. Alpha and Beta Activity in Tank 9H Batch 1A VDS (HTF-09-21-33)

	Cs Removed (dpm/mL)	%RSD[a]	Without Cs Removal (dpm/mL)	%RSD[a]
Alpha count	< 2.99E+04[b]	n/a	< 2.59E+07[b]	n/a
Beta count	6.82E+05	0.56	9.18E+08	0.03

[a]The %RSD is based on the standard deviation of duplicate samples. The reported method uncertainty is 10% at one sigma. [b]Upper limit value.

The majority of the remaining radionuclides that were analyzed for were below the method detection limit with the exception of ^{79}Se, ^{126}Sb, ^{126}Sn, and ^{232}U. In the case of ^{79}Se and ^{232}U one sample of each pair of replicates was above the method detection limit, while the other was below and was reported as a less than value. A summary of the activities of other radionuclides analyzed for is provided in Table 3-8.

Table 3-8. Activities of Other Radionuclides in Tank 9H Batch 1A VDS (HTF-09-21-33)

Radionuclides	Avg. Activity	%RSD[a]	Avg. Method Unc.
^{3}H	< 5.68E-04 µCi/mL	n/a	MDA[b]
^{14}C	< 1.25E+03 dpm/mL	n/a	Upper Limit
^{59}Ni	< 3.36E+01 dpm/mL	n/a	MDA
^{60}Co	< 1.31E+02 dpm/mL	n/a	MDA
^{63}Ni	<1.09E+02 dpm/mL	n/a	Upper Limit
^{79}Se	6.71E+02 dpm/mL[c]	n/a	35.9%
^{106}Ru	< 9.59E+02 dpm/mL	n/a	MDA
^{125}Sb	< 5.76E+02 dpm/mL	n/a	MDA
^{126}Sb	2.68E+03 dpm/mL	0.44%	5.00%
^{126}Sn	2.68E+03 dpm/mL	0.44%	5.00%
^{135}Cs	5.03E+03 dpm/mL	9.71%	20.0%
^{144}Ce	< 1.05E+03 dpm/mL	n/a	MDA
^{147}Pm	< 8.43E+02 dpm/mL	n/a	Upper Limit
^{151}Sm	< 1.08E+03 dpm/mL[d]	n/a	Upper Limit/MDA[d]
^{154}Eu	< 2.96E+02 dpm/mL	n/a	MDA
^{232}U	2.56E+00 dpm/mL[e]	n/a	20.0%
^{241}Am	< 1.45E+03 dpm/mL	n/a	MDA

[a]The %RSD is based on the standard deviation of duplicate samples. [b]MDA = minimum detectable activity. [c]Value represents single value above the method detection limit, replicate sample reported as < 1.55E+02 dpm/mL. [d]Duplicate samples were both reported as less than values, although one was reported as an upper limit (< 1.48E+03) and the other was reported as MDA (< 6.69E+02). [e]Value represents single value above the method detection limit, replicate sample reported as < 6.34E-01 dpm/mL.

3.2 Tank 9H TCCR Batch 1B Samples (HTF-09-21-41 and HTF-09-21-42)

Photographs of the Tank 9H Batch 1B samples are provided in Figure 3-2. The surface sample (HTF-09-21-41) appeared clear and colorless, with no evidence of significant solids, while the VDS (HTF-09-21-42) contained a significant amount of light gray colored solids. The solids were found to be insoluble in water based on the attempted dissolution of the solids with up to a 15:1 mass ratio of water to sample. Photographs of the dissolution experiments are provided in Figures 3-3 and 3-4.

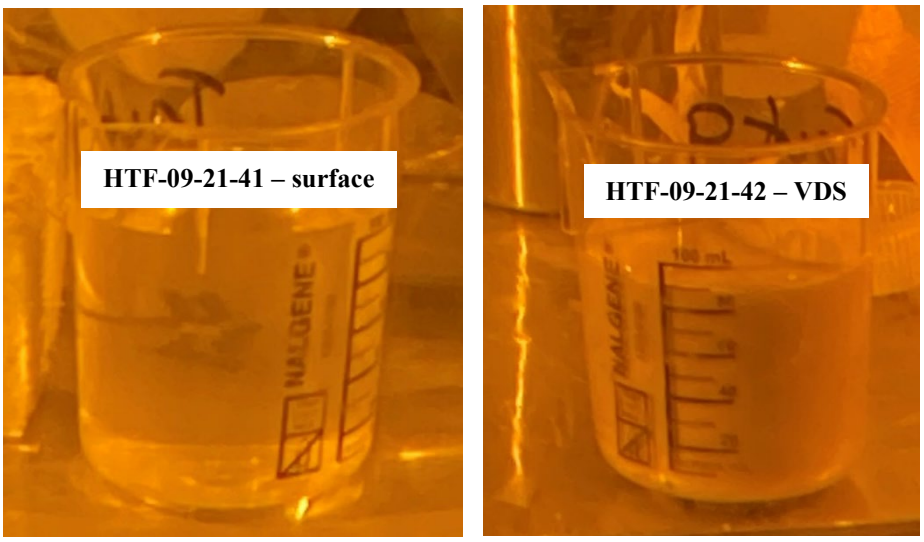

Figure 3-2. Photographs of Tank 9H Batch 1B samples HTF-09-21-41 (left) and HTF-09-21-42 (right).

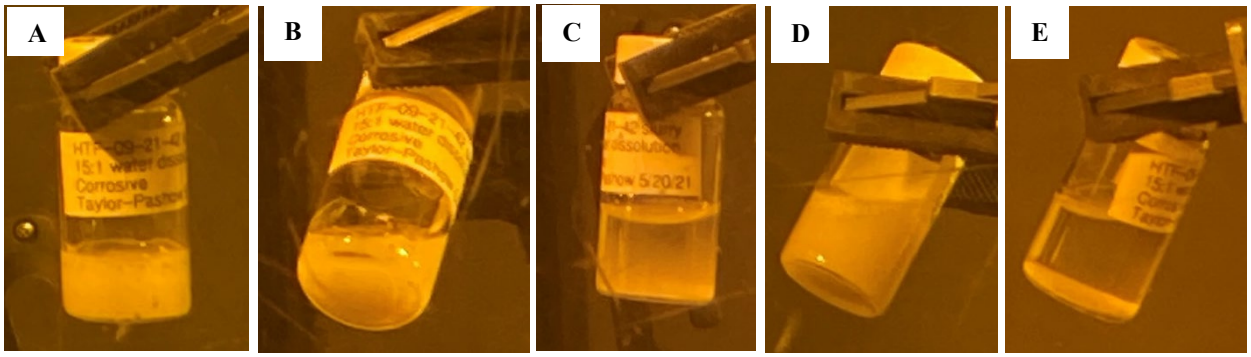

Figure 3-3. Photographs of the attempted dissolution of the solids in HTF-09-21-42 slurry. A) Water added – dispersing solids. B) Water added – dispersing solids. C) Water added up to the 15:1 ratio. D) 15:1 ratio after agitation. E) 15:1 ratio after standing undisturbed for ~30 minutes.

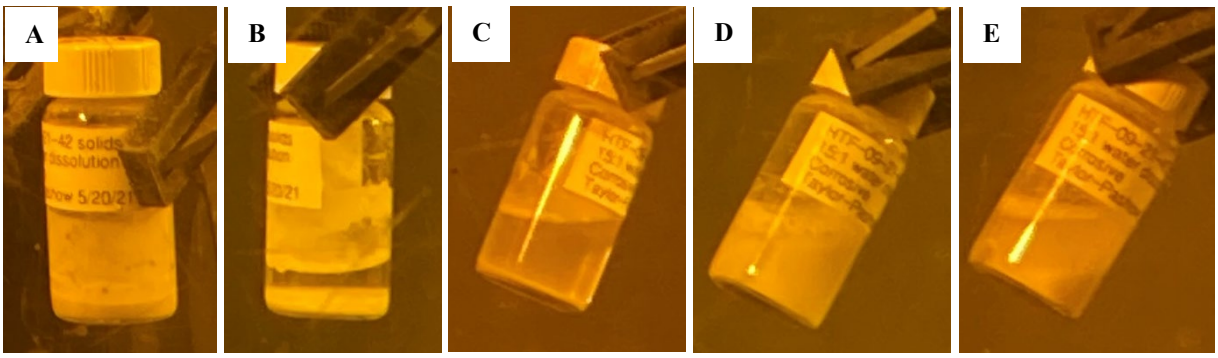

Figure 3-4. Photographs of the attempted dissolution of the HTF-09-21-42 solids. A) Small amount of water added – dispersing solids/solids coating inside of vial. B) Small amount of solids on the surface after standing for ~30 minutes. C) Water added up to the 15:1 ratio. D) 15:1 ratio after agitation. E) 15:1 ratio – solids beginning to settle (< 1 minute elapsed time between pictures D and E).

The densities of the Tank 9H Batch 1B samples are reported in Table 3-9. The density of the surface sample was approximately 12% lower than the filtered VDS. The density of the VDS slurry was higher than the filtrate, as expected due to the presence of solids.

Table 3-9. Density Measurements of Tank 9H Batch 1B Samples

Sample	Sample Location	Sample Type	Avg. Density (g/mL)	% RSD
HTF-09-21-41	surface	as received	1.223	0.10
HTF-09-21-42	variable depth	filtrate	1.378	0.036
HTF-09-21-42	variable depth	as received (slurry)	1.446	0.34

To determine the amount of insoluble solids present in the VDS sample, total solids measurements were performed on samples of the filtrate and slurry from HFT-09-21-42. The measurements were performed by drying duplicate samples of each (filtrate and slurry) at 115 °C until a constant weight was achieved (3 days). Results from the total solids measurements are provided in Table 3-10, along with the amount of insoluble solids in the slurry calculated using equation 1 shown below.

$$Wt\%_{insoluble} = (Wt\%_{total} - Wt\%_{dissolved}) / (100 - Wt\%_{dissolved}) \quad (1)$$

Table 3-10. Weight Percent Solids Measurements

Sample	Trial 1 (wt%)	Trial 2 (wt%)	Average (wt%)	% RSD
Filtrate (Dissolved Solids)	44.68	44.83	44.76	0.24
Slurry (Total Solids)	50.04	50.67	50.36	0.88
Insoluble Solids (Calculated)	n/a	n/a	10.14	n/a

The ICP-ES results of the Tank 9H Batch 1B samples are shown in Table 3-11. The only elements above the detection limit in both the surface and variable depth filtrate samples were Al and Na. The VDS filtrate also contained detectable amounts of Cr and S. ICP-ES results indicate that the filtered depth sample is a more concentrated salt solution (7.57 M Na^+) compared to the surface sample (4.27 M Na^+), indicating some stratification within the tank. The Batch 1B samples were less concentrated than the Batch 1A samples which had sodium concentrations of 8.53 and 8.57 M for the surface and filtered VDS samples, respectively. This is also consistent with the measured densities. Analysis of the digested slurry sample

shows a large increase in the aluminum concentration indicating that aluminum rich solids are present in the slurry. A number of other elements were also measured in the digested slurry that were either not present in the filtrate or were present in lower concentrations. Those elements included Ba, Ca, Cr (increased amount), Fe, K, Mg, Mn, Mo, Si, U, and Zn. Powder X-ray diffraction was performed on a sample of the unwashed solids collected on the filter. As can be seen in Figure 3-5, the identified species included $Al(OH)_3$ (gibbsite), $NaNO_3$, Al_2O_3, Fe_2O_3, and a potential aluminum silicate phase with the formula $Al_{4.68}Si_{1.32}O_{9.66}$. As aluminum can be present in both the sludge and salt phases, data from Waste Characterization System (WCS) Online was used to determine what fraction of the measured aluminum in the digested slurry could be attributed to sludge solids. The ratio of Al to Fe in the sludge phase in Tank 9H as reported in WCS was used for this calculation. Using the calculated Al/Fe ratio of 0.1435 from the WCS data and the amount of Fe measured in the slurry (456 mg/L), it was estimated that ~65 mg/L of Al (of the 73,000 mg/L measured) could be attributed to sludge. Using this amount, as well as the amounts of other elements present in the sludge a total concentration of sludge solids was calculated to be 3229 mg/L. Using the measured density of the slurry, the sludge solids account for 0.22 wt% of the total mass of the slurry.

Table 3-11. ICP-ES Results for the Tank 9H Batch 1B Samples

Element	HTF-09-21-41 Original (M)	Uncertainty[a]	HTF-09-21-42 Filtrate (M)	Uncertainty[a]	HTF-09-21-42 Slurry (M)	%RSD[b]
Ag	< 1.19E-05	n/a	< 1.24E-05	n/a	< 6.24E-06	n/a
Al	0.034	10%	0.083	10%	2.71	2.24%
B	< 1.15E-02	n/a	< 1.20E-02	n/a	< 6.06E-03	n/a
Ba	< 6.56E-06	n/a	< 6.87E-06	n/a	1.74E-04	1.28%
Be	< 6.72E-05	n/a	< 7.05E-05	n/a	< 4.81E-05	n/a
Ca	< 5.96E-04	n/a	< 6.24E-04	n/a	3.90E-03	43.2%
Cd	< 2.67E-05	n/a	< 2.79E-05	n/a	< 7.46E-06	n/a
Ce	< 5.04E-05	n/a	< 5.27E-05	n/a	< 2.66E-05	n/a
Co	< 6.40E-05	n/a	< 6.70E-05	n/a	< 1.51E-05	n/a
Cr	< 2.00E-04	n/a	5.00E-04	10.3%	8.10E-04	1.70%
Cu	< 2.79E-04	n/a	< 2.91E-04	n/a	< 7.69E-05	n/a
Fe	< 1.06E-04	n/a	< 1.10E-04	n/a	8.17E-03	2.02
Gd	< 4.93E-05	n/a	< 5.16E-05	n/a	< 9.57E-06	n/a
K	< 0.010	n/a	< 0.010	n/a	9.93E-03	3.95%
La	< 1.67E-05	n/a	< 1.75E-05	n/a	< 8.81E-06	n/a
Li	< 1.53E-03	n/a	< 1.60E-03	n/a	< 6.20E-04	n/a
Mg	< 6.99E-05	n/a	< 7.32E-05	n/a	7.88E-04	22.9%
Mn	< 5.44E-05	n/a	< 5.70E-05	n/a	1.48E-03	1.76%
Mo	< 2.00E-04	n/a	< 2.00E-04	n/a	1.45E-04	1.54%
Na	4.27	10%	7.57	10%	6.06	0.07%
Ni	< 7.14E-05	n/a	< 7.46E-05	n/a	< 3.76E-05	n/a
P	< 4.00E-03	n/a	< 4.00E-03	n/a	< 3.70E-03	n/a
Pb	< 4.39E-04	n/a	< 4.60E-04	n/a	< 1.53E-04	n/a
S	< 1.63E-02	n/a	0.030	10.1%	2.18E-02	0.44%
Sb	< 3.77E-04	n/a	< 3.94E-04	n/a	< 9.73E-05	n/a
Si	< 2.43E-03	n/a	< 2.55E-03	n/a	7.28E-03	9.49%
Sn	< 2.58E-03	n/a	< 2.70E-03	n/a	< 1.45E-04	n/a
Sr	< 4.19E-05	n/a	< 4.39E-05	n/a	< 3.65E-06	n/a
Th	< 1.90E-04	n/a	< 1.99E-04	n/a	< 1.87E-05	n/a
Ti	< 4.16E-04	n/a	< 4.35E-04	n/a	< 7.91E-05	n/a
U	< 2.44E-04	n/a	< 2.56E-04	n/a	1.67E-04	4.89%
V	< 3.00E-04	n/a	< 3.00E-04	n/a	< 9.90E-05	n/a
Zn	< 9.16E-05	n/a	< 9.59E-05	n/a	3.56E-04	1.76%
Zr	< 2.17E-05	n/a	< 2.27E-05	n/a	< 1.49E-05	n/a

[a]The uncertainty is the reported analytical method uncertainty (at two sigma). [b]Percent relative standard deviation from duplicate samples.

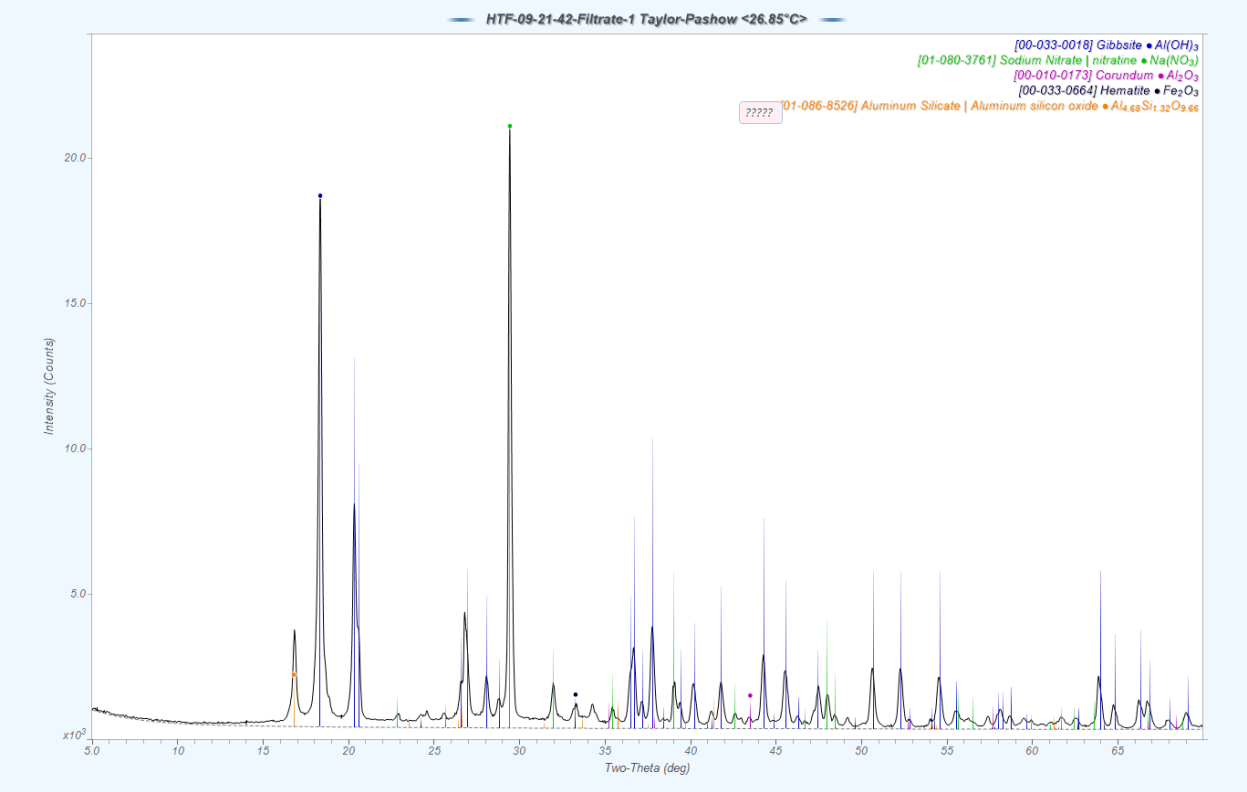

Figure 3-5. Powder X-ray diffraction of the unwashed solids from HTF-09-21-42 that were collected on a 0.2 µm filter.

Table 3-12 provides the anion and carbon results for the Tank 9H Batch 1B samples (surface and VDS filtrate). With the exception of carbonate, the anion concentrations were higher in the depth sample when compared to the surface sample. This is consistent with the ICP-ES results, which indicated the depth sample was a more concentrated salt solution than the surface sample. The largest difference between the samples was for the free hydroxide, where the surface sample had a relatively low concentration of hydroxide compared to recent samples. The carbonate and total organic carbon (TOC) concentrations in the two samples were similar. The anion and cation balance showed a percent difference of 7.9% for the surface sample and 11.5% for the VDS, with the anions being higher in both cases.

Table 3-12. Anion and Carbon Results for the Tank 9H Batch 1B Sample[10]

Analyte	HTF-09-21-41	Uncertainty[a]	HTF-09-21-42	Uncertainty[a]	% Difference[b]
Free OH^- (M)	0.070[c]	10%	0.654	10%	159%
NO_3^- (M)	3.06	10%	5.96	10%	64.3%
SO_4^{2-} (M)	7.66E-03	10%	1.88E-02	10%	84.2%
NO_2^- (M)	0.162	10%	0.505	10%	103%
$C_2O_4^{2-}$ (M)	4.55E-03	10%	2.63E-03	10%	53.5%
F^- (M)	< 5.26E-03	n/a	< 5.26E-03	n/a	n/a
Cl^- (M)	< 2.82E-03	n/a	< 2.82E-03	n/a	n/a
CO_3^{2-} (M)	0.630	10%	0.623	10%	1.32%
$Al(OH)_4^-$ (M)[d]	0.034	10%	0.083	10%	83.8%
TOC[e] (mg/L)	253	10%	292	10%	14.3%

[a]The uncertainty is the reported analytical method uncertainty at one sigma for all anions except Free OH^-, CO_3^{2-}, and $Al(OH)_4$- where the method uncertainty is 2 sigma. Method uncertainty for TOC is also 2 sigma. [b]The percent difference between the measured values for surface and variable depth samples. [c]Analysis repeated with larger sample size to confirm the low concentration. Value reported is the average of the two measurements. %RSD of the duplicate measurements was 10.1%. [d]Based on Al concentration measured by ICP-ES (see Table 3-11). [e]Total organic carbon.

Gamma spectroscopy was performed for both the unfiltered surface and variable depth filtrate samples as part of the corrosion control analysis.[10] Gamma spectroscopy was also performed on the digested slurry sample. The results from these analyses are provided in Table 3-13. As can be seen, the ^{137}Cs activity was approximately 3.5 times higher in the variable depth filtrate sample compared to the surface sample. The ^{137}Cs activity in the HTF-09-21-42 slurry was approximately 32% lower than measured in the filtrate, indicating the solids present in the slurry contained little ^{137}Cs.

Table 3-13. ^{137}Cs Activity from Gamma Counting for Tank 9H Batch 1B Samples

	HTF-09-21-41 Original (dpm/mL)	Uncertainty[a]	HTF-09-21-42 Filtrate (dpm/mL)	Uncertainty[a]	HTF-09-21-42 Slurry (dpm/mL)	Uncertainty[a]
^{137}Cs	1.08E+08	5.00%	3.85E+08	5.00%	2.60E+08	5.00%

[a]The uncertainty is the reported analytical method uncertainty at one sigma. Duplicate samples were analyzed for the HTF-09-21-42 slurry sample and gave identical results.

The isotopic distribution of Cs based on the mass spectrometry results is provided in Table 3-14. The total Cs calculated using the isotopic ratios from the ICP-MS data and the ^{137}Cs amount from the gamma data is 12.0 mg/L, which is about 14% higher than the sum of the Cs isotope masses reported by ICP-MS (10.5 mg/L). The full suite of ICP-MS results is provided in Table 3-15. The total Cs in this depth sample is 54% of the value measured in the Batch 1A sample.

Table 3-14. Cs Isotopes from ICP-MS for the Tank 9H Batch 1B VDS Filtrate (HTF-09-21-42)

Isotope	Mean Concentration (mg/L)	%RSD[a]	Isotopic Distribution, Mass %	Mole Fraction
Cs-133	8.01	0.16	76.3	0.768
Cs-134	< 5.06E-03	n/a	< 0.04	< 0.0005
Cs-135	0.741	0.11	7.04	0.070
Cs-137	1.74	0.04	16.6	0.162

[a]The %RSD is based on the standard deviation of duplicate samples. The reported method uncertainty is 20% at two sigma.

Table 3-15. ICP-MS Results for Tank 9H Batch 1B VDS (HTF-09-21-42)

m/z	Avg. Conc. (µg/L)	% RSD[a]	m/z	Avg. Conc. (µg/L)	% RSD[a]	m/z	Avg. Conc. (µg/L)	% RSD[a]
59	< 5.06E+00	n/a	**134**	< 5.06E+00	n/a	**180**	< 5.06E+00	n/a
84	< 5.06E+00	n/a	**135**	7.41E+02	0.11%	**181**	< 5.06E+00	n/a
85	1.01E+03	2.55%	**136**	< 5.06E+00	n/a	**182**	6.15E+01	1.21%
86	< 5.06E+00	n/a	**137**	1.74E+03	0.04%	**183**	3.38E+01	2.04%
87	2.12E+03	2.39%	**138**	2.39E+01	10.41%	**184**	7.27E+01	0.78%
88	< 5.06E+01	n/a	**139**	< 5.06E+00	n/a	**185**	< 5.06E+00	n/a
89	< 5.06E+00	n/a	**140**	< 5.06E+00	n/a	**186**	6.79E+01	0.23%
90	< 5.06E+00	n/a	**141**	< 5.06E+00	n/a	**187**	< 5.06E+00	n/a
91	< 5.06E+00	n/a	**142**	< 5.06E+00	n/a	**188**	< 5.06E+00	n/a
92	8.86E+01	0.23%	**143**	< 5.06E+00	n/a	**189**	< 5.06E+00	n/a
93	< 5.06E+00	n/a	**144**	< 5.06E+00	n/a	**191**	< 5.06E+00	n/a
94	5.85E+01	2.06%	**145**	< 5.06E+00	n/a	**193**	< 5.06E+00	n/a
95	4.90E+03	0.26%	**146**	< 5.06E+00	n/a	**194**	< 5.06E+00	n/a
96	1.01E+02	0.15%	**147**	< 5.06E+00	n/a	**195**	< 5.06E+00	n/a
97	4.46E+03	0.63%	**148**	< 5.06E+00	n/a	**196**	< 5.06E+00	n/a
98	4.50E+03	0.35%	**149**	< 5.06E+00	n/a	**198**	1.67E+02	0.13%
99	2.94E+03	0.41%	**150**	< 5.06E+00	n/a	**203**	< 5.06E+00	n/a
100	4.67E+03	0.32%	**151**	< 5.06E+00	n/a	**204**	9.94E+01	1.27%
101	2.55E+02	0.36%	**152**	< 5.06E+00	n/a	**205**	< 5.06E+00	n/a
102	2.26E+02	0.58%	**153**	< 5.06E+00	n/a	**206**	2.00E+02	1.59%
103	6.60E+02	0.16%	**154**	< 5.06E+00	n/a	**207**	1.74E+02	1.97%
104	1.09E+02	2.21%	**155**	< 5.06E+00	n/a	**208**	4.23E+02	1.30%
105	< 5.06E+00	n/a	**156**	< 5.06E+00	n/a	**229**	< 5.06E+00	n/a
106	< 5.06E+00	n/a	**157**	< 5.06E+00	n/a	**230**	< 5.06E+00	n/a
107	< 1.26E+01	n/a	**158**	< 5.06E+00	n/a	**232**	< 5.06E+00	n/a
108	< 5.06E+00	n/a	**159**	< 5.06E+00	n/a	**233**	< 5.06E+00	n/a
109	7.73E+00[b]	n/a	**160**	< 5.06E+00	n/a	**234**	< 5.06E+00	n/a
110	< 5.06E+00	n/a	**161**	< 5.06E+00	n/a	**235**	7.55E+00	0.91%
111	< 5.06E+00	n/a	**162**	< 5.06E+00	n/a	**236**	< 5.06E+00	n/a
112	9.36E+00	8.52%	**163**	< 5.06E+00	n/a	**237**	< 5.06E+00	n/a
113	< 5.06E+00	n/a	**164**	< 5.06E+00	n/a	**238**	2.08E+02	1.62%
114	9.00E+00	6.32%	**165**	< 5.06E+00	n/a	**239**	< 5.06E+00	n/a
116	1.26E+02	0.53%	**166**	< 5.06E+00	n/a	**240**	< 5.06E+00	n/a
117	9.04E+01	0.31%	**167**	< 5.06E+00	n/a	**241**	< 5.06E+00	n/a
118	2.64E+02	1.15%	**168**	< 5.06E+00	n/a	**242**	< 5.06E+00	n/a
119	7.92E+01	0.71%	**169**	< 5.06E+00	n/a	**243**	< 5.06E+00	n/a
120	3.48E+02	1.09%	**170**	< 5.06E+00	n/a	**244**	< 5.06E+00	n/a
121	< 5.06E+00	n/a	**171**	< 5.06E+00	n/a	**245**	< 5.06E+00	n/a
122	8.69E+01	2.84%	**172**	< 5.06E+00	n/a	**246**	< 5.06E+00	n/a
123	< 5.06E+00	n/a	**173**	< 5.06E+00	n/a	**247**	< 5.06E+00	n/a
124	1.35E+02	1.59%	**174**	< 5.06E+00	n/a	**248**	< 5.06E+00	n/a
125	< 5.06E+00	n/a	**175**	< 5.06E+00	n/a	**249**	< 5.06E+00	n/a
126	4.34E+02	0.03%	**176**	< 5.06E+00	n/a	**250**	< 5.06E+00	n/a
128	< 5.06E+00	n/a	**177**	< 5.06E+00	n/a	**251**	< 5.06E+00	n/a
130	< 5.06E+00	n/a	**178**	< 5.06E+00	n/a	**252**	< 5.06E+00	n/a
133	8.01E+03	0.16%	**179**	< 5.06E+00	n/a			

[a]The %RSD is based on the standard deviation of duplicate samples. The reported method uncertainty is 20% at two sigma. [b]Single result above the method detection limit. The duplicate sample had a reported concentration of < 5.08 µg/L.

Total alpha and beta activities in the samples as measured by LSC are provided in Table 3-16. The LSC counting was performed both with and without Cs removal to obtain a lower detection limit for the alpha. Both sets of values are reported below.

Table 3-16. Alpha and Beta Activity in Tank 9H Batch 1B Samples

	HTF-09-21-41 Original (dpm/mL)	%RSD[a]	HTF-09-21-42 Filtrate (dpm/mL)	%RSD[a]	HTF-09-21-42 Slurry (dpm/mL)	%RSD[a]
Alpha count	< 8.05E+04	n/a	< 3.12E+05	n/a	< 1.83E+06	n/a
Beta count	1.44E+08	0.67	4.56E+08	0.44	3.58E+08	0.29
Cs-Removed Results						
Alpha count	< 1.37E+04	n/a	< 1.29E+04	n/a	< 1.63E+06	n/a
Beta count	3.58E+05	9.91	2.95E+05	3.29	1.62E+07	1.26

[a]The %RSD is based on the standard deviation of duplicate samples. The reported method uncertainty is 20% at one sigma.

Using the alpha activity measured in the digested slurry sample, a maximum inhalation dose potential (IDP) can be calculated assuming all of the alpha activity is attributed to ^{239}Pu, which has the highest Inhalation Dose Conversion Factor (IDCF).[11] The IDCF for ^{239}Pu is 1.9E+08 rem/Ci.[11] Using this value and the alpha activity measured in the digested slurry sample after Cs removal, the IDP is < 5.29E+05 rem/gal.

Based on the low concentrations of elements typically present predominately in sludge as well as the low alpha activity measured in the digested slurry, it appears that the solids present in HTF-09-21-42 contain only a small fraction of sludge solids.

4.0 Conclusions

SRNL received and characterized samples from two batches of dissolved salt in Tank 9H. For each batch both a surface and variable depth sample were collected. In both batches solids were observed in the depth sample; however, the amount of solids was much more significant in the depth sample received from Batch 1B where the insoluble solids represented 10.14 wt% of the sample. Those solids were determined to be primarily aluminum containing solids, with only a small fraction (0.22 wt%) being sludge solids.

In terms of chemistry and cesium concentrations, the samples received from Batch 1A were more concentrated salt solutions than Batch 1B, with sodium concentrations of 8.53 and 8.57 M for the surface and VDS, respectively. The sodium concentrations in Batch 1B ranged from 4.27 M for the surface sample to 7.57 M for the depth sample, indicating some stratification within the tank. The ^{137}Cs activity as well as total cesium concentration in the Batch 1A depth sample were approximately double the activity and concentration measured in the filtrate from the Batch 1B depth sample.

5.0 Future Work

Both batches of material have been transferred from Tank 9H to Tank 10H in preparation of the first batch of material to be processed through TCCR 1A. SRNL has since received and characterized samples collected from Tank 10H after the transfers were complete. Results from the characterization of those samples has been reported in an interim memo[12] and will be later documented in a technical report.

6.0 References

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[3] Emails from T. L. Fellinger to K. M. L. Taylor-Pashow, 3/26/21, 5/19/21, and 5/21/21 documented in SRNL Electronic Laboratory Notebook #E7518-00472-02.
[4] L. N. Oji, "Task Technical and Quality Assurance Plan for the Analysis of Tank 9H Salt Solution Supernatant", SRNL-RP-2019-00463, Rev. 0, July 2019.
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21998 and 21999.
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[12] K. M. L. Taylor-Pashow and W. D. King, "Summary of Results from Tank 10H Tank Closure Cesium Removal (TCCR) Batch 1 Preliminary Samples", SRNL-L3100-2021-00022, Rev. 0, July 28, 2021.

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