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Characterization of Tank 11H Samples from Tank Closure Cesium Removal 1A (TCCR 1A) Batch 1

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EXECUTIVE SUMMARY

Savannah River Mission Completion (SRMC) is currently operating the Tank Closure Cesium Removal 1A (TCCR 1A) process to remove ^{137}Cs from tank waste supernate using an ion exchange process. The TCCR 1A unit processes dissolved salt fed from Tank 10H through a series of ion exchange columns containing crystalline silicotitanate (CST, IONSIVTM R9120-B, 30x60) and the effluent is then discharged to Tank 11H. In support of the TCCR 1A program, SRNL analyzed samples taken from Tank 11H (without tank mixing) before, during, and at the completion of TCCR 1A Batch 1 processing. Tank 11H serves as the receipt tank for the filtered and cesium removed product from the TCCR 1A system. Processing of Batch 1 commenced on January 13, 2022 and completed on February 17, 2022, after processing approximately 70,100 gallons. A pre-production sample was collected from the heel remaining in Tank 11H just before Batch 1 processing began. In addition, five interim surface samples were collected from Tank 11H during processing, and both a surface and a variable depth sample (VDS, ~7" from tank bottom) were collected just after processing completed. Analysis of all samples included density and gamma spectroscopy, in addition to a more comprehensive suite of analytes for the pre- and post-production samples. The density of the pre-production sample was the highest of all samples measured and was then observed to decrease for the first two interim samples, followed by becoming fairly consistent for the remainder of the samples (~1.3 g/mL). The density of 1.3 g/mL is similar to the density measured for one of the three Tank 10H qualification samples (HTF-10-21-126). The ^{137}Cs activity was found to decrease as additional decontaminated effluent from the TCCR columns was added to Tank 11H during processing; however, an increase in activity was observed during periods of no processing which can be attributed to leaching of ^{137}Cs from the known solids in Tank 11H. The Cs isotope concentrations in the Tank 11H post-production surface sample were determined to be 99.7-99.8% lower than the concentrations measured in the Tank 10H feed as measured by mass spectrometry.

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LIST OF ABBREVIATIONS

CST	crystalline silicotitanate
ELN	electronic laboratory notebook
IC	ion chromatography
ICP-ES	inductively coupled plasma – emission spectroscopy
ICP-MS	inductively coupled plasma – mass spectrometry
LSC	liquid scintillation counting
M&TE	measurement and test equipment
PMP	polymethylpentene
RSD	relative standard deviation
SaM	Sensing and Metrology
SRMC	Savannah River Mission Completion
SRNL	Savannah River National Laboratory
TCCR	Tank Closure Cesium Removal
TTQAP	Task Technical and Quality Assurance Plan
TTR	Task Technical Request
VDS	variable depth sample

1.0 Introduction

Savannah River Mission Completion (SRMC) is currently operating the Tank Closure Cesium Removal 1A (TCCR 1A) process to remove ^{137}Cs from tank waste supernate using an ion exchange process. The TCCR 1A unit processes dissolved salt fed from Tank 10H through a series of ion exchange columns containing crystalline silicotitanate (CST, IONSIVTM R9120-B, 30x60) and the effluent is then discharged to Tank 11H. In support of the TCCR 1A program, SRNL analyzed samples taken from Tank 11H before, during, and at the completion of TCCR 1A Batch 1 processing. Tank 11H serves as the receipt tank for the filtered and cesium removed product from the TCCR 1A system. Processing of Batch 1 commenced on January 13, 2022 and completed on February 17, 2022, after processing approximately 70,100 gallons. A pre-production sample was collected from the heel remaining in Tank 11H just before Batch 1 processing began. In addition, five interim surface samples were collected from Tank 11H during processing, and both a surface and a variable depth sample (VDS) were collected just after processing completed.

When processing began, the salt solution was processed through two columns in series. After approximately 6 days processing was paused and a third column was brought online so three columns were used in series. Just after restarting with three columns in series a leak was observed, and therefore the process was shut down. Processing resumed after an outage of ~24 days for repairs, with salt solution being processed through three columns in series. Several of the interim samples were collected during the outage period to examine the leach rate of ^{137}Cs from the heel present in Tank 11H. Immediately after the conclusion of Batch 1 processing a surface sample was collected from Tank 11H (post-production sample). A VDS was also collected approximately 12 days later. A summary of the samples collected from Tank 11H during Batch 1 processing is provided in Table 1-1.

Table 1-1. Summary of Tank 11H Samples Collected During Batch 1 Processing

Sample ID	Date Collected	Location ^a	Notes
HTF-11-22-5	1/8/22	Surface (~7.3")	pre-production sample
HTF-11-22-9	1/17/22	Surface (~20.5")	interim sample
HTF-11-22-10	1/20/22	Surface (~25.3")	interim sample (processing paused after collecting this sample)
HTF-11-22-11	1/30/22	Surface (~25.1")	interim sample (no processing since previous sample)
HTF-11-22-22	2/4/22	Surface (~25.1")	interim sample (no processing since previous sample)
HTF-11-22-23	2/12/22	Surface (~26.5")	interim sample (collected just before restart)
HTF-11-22-27	2/18/22	Surface (~35.7")	post-production surface sample taken several hours after completion of Batch 1 processing
HTF-11-22-29	3/2/22	VDS (~ 7")	post-production variable depth sample (VDS)

^a Approximate location given for the surface samples is the reported tank level on that day. All locations are given as inches from the tank bottom.

2.0 Experimental Procedure

2.1 Tank 11H Pre-Production Sample HTF-11-22-5

A single surface sample (HTF-11-22-5) was collected from Tank 11H with no prior tank mixing on January 8, 2022, before the start-up of TCCR 1A Batch 1 processing. The sample was received at SRNL in an 82-mL dip sample bottle and was placed into the Shielded Cells. The sample was then opened and transferred into a clear polymethylpentene (PMP) beaker for visual observation. The density of the sample was measured in duplicate using 2-mL density tubes and a measurement and test equipment (M&TE) calibrated balance at ambient temperature. Samples used for density measurements were returned to the

sample bottle. Aliquots of the sample were then submitted to Sensing and Metrology (SaM) for a suite of analyses as described in the Task Technical and Quality Assurance Plan (TTQAP).¹

2.2 Tank 11H Interim Samples

A total of 5 interim surface samples were collected from Tank 11H without tank mixing during processing of Batch 1. Each sample was received in an 82-mL dip sample bottle. The first two interim samples (HTF-11-22-9 and HTF-11-22-10) were placed into the Shielded Cells upon receipt, while the remaining three interim samples (HTF-11-22-11, HTF-11-22-22, and HTF-11-22-23) were handled in a radiological hood since dose rates were relatively low. Each sample to be handled in a radiological hood was first repackaged by the Shielded Cells facility prior to moving to the radiological hood where the sample was to be opened. During repackaging of sample HTF-11-22-22 a small amount of liquid was observed within the original bag. Each sample was opened and transferred to a PMP beaker for visual observation. The density of each sample was also measured using an M&TE calibrated balance and 2-mL density tubes at ambient temperature. Aliquots from each interim sample were then submitted to SaM undiluted for gamma spectroscopy analysis. Samples HTF-11-22-9 and HTF-11-22-10 were counted using the standard procedure and count times for gamma spectroscopy. Samples HTF-11-22-11, HTF-11-22-22, and HTF-11-22-23 were counted utilizing a special procedure in an attempt to increase the precision of these analyses.

2.3 Tank 11H Post-Production Samples

Immediately upon the conclusion of processing of Batch 1A, a Tank 11H surface sample (HTF-11-22-25) was collected with no tank mixing and sent to SRNL for analysis. The sample was placed inside of a radiological hood, opened, and was found to be empty. A second surface sample (HTF-11-22-27 in an 82-mL dip sample vial) was collected the same day the empty sample vial was reported (February 18, 2022). Approximately 12 days later a VDS (HTF-11-22-29 in a 200-mL dip sample vial) was collected from the tank at a depth of ~7" from the bottom of the tank and was also sent to SRNL for analysis. Both the surface and the VDS samples were handled in a radiological hood, where they were opened and transferred to PMP beakers for observation. The VDS was found to contain some solids, and therefore a portion of the sample was filtered through a 0.2- μ m cellulose nitrate filter to create a filtrate sample for analysis. Aliquots of the surface sample and the VDS filtrate were submitted to SaM for a suite of analysis as described in the TTQAP.¹ Samples of the as-received VDS containing some suspended solids were submitted to SaM for aqua regia digestion followed by gamma spectroscopy, liquid scintillation counting (LSC), inductively coupled plasma – emission spectroscopy (ICP-ES) and inductively coupled plasma – mass spectrometry (ICP-MS) analyses.

2.4 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.² This work was performed following the applicable TTQAP.¹ The Task Technical Request (TTR) associated with this work³ requested a functional classification of Safety Class (see section 9.5 of the TTQAP entitled "Clarification of Safety Class Functional Classification"). To match the requested functional classification this report and calculations within received a technical review by design verification by document review.⁴ Data are recorded in the Electronic Laboratory Notebook (ELN) system.⁵

3.0 Results and Discussion

All of the Tank 11H surface samples received before, during, and after Batch 1 processing were similar in appearance and did not contain any visible solids. Photographs of two representative samples (HTF-11-22-22 and HTF-11-22-27) are provided in Figure 3-1. In contrast, the VDS post-production sample (HTF-11-22-29) did contain some suspended solids. Photographs of HTF-11-22-29 are provided in Figure 3-2.

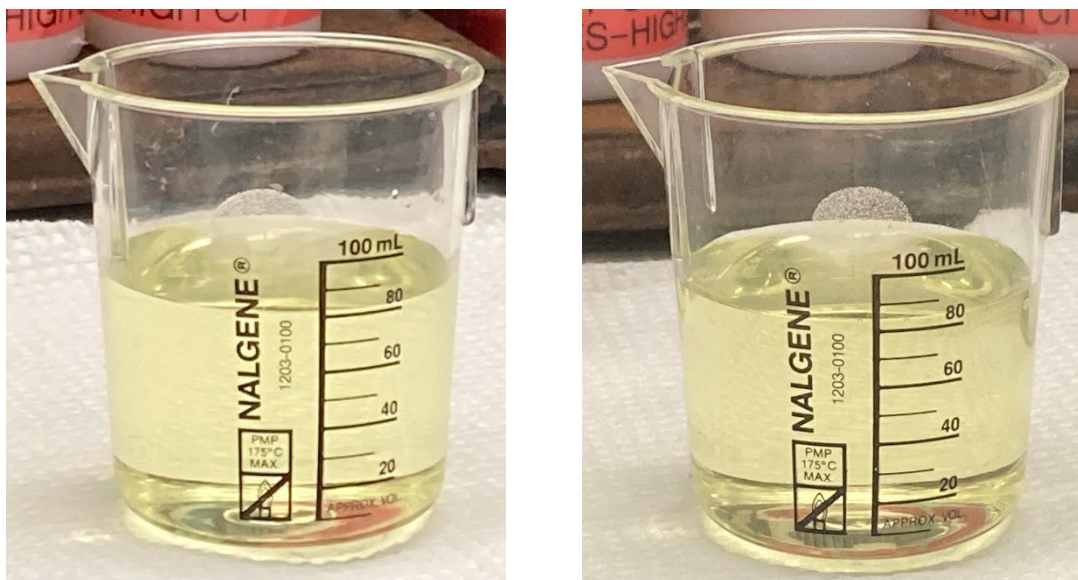


Figure 3-1. Photographs of HTF-11-22-22 (left) and HTF-11-22-27 (right).

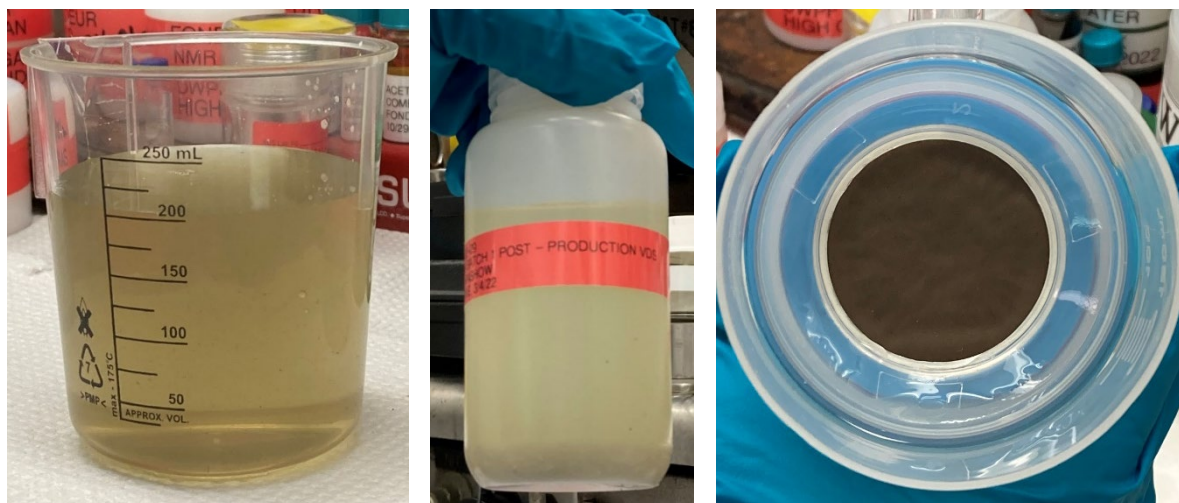


Figure 3-2. Photographs of HTF-11-22-29 with solids suspended (left), after settling for ~3 days (center), and solids collected from filtering approximately 100 mL of sample (right).

A summary of the measured densities for each sample is provided in Table 3-1. The density of the pre-production sample collected on 1/8/22 was slightly higher than the reported density for the most recent corrosion control sample analyzed in June 2021, which had a reported density of 1.311 g/mL (0.243 %RSD).⁶ The density decreased for the first two interim samples (HTF-11-22-9 and HTF-11-22-10), and then remained fairly consistent for the remaining samples, around 1.3 g/mL. The as-received post-production VDS (HTF-11-22-29) had a slightly higher density (1.319 g/mL) compared to the filtrate sample (1.300 g/mL), indicative of the presence of higher density solids suspended in the sample. For comparison the average density for the three Tank 10H Batch 1 qualification samples was 1.35 g/mL; however, the values ranged from 1.302 g/mL to 1.384 g/mL.⁷ Sample HTF-10-21-126, which was reported as a Tank 10H VDS, but is believed to be a surface sample based on density, had a density of 1.302 g/mL, consistent with the densities observed for the Tank 11H samples.

Table 3-1. Densities of Tank 11H Batch 1 Samples

Sample ID	Sample Type	Density (g/mL)	%RSD ^a	Temp. During Density Measurement
HTF-11-22-5	surface(as-received)	1.327	0.0266%	15 °C
HTF-11-22-9	surface(as-received)	1.312	1.05%	15 °C
HTF-11-22-10	surface(as-received)	1.297	0.681%	14 °C
HTF-11-22-11	surface(as-received)	1.299	0.109%	21 °C
HTF-11-22-22	surface(as-received)	1.300	0.0800%	19 °C
HTF-11-22-23	surface(as-received)	1.304	0.298%	23 °C
HTF-11-22-27	surface(as-received)	1.297	0.0273%	23 °C
HTF-11-22-29	VDS (as-received)	1.319	0.107%	15 °C
HTF-11-22-29	VDS (filtrate)	1.300	0.163%	24 °C

^a %RSD is the percent relative standard deviation of replicate measurements.

All Tank 11H samples were submitted for gamma spectroscopy in duplicate along with a blank (deionized water) to check for potential cross contamination during sample preparation. All samples were submitted for analysis undiluted since dose rates were relatively low. As described above, samples from the pre-production and first two interim samples were prepared in the Shielded Cells and were counted using the standard gamma spectroscopy methods. Later interim samples and the post-production samples were prepared in a radiological hood. Each of the duplicate aliquots submitted from the later interim samples and the post-production surface sample were counted in triplicate using a method to obtain a higher precision result. The post-production VDS sample was counted using the standard procedure. A summary of the ¹³⁷Cs activities in the Tank 11H samples is provided in Table 3-2. As can be seen from the reported activities, the ¹³⁷Cs activity in Tank 11H decreased as expected upon the addition of decontaminated effluent from the TCCR 1A unit, as can be seen by the decreasing trend in the first two interim samples when compared to the pre-production sample. Due to the known heel present in Tank 11H leaching occurs over time causing an increase in the ¹³⁷Cs activity during the period where no processing was occurring. Over the ~24 day period between samples HTF-11-22-10 and HTF-11-22-23 where no processing was occurring, the ¹³⁷Cs activity was observed to increase by ~50 – 70%. Initial analysis of duplicate samples of HTF-11-22-23 (interim sample collected just prior to Batch 1 restart) resulted in a reported activity of 5.14E+05 dpm/mL. Upon request from SRMC, this sample was reanalyzed. The original aliquots submitted for analysis were reprepared and recounted, resulting an activity about 10% higher than originally measured. In addition, new aliquots were prepared and submitted, and results from analysis of the new aliquots resulted in a ¹³⁷Cs activity ~15% higher than the original result. It was noted during preparation of the samples for gamma counting which includes acidification of the samples, bubbles were observed possibly indicating the presence of fine suspended solids that were dissolving. After processing of the final portion of the batch, the ¹³⁷Cs activity again decreased due to the addition of decontaminated effluent. The post-production surface sample had the lowest activity measured across all samples. A significant increase was seen from the post-production surface sample to the post-production VDS, which is likely due to the sample being collected near the original level in the tank prior to processing of Batch 1, and the time elapsed (~12 days) between collection of the surface sample and the VDS resulting in additional leaching from the heel. For comparison the Tank 10H Batch 1 qualification samples had an average ¹³⁷Cs activity of 2.37E+08 dpm/mL (ranged from 1.85E+08 to 2.65E+08 dpm/mL).⁷

Table 3-2. ^{137}Cs Activities in Tank 11H Batch 1 Samples

Sample ID	^{137}Cs (dpm/mL)	%RSD ^a	Notes
HTF-11-22-5	1.91E+06	2.60%	Pre-production sample
HTF-11-22-9	3.82E+05	0.370%	Interim sample collected after ~4 days of processing.
HTF-11-22-10	3.49E+05	0.405%	Interim sample collected after ~3 additional days of processing. Processing paused after this sample.
HTF-11-22-11	3.82E+05	2.29%	No processing since previous sample.
HTF-11-22-22	4.34E+05	3.87%	No processing since previous sample.
HTF-11-22-23	5.14E+05	0.346%	No processing since previous sample. Collected just before processing resumed.
HTF-11-22-23	5.67E+05	2.24%	Reprep and recount of previous HTF-11-22-23 samples.
HTF-11-22-23	5.93E+05	2.03%	Analysis of new aliquots from original HTF-11-22-23 sample.
HTF-11-22-27	3.36E+05	1.30%	Post-production surface sample. Collected after ~5 days of additional processing.
HTF-11-22-29-F	9.10E+05	0.622%	Post-production VDS filtrate. Collected ~12 days after processing concluded.
HTF-11-22-29-S	8.83E+05	3.27%	Post-production VDS digested slurry sample.

^a %RSD is the percent relative standard deviation of replicate measurements.

The anion concentrations were measured in the pre-production sample as well as the post-production surface and VDS filtrate samples. A summary of the anion concentrations in these three samples is provided in Table 3-3. There were slight differences observed between the surface and VDS samples collected at the end of processing Batch 1, but the major anions (aluminate, hydroxide, nitrate, nitrite, and carbonate) were within 15% when comparing the surface and VDS concentrations. The largest differences between the surface and VDS concentrations were for sulfate which was 39% higher in the VDS, phosphate which was at least 79% higher in the VDS, and oxalate which was at least 290% higher in the VDS. The changes between the pre-production and post-production samples (including decreased hydroxide and sulfate and increased nitrate in the post-production samples) were consistent with the addition of the Batch 1 composition to Tank 11H.⁷

Table 3-3. Anion and Carbon Results for the Pre- and Post-Batch 1 Processing Tank 11H Samples

Analyte	HTF-11-22-5 Pre-Production	%RSD ^a	HTF-11-22-27 Post-Production Surface	%RSD ^a	HTF-11-22-29 Post-Production VDS Filtrate	%RSD ^a
$\text{Al}(\text{OH})_4^-$ (M) ^b	0.135	0.389%	0.0921	0.854%	0.105	2.75%
Free OH^- (M)	2.43	0.00% ^c	0.702	0.806%	0.784	1.35%
NO_3^- (M)	1.88	0.607%	3.01	0.379%	3.10	0.737%
SO_4^{2-} (M)	0.279	0.00% ^c	0.103	0.143%	0.143	0.514%
NO_2^- (M)	0.390	0.394%	0.422	0.00% ^c	0.435	0.707%
Br^- (M)	< 6.26E-03	n/a	< 6.26E-03	n/a	< 6.26E-03	n/a
$\text{C}_2\text{O}_4^{2-}$ (M)	5.15E-03	0.156%	< 1.14E-03	n/a	4.47E-03	0.898%
F^- (M)	< 5.26E-03	n/a	< 5.26E-03	n/a	< 5.26E-03	n/a
Cl^- (M)	8.56E-03	0.233%	< 2.82E-03	n/a	2.92E-03	0.683%
CHO_2^- (M)	< 2.22E-03	n/a	< 2.22E-03	n/a	< 2.22E-03	n/a
PO_4^{3-} (M)	< 1.05E-03	n/a	< 1.05E-03	n/a	1.88E-03	1.19%
CO_3^{2-} (M)	0.774	0.457%	0.742	0.159%	0.799	2.21%
TOC (mg/L)	199	3.21%	< 94.4	n/a	139	3.05%

^a %RSD is the percent relative standard deviation of replicate measurements. ^b Based on aluminum concentration measured by ICP-ES (Tables 3-4 and 3-5). ^c Duplicate samples gave identical results.

The results of the ICP-ES analysis of the pre-production sample as well as the post-production surface sample are provided in Table 3-4, and the ICP-ES results of the post-production VDS filtrate and digested slurry samples are provided in Table 3-5. The pre-production sample had a sodium concentration of 6.66 M and an Al concentration of 0.135 M along with minor amounts of several other elements including Cr, Fe, Mo, and Ni. The P and S concentrations were consistent with the phosphate and sulfate concentrations measured by ion chromatography (IC). The post-production surface sample had a Na concentration of 6.11 M and an Al concentration of 0.092 M. The post-production sample contained minor amounts of Cr, Mo, and Nb, but no Fe or Ni above instrument detection limits. The Na concentration in the VDS filtrate sample was 6.4% higher than the surface (within analytical uncertainty). The only element that showed a significant change between the filtrate and digested slurry in the VDS was Fe indicating that the solids were Fe containing.

Table 3-4. ICP-ES Results for the Pre- and Post-Batch 1 Tank 11H Surface Samples

Element	HTF-11-22-5 Pre-Production Avg. Conc. (mg/L)	%RSD ^a	HTF-11-22-27 Post-Production Avg. Conc. (mg/L)	%RSD ^a
Ag	< 0.165	n/a	< 0.165	n/a
Al	3640	0.389%	2485	0.854%
B	< 0.683	n/a	< 1.22	n/a
Ba	< 0.135	n/a	< 0.114	n/a
Be	< 0.103	n/a	< 0.103	n/a
Ca	< 1.89	n/a	< 0.936	n/a
Cd	< 0.232	n/a	< 0.135	n/a
Ce	< 4.2	n/a	< 2.66	n/a
Co	< 0.542	n/a	< 0.295	n/a
Cr	9.39	1.36%	19.6	0.722%
Cu	< 1.15	n/a	< 0.702	n/a
Fe	3.00	0.236%	< 2.04	n/a
Gd	< 0.447	n/a	< 0.371	n/a
K	< 94.4	n/a	< 311	n/a
La	< 0.270	n/a	< 0.220	n/a
Li	< 3.70	n/a	< 3.70	n/a
Mg	< 0.481	n/a	< 0.481	n/a
Mn	< 0.063	n/a	< 0.096	n/a
Mo	3.16	0.895%	10.4	0.00% ^b
Na	153000	0.00% ^b	140500	0.503%
Nb	NM	NM	10.2	0.00% ^b
Ni	< 0.520	n/a	< 0.466	n/a
P	32.7	1.73%	75.9	0.466%
Pb	< 7.52	n/a	< 3.74	n/a
S	8570	0.825%	3390	0.00% ^b
Sb	< 2.52	n/a	< 1.98	n/a
Si	< 14.6	n/a	< 17.9	n/a
Sn	< 8.13	n/a	< 8.13	n/a
Sr	< 0.14	n/a	< 0.140	n/a
Th	< 6.210	n/a	< 1.99	n/a
Ti	< 1.42	n/a	< 1.42	n/a
U	< 5.16	n/a	< 5.16	n/a
V	< 1.95	n/a	< 1.95	n/a
Zn	< 0.437	n/a	< 0.041	n/a
Zr	< 0.966	n/a	< 1.85	n/a

^a %RSD is the percent relative standard deviation of replicate measurements. ^b Duplicate samples gave identical results.

Table 3-5. ICP-ES Results for the Post-Batch 1 Tank 11H VDS (Filtrate and Slurry)

Element	HTF-11-22-29 Filtrate Avg. Conc. (mg/L)	%RSD ^a	HTF-11-22-29 Slurry Avg. Conc. (mg/L)	%RSD ^a	% Difference ^b
Ag	< 0.165	n/a	< 0.166	n/a	n/a
Al	2830	2.75%	2910	0.321%	2.95%
B	< 1.27	n/a	< 2.45	n/a	n/a
Ba	< 0.135	n/a	< 0.458	n/a	n/a
Be	< 0.103	n/a	< 0.416	n/a	n/a
Ca	< 3.86	n/a	< 1.87	n/a	n/a
Cd	< 0.232	n/a	< 0.416	n/a	n/a
Ce	< 2.66	n/a	< 2.68	n/a	n/a
Co	< 0.295	n/a	< 0.297	n/a	n/a
Cr	17.5	2.42%	19.9	0.470%	13.4%
Cu	< 0.346	n/a	< 0.708	n/a	n/a
Fe	< 1.04	n/a	5.32	2.81%	> 411%
Gd	< 0.371	n/a	< 0.450	n/a	n/a
K	< 311	n/a	< 245	n/a	n/a
La	< 0.274	n/a	< 0.280	n/a	n/a
Li	< 3.70	n/a	< 8.88	n/a	n/a
Mg	< 0.481	n/a	< 0.484	n/a	n/a
Mn	< 0.290	n/a	< 1.17	n/a	n/a
Mo	9.30	1.98%	9.90	0.848%	6.44%
Na	150000	2.36%	154000	1.82%	2.78%
Nb	6.30	4.04%	< 7.68	n/a	n/a
Ni	< 0.466	n/a	< 2.10	n/a	n/a
P	68.1	2.70%	71.7	0.130%	5.27%
Pb	< 3.74	n/a	< 5.63	n/a	n/a
S	4340	2.12%	4620	1.41%	6.65%
Sb	< 1.98	n/a	< 2.55	n/a	n/a
Si	< 13.9	n/a	< 16.2	n/a	n/a
Sn	< 8.13	n/a	< 12.6	n/a	n/a
Sr	< 0.140	n/a	< 0.255	n/a	n/a
Th	< 1.99	n/a	< 2.69	n/a	n/a
Ti	< 1.42	n/a	< 2.49	n/a	n/a
U	< 5.16	n/a	< 5.20	n/a	n/a
V	< 1.95	n/a	< 2.52	n/a	n/a
Zn	< 0.336	n/a	< 0.416	n/a	n/a
Zr	< 0.912	n/a	< 1.97	n/a	n/a

^a %RSD is the percent relative standard deviation of replicate measurements. ^b The % Difference is defined here as (VDS-surface)/surface x 100%.

The ICP-MS results for the pre- and post-production Tank 11H samples are provided in Tables 3-6 through 3-9. Table 3-6 provides the composition of the pre-production sample, Table 3-7 provides the composition of the post-production surface sample, and Tables 3-8 and 3-9 provide the composition of the post-production VDS filtrate and digested slurry, respectively. When comparing the post-production surface sample (Table 3-7) to the Tank 10H Batch 1 qualification samples,⁷ there were several expected changes. Significant increases in concentration of the isotopes from 90 to 94 were observed, and these are attributed to the expected Zr and Nb leaching from the CST in the columns. Similarly, an increase was observed for the Hf isotopes from 176 to 180 in the post-production sample. Hf is a known contaminant in the Zr binder. An increase of ~50-220% was observed for m/z values of 196, 198, and 204 over the Tank 10H feed concentration (varied with the three qualification sample compositions). These isotopes are likely Hg, presumably leaching from the Tank 11H heel. The concentrations of m/z 196, 198, and 204 were highest

in the pre-production sample (HTF-11-22-5). The major Hg isotopes between 199 and 203 are not reported by ICP-MS. The Cs isotope concentrations were measured to be 99.7-99.8% lower than measured in the Tank 10H feed. The Rb (m/z 85 and 87) concentrations ranged from 15% to 40% lower than the Tank 10H Batch 1 feed and the Sr (m/z 88) ranged from 41 to 56% lower depending on which of the three qualification samples it was compared to. The Pb isotopes (m/z 206, 207, and 208) ranged from 60-77% lower than the Tank 10H Batch 1 samples. The minor uranium isotopes (235-236) showed a decrease in concentration on the order of 70-90% from the Tank 10H Batch 1 feed, however, ^{238}U showed varying change due to the varying concentrations of ^{238}U measured in the Tank 10H samples. When compared to HTF-10-21-125, the ^{238}U concentration was 57% lower in the Tank 11H surface sample; however, when compared to HTF-10-21-126 the ^{238}U was 3% higher (same within analytical uncertainty) in the Tank 11H sample. The ^{237}Np concentration in the Tank 11H surface sample was ~80-90% lower than the Tank 10H feed indicating significant retention by the CST.

Comparison of the post-production surface sample (Table 3-7) to the post-production VDS filtrate (Table 3-8) showed changes in several elements. Specifically, m/z 88 and 89 (Sr and Y) were 79% and 68% higher, respectively, in the VDS filtrate when compared to the surface sample. The Cs isotopes also measured higher in the VDS when compared to the surface sample (96% higher for m/z 133, 56% higher for m/z 135, and 64% higher for m/z 137). In contrast the major Ba isotope (m/z 138) showed a decrease of 70%. The Pb isotopes (m/z 206-208) were 540-600% higher in the VDS filtrate when compared to the surface sample. The U isotopes (m/z 234-236, 238) increased from 68-87% in the VDS filtrate compared to the surface sample; while the other actinides (m/z 237 and 239) were 140% and 120% higher, respectively.

Finally, comparison of the VDS digested slurry (Table 3-9) to the filtrate (Table 3-8) showed increases in a number of elements in the VDS. The most significant changes (>200% increase) were seen for the following masses: m/z 89 (Y, 460%); m/z 111 (Cd, 260%); m/z 130 (Te/Ba, 1000%); m/z 137 (Cs/Ba, 260%); m/z 138 (Ba, 4700%); m/z 139 (La, 2200%); m/z 140 (Ce, >4400%); m/z 141 (Pr, 1800%); m/z 142 (Ce/Nd, >3500%); m/z 143 (Nd, 1500%); m/z 144 (Nd/Sm, 1600%); m/z 145 (Nd, 1400%); m/z 146 (Nd, 1500%); m/z 147 (Sm, >1100%); m/z 148 (Sm/Nd, >1100%); m/z 150 (Sm/Nd, >1000%); m/z 196 (Hg, 340%); m/z 198 (Hg, 480%); m/z 204 (Hg, 420%); m/z 232 (Th, >22,000%); and m/z 238 (U, 390%).

Table 3-10 provides a comparison of the Cs isotopic ratios based on the ICP-MS results for the Tank 11H pre-production and post-production samples. The Cs concentrations reported in Table 3-10 are after subtraction of the mass attributed to Ba in these samples (based upon the m/z 138 concentration). Comparison of the pre-production and post-production samples showed a shift toward higher ^{133}Cs fraction and lower ^{137}Cs fraction for the post-production samples. The post-production surface sample and VDS filtrate had similar distributions. In comparison to the Tank 10H Batch 1 qualification samples,⁷ the ^{133}Cs isotopic fraction was higher in the Tank 11H samples (average of 77% in the Tank 10H samples). The ^{137}Cs isotopic fraction in the pre-production Tank 11H sample was similar to the Tank 10H isotopic fraction (average of 15.6% in the Tank 10H samples); however, in the post-production samples the ^{137}Cs isotopic fraction was several percent lower. Due to the relatively low concentration of Cs in the Tank 11H samples there may be more uncertainty in these values as compared to measurements made on the Tank 10H samples where Cs is much higher in concentration.

Table 3-6. ICP-MS Results for the Pre-Batch 1 Tank 11H Sample (HTF-11-22-5)

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	< 1.00E+00	n/a	134	< 1.00E+00	n/a	180	1.49E+02	0.597%
84	< 1.00E+00	n/a	135	3.82E+00	4.65%	181	1.43E+01	1.74%
85	1.49E+02	1.58%	136	< 1.00E+00	n/a	182	1.74E+01	0.0645%
86	1.89E+00	1.24%	137	1.12E+01	2.82%	183	9.68E+00	1.39%
87	2.98E+02	1.05%	138	1.69E+01	3.62%	184	2.06E+01	1.74%
88	4.67E+01	2.06%	139	1.17E+01	1.56%	185	< 1.00E+00	n/a
89	1.22E+01	8.56%	140	< 2.00E+01	n/a	186	1.90E+01	0.0399%
90	4.54E+02	0.459%	141	1.10E+01	6.71%	187	< 1.00E+00	n/a
91	1.06E+02	0.986%	142	< 2.00E+01	n/a	188	< 1.00E+00	n/a
92	1.66E+02	0.633%	143	1.10E+01	12.0%	189	1.21E+00	8.84%
93	1.68E+04	0.734%	144	1.18E+01	7.68%	191	< 1.00E+00	n/a
94	1.62E+02	0.563%	145	9.02E+00	7.71%	193	1.26E+00	5.70%
95	9.95E+02	0.0544%	146	6.46E+00	11.5%	194	2.54E+00	3.11%
96	6.70E+01	0.584%	147	4.28E+00	1.45%	195	1.75E+00	6.31%
97	9.24E+02	0.362%	148	4.06E+00	14.5%	196	9.08E+01	2.89%
98	9.22E+02	1.11%	149	< 1.00E+00	n/a	198	6.16E+03	2.48%
99	6.88E+02	0.329%	150	3.73E+00	0.230%	203	< 1.00E+00	n/a
100	9.72E+02	0.584%	151	< 1.00E+00	n/a	204	3.21E+03	2.08%
101	3.08E+02	0.122%	152	< 2.00E+00	n/a	205	< 1.00E+00	n/a
102	2.67E+02	0.247%	153	< 1.00E+00	n/a	206	1.54E+03	0.927%
103	4.36E+02	0.856%	154	< 1.00E+00	n/a	207	1.37E+03	2.20%
104	1.89E+02	0.352%	155	< 1.00E+00	n/a	208	3.27E+03	1.28%
105	2.65E+02	0.288%	156	1.01E+00 ^b	n/a	229	< 1.00E+00	n/a
106	2.17E+02	0.453%	157	< 1.00E+00	n/a	230	< 1.00E+00	n/a
107	8.74E+01	0.356%	158	1.07E+00 ^b	n/a	232	5.59E+01	18.8%
108	3.55E+01	0.266%	159	< 1.00E+00	n/a	233	1.95E+00	5.15%
109	2.60E+01	2.68%	160	< 1.00E+00	n/a	234	3.52E+00	2.12%
110	3.34E+01	0.656%	161	< 1.00E+00	n/a	235	3.73E+01	1.46%
111	2.54E+01	0.282%	162	< 1.00E+00	n/a	236	5.65E+00	2.92%
112	4.65E+01	0.548%	163	< 1.00E+00	n/a	237	3.72E+00	2.48%
113	2.37E+01	0.732%	164	< 1.00E+00	n/a	238	2.71E+03	2.69%
114	5.54E+01	1.98%	165	< 1.00E+00	n/a	239	7.13E+00	1.77%
116	2.95E+01	4.45%	166	< 1.00E+00	n/a	240	1.07E+00 ^b	n/a
117	1.14E+01	1.26%	167	< 1.00E+00	n/a	241	< 1.00E+00	n/a
118	3.10E+01	1.26%	168	< 1.00E+00	n/a	242	< 1.00E+00	n/a
119	4.09E+01	0.631%	169	< 1.00E+00	n/a	243	< 1.00E+00	n/a
120	4.08E+01	0.365%	170	< 1.00E+00	n/a	244	< 1.00E+00	n/a
121	1.21E+00	10.4%	171	< 1.00E+00	n/a	245	< 1.00E+00	n/a
122	1.16E+01	2.30%	172	< 1.00E+00	n/a	246	< 1.00E+00	n/a
123	< 1.00E+00	n/a	173	< 1.00E+00	n/a	247	< 1.00E+00	n/a
124	1.83E+01	1.89%	174	< 1.00E+00	n/a	248	< 1.50E+00	n/a
125	< 1.00E+00	n/a	175	< 1.00E+00	n/a	249	< 1.00E+00	n/a
126	7.01E+01	0.789%	176	2.22E+01	1.96%	250	< 1.00E+00	n/a
128	< 1.00E+00	n/a	177	7.85E+01	2.63%	251	< 1.00E+00	n/a
130	2.85E+00	4.90%	178	1.16E+02	0.740%			
133	4.42E+01	3.94%	179	5.80E+01	1.16%			

^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 < 1.00E+00 µg/L.

Table 3-7. ICP-MS Results for the Post-Batch 1 Tank 11H Surface Sample (HTF-11-22-27)

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	< 1.00E+00	n/a	134	< 1.00E+00	n/a	180	1.10E+02	1.23%
84	< 1.00E+00	n/a	135	1.01E+00 ^c	n/a	181	9.29E+00	1.40%
85	4.65E+02	1.63%	136	< 1.00E+00	n/a	182	3.41E+01	1.66%
86	< 1.00E+00	0.00%	137	2.37E+00	0.48%	183	1.92E+01	2.35%
87	1.03E+03	0.0396%	138	3.40E+00	14.9%	184	4.12E+01	2.29%
88	1.43E+01	0.299%	139	1.02E+00 ^c	n/a	185	< 1.00E+00	n/a
89	2.52E+00	16.5%	140	< 1.00E+00	n/a	186	3.76E+01	2.62%
90	7.31E+02	0.182%	141	1.02E+00	1.40%	187	1.39E+00	8.08%
91	1.73E+02	0.696%	142	< 1.00E+00	n/a	188	< 1.00E+00	n/a
92	2.85E+02	0.239%	143	1.26E+00	0.750%	189	< 1.00E+00	n/a
93	1.16E+04	0.197%	144	1.29E+00	1.22%	191	< 1.00E+00	n/a
94	2.82E+02	0.595%	145	1.00E+00 ^c	n/a	193	< 1.00E+00	n/a
95	2.93E+03	0.00% ^b	146	< 1.00E+00	n/a	194	2.31E+00	3.71%
96	1.00E+02	0.297%	147	< 1.00E+00	n/a	195	1.88E+00	2.23%
97	2.83E+03	0.268%	148	< 1.00E+00	n/a	196	1.72E+01	1.57%
98	2.76E+03	0.145%	149	< 1.00E+00	n/a	198	9.62E+02	0.333%
99	1.96E+03	0.0523%	150	< 1.00E+00	n/a	203	< 1.00E+00	n/a
100	2.97E+03	0.382%	151	< 1.00E+00	n/a	204	4.67E+02	0.194%
101	2.02E+02	2.96%	152	< 1.00E+00	n/a	205	< 1.00E+00	n/a
102	1.73E+02	2.22%	153	< 1.00E+00	n/a	206	6.67E+01	0.256%
103	3.95E+02	0.184%	154	< 1.00E+00	n/a	207	5.95E+01	1.77%
104	1.13E+02	0.914%	155	< 1.00E+00	n/a	208	1.43E+02	0.202%
105	1.15E+02	0.775%	156	< 1.00E+00	n/a	229	< 1.00E+00	n/a
106	9.50E+01	0.760%	157	< 1.00E+00	n/a	230	< 1.00E+00	n/a
107	3.60E+01	0.619%	158	< 1.00E+00	n/a	232	< 1.00E+00	n/a
108	1.89E+01	3.92%	159	< 1.00E+00	n/a	233	< 1.00E+00	n/a
109	2.01E+01	0.565%	160	< 1.00E+00	n/a	234	< 1.00E+00	n/a
110	9.38E+00	3.69%	161	< 1.00E+00	n/a	235	6.80E+00	0.231%
111	1.77E+00	0.173%	162	< 1.00E+00	n/a	236	1.12E+00	3.65%
112	5.63E+00	1.41%	163	< 1.00E+00	n/a	237	1.34E+00	1.80%
113	< 2.00E+00	n/a	164	< 1.00E+00	n/a	238	3.09E+02	3.15%
114	5.53E+00	3.34%	165	< 1.00E+00	n/a	239	2.34E+00	2.75%
116	7.03E+01	0.474%	166	< 1.00E+00	n/a	240	< 1.00E+00	n/a
117	4.85E+01	0.578%	167	< 1.00E+00	n/a	241	< 1.00E+00	n/a
118	1.37E+02	0.120%	168	< 1.00E+00	n/a	242	< 1.00E+00	n/a
119	4.89E+01	0.138%	169	< 1.00E+00	n/a	243	< 1.00E+00	n/a
120	1.86E+02	0.181%	170	< 1.00E+00	n/a	244	< 1.00E+00	n/a
121	< 1.00E+00	n/a	171	< 1.00E+00	n/a	245	< 1.00E+00	n/a
122	5.01E+01	1.57%	172	< 1.00E+00	n/a	246	< 1.00E+00	n/a
123	< 1.00E+00	n/a	173	< 1.00E+00	n/a	247	< 1.00E+00	n/a
124	7.70E+01	0.0741%	174	< 1.00E+00	n/a	248	< 1.00E+00	n/a
125	< 1.00E+00	n/a	175	< 1.00E+00	n/a	249	< 1.00E+00	n/a
126	2.44E+02	0.594%	176	1.64E+01	1.24%	250	< 1.00E+00	n/a
128	< 1.00E+00	n/a	177	5.67E+01	1.29%	251	< 1.00E+00	n/a
130	< 1.00E+00	n/a	178	8.52E+01	1.28%			
133	1.29E+01	10.1%	179	4.24E+01	0.563%			

^a The %RSD is based on the standard deviation of duplicate samples. The reported method uncertainty is 20% at two sigma. ^b Duplicate samples gave identical results. ^c Single result above the method detection limit. The duplicate sample had a reported concentration of < 1.00E+00 µg/L.

Table 3-8. ICP-MS Results for the Post-Batch 1 Tank 11H VDS Filtrate (HTF-11-22-29)

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	< 1.00E+00	n/a	134	< 1.00E+00	n/a	180	6.96E+01	1.20%
84	< 1.00E+00	n/a	135	1.58E+00	9.77%	181	6.34E+00	1.00%
85	4.02E+02	0.287%	136	< 1.00E+00	n/a	182	3.00E+01	0.176%
86	1.11E+00	10.7%	137	3.87E+00	4.53%	183	1.71E+01	1.69%
87	8.86E+02	0.367%	138	1.06E+00 ^b	n/a	184	3.60E+01	0.845%
88	2.55E+01	0.422%	139	1.65E+00	2.35%	185	< 1.00E+00	n/a
89	4.23E+00	1.34%	140	< 1.00E+00	n/a	186	3.27E+01	1.41%
90	3.48E+02	0.155%	141	1.77E+00	3.74%	187	1.30E+00	1.96%
91	8.04E+01	0.878%	142	< 1.00E+00	n/a	188	< 1.00E+00	n/a
92	1.57E+02	0.675%	143	2.11E+00	2.74%	189	< 1.00E+00	n/a
93	7.62E+03	3.06%	144	2.20E+00	2.49%	191	< 1.00E+00	n/a
94	1.47E+02	1.28%	145	1.60E+00	1.22%	193	< 1.00E+00	n/a
95	2.68E+03	2.60%	146	1.25E+00	3.82%	194	1.97E+00	17.0%
96	7.77E+01	0.124%	147	< 1.00E+00	n/a	195	1.61E+00	9.16%
97	2.58E+03	2.71%	148	< 1.00E+00	n/a	196	7.30E+00	1.41%
98	2.51E+03	3.44%	149	< 1.00E+00	n/a	198	3.47E+02	10.5%
99	1.77E+03	3.56%	150	< 1.00E+00	n/a	203	< 1.00E+00	n/a
100	2.71E+03	3.76%	151	< 1.00E+00	n/a	204	1.94E+02	8.16%
101	2.02E+02	1.29%	152	< 1.00E+00	n/a	205	< 1.00E+00	n/a
102	1.76E+02	1.59%	153	< 1.00E+00	n/a	206	4.26E+02	1.97%
103	4.12E+02	0.463%	154	< 1.00E+00	n/a	207	3.89E+02	1.52%
104	1.16E+02	0.983%	155	< 1.00E+00	n/a	208	1.01E+03	2.18%
105	1.26E+02	0.385%	156	< 1.00E+00	n/a	229	< 1.00E+00	n/a
106	1.05E+02	0.594%	157	< 1.00E+00	n/a	230	< 1.00E+00	n/a
107	3.90E+01	1.35%	158	< 1.00E+00	n/a	232	< 1.00E+00	n/a
108	1.92E+01	0.238%	159	< 1.00E+00	n/a	233	< 1.00E+00	n/a
109	1.29E+01	1.21%	160	< 1.00E+00	n/a	234	1.79E+00	3.06%
110	8.74E+00	3.46%	161	< 1.00E+00	n/a	235	1.17E+01	0.745%
111	1.17E+00	3.96%	162	< 1.00E+00	n/a	236	1.89E+00	1.83%
112	4.17E+00	0.129%	163	< 1.00E+00	n/a	237	3.22E+00	4.83%
113	< 2.00E+00	n/a	164	< 1.00E+00	n/a	238	5.77E+02	1.03%
114	4.30E+00	0.0297%	165	< 1.00E+00	n/a	239	5.21E+00	0.178%
116	6.29E+01	0.712%	166	< 1.00E+00	n/a	240	< 1.00E+00	n/a
117	4.38E+01	0.480%	167	< 1.00E+00	n/a	241	< 1.00E+00	n/a
118	1.22E+02	0.181%	168	< 1.00E+00	n/a	242	< 1.00E+00	n/a
119	4.77E+01	0.392%	169	< 1.00E+00	n/a	243	< 1.00E+00	n/a
120	1.66E+02	0.371%	170	< 1.00E+00	n/a	244	< 1.00E+00	n/a
121	< 1.00E+00	n/a	171	< 1.00E+00	n/a	245	< 1.00E+00	n/a
122	4.48E+01	0.295%	172	< 1.00E+00	n/a	246	< 1.00E+00	n/a
123	< 1.00E+00	n/a	173	< 1.00E+00	n/a	247	< 1.00E+00	n/a
124	6.93E+01	0.360%	174	< 1.00E+00	n/a	248	< 1.00E+00	n/a
125	< 1.00E+00	n/a	175	< 1.00E+00	n/a	249	< 1.00E+00	n/a
126	2.22E+02	0.341%	176	1.06E+01	2.11%	250	< 1.00E+00	n/a
128	< 1.00E+00	n/a	177	3.61E+01	1.52%	251	< 1.00E+00	n/a
130	< 1.00E+00	n/a	178	5.41E+01	0.372%			
133	2.52E+01	1.58%	179	2.72E+01	1.58%			

^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 < 1.00E+00 µg/L.

Table 3-9. ICP-MS Results for the Post-Batch 1 Tank 11H VDS Digested Slurry (HTF-11-22-29)

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	< 4.03E+00	n/a	134	< 4.03E+00	n/a	180	9.36E+01	1.06%
84	< 4.03E+00	n/a	135	< 4.03E+00	n/a	181	9.93E+00	7.41%
85	3.88E+02	1.59%	136	< 4.03E+00	n/a	182	3.24E+01	0.318%
86	< 4.03E+00	n/a	137	1.41E+01	3.75%	183	1.83E+01	2.19%
87	8.59E+02	0.174%	138	4.92E+01	0.350%	184	3.88E+01	0.505%
88	4.42E+01	3.05%	139	3.82E+01	1.07%	185	< 4.03E+00	n/a
89	2.37E+01	1.24%	140	4.57E+01	4.13%	186	3.59E+01	3.02%
90	5.70E+02	0.342%	141	3.37E+01	1.19%	187	< 4.03E+00	n/a
91	1.47E+02	0.0207%	142	3.68E+01	2.02%	188	< 4.03E+00	n/a
92	2.41E+02	0.302%	143	3.28E+01	1.42%	189	< 4.03E+00	n/a
93	9.56E+03	0.155%	144	3.66E+01	1.29%	191	< 4.03E+00	n/a
94	2.40E+02	0.312%	145	2.43E+01	1.35%	193	< 8.06E+00	n/a
95	2.74E+03	0.565%	146	1.94E+01	1.71%	194	< 4.03E+00	n/a
96	1.12E+02	0.386%	147	1.19E+01	1.50%	195	< 4.03E+00	n/a
97	2.64E+03	1.13%	148	1.20E+01	3.63%	196	3.18E+01	6.60%
98	2.61E+03	1.36%	149	< 4.03E+00	n/a	198	2.01E+03	0.658%
99	1.73E+03	0.535%	150	1.08E+01	0.14%	203	< 4.03E+00	n/a
100	2.71E+03	0.995%	151	< 4.03E+00	n/a	204	1.00E+03	1.40%
101	2.46E+02	0.289%	152	< 4.03E+00	n/a	205	< 4.03E+00	n/a
102	2.17E+02	1.49%	153	< 4.03E+00	n/a	206	8.29E+02	1.54%
103	4.55E+02	0.602%	154	< 4.03E+00	n/a	207	7.48E+02	2.44%
104	1.41E+02	2.32%	155	< 4.03E+00	n/a	208	1.80E+03	2.08%
105	1.47E+02	1.70%	156	< 4.03E+00	n/a	229	< 4.03E+00	n/a
106	1.17E+02	1.21%	157	< 4.03E+00	n/a	230	< 4.03E+00	n/a
107	< 8.06E+01	n/a	158	< 4.03E+00	n/a	232	2.30E+02	0.0892%
108	1.37E+01	4.89%	159	< 4.03E+00	n/a	233	< 4.03E+00	n/a
109	< 8.06E+01	n/a	160	< 4.03E+00	n/a	234	< 4.03E+00	n/a
110	8.20E+00	2.41%	161	< 4.03E+00	n/a	235	3.46E+01	0.866%
111	4.25E+00 ^b	n/a	162	< 4.03E+00	n/a	236	4.80E+00	3.90%
112	9.97E+00	1.20%	163	< 4.03E+00	n/a	237	5.19E+00	5.05%
113	< 4.03E+00	n/a	164	< 4.03E+00	n/a	238	2.81E+03	0.558%
114	1.08E+01	3.43%	165	< 4.03E+00	n/a	239	1.16E+01	3.02%
116	7.28E+01	1.01%	166	< 4.03E+00	n/a	240	< 4.03E+00	n/a
117	4.51E+01	0.0439%	167	< 4.03E+00	n/a	241	< 4.03E+00	n/a
118	1.29E+02	2.95%	168	< 4.03E+00	n/a	242	< 4.03E+00	n/a
119	8.10E+01	1.19%	169	< 4.03E+00	n/a	243	< 4.03E+00	n/a
120	1.75E+02	3.35%	170	< 4.03E+00	n/a	244	< 4.03E+00	n/a
121	< 4.03E+00	n/a	171	< 4.03E+00	n/a	245	< 4.03E+00	n/a
122	4.70E+01	2.12%	172	< 4.03E+00	n/a	246	< 4.03E+00	n/a
123	< 4.03E+00	n/a	173	< 4.03E+00	n/a	247	< 4.03E+00	n/a
124	7.17E+01	0.203%	174	< 4.03E+00	n/a	248	< 8.06E+00	n/a
125	< 4.03E+00	n/a	175	< 4.03E+00	n/a	249	< 4.03E+00	n/a
126	2.32E+02	1.11%	176	1.40E+01	3.64%	250	< 4.03E+00	n/a
128	< 4.03E+00	n/a	177	4.81E+01	3.67%	251	< 4.03E+00	n/a
130	1.11E+01	7.10%	178	7.08E+01	1.60%			
133	2.05E+01	0.414%	179	3.59E+01	0.896%			

^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 < 4.06E+00 µg/L.

Table 3-10. Cs Isotopes from ICP-MS for the Tank 11H Batch 1 Samples

HTF-11-22-5 (Pre-Production Surface Sample)				
Isotope	Mean Concentration^a (μg/L)	%RSD^b	Isotopic Distribution, Mass %	Mole Fraction
Cs-133	44.2	3.94	80.3	0.807
Cs-134	< 0.430	n/a	< 0.780	< 7.78E-03
Cs-135	2.26	4.65	4.11	0.0407
Cs-137	8.57	2.82	15.6	0.152
HTF-11-22-27 (Post-Production Surface Sample)				
Isotope	Mean Concentration^a (mg/L)	%RSD^b	Isotopic Distribution, Mass %	Mole Fraction
Cs-133	12.9	10.1	83.6	0.839
Cs-134	< 0.885	n/a	< 5.74	< 0.0572
Cs-135	0.700 ^c	n/a	4.54	0.0449
Cs-137	1.84	0.475	11.9	0.116
HTF-11-22-29 (Post-Production VDS Filtrate)				
Isotope	Mean Concentration^a (mg/L)	%RSD^b	Isotopic Distribution, Mass %	Mole Fraction
Cs-133	25.2	1.58	82.9	0.833
Cs-134	< 0.964	n/a	< 3.17	< 0.0316
Cs-135	1.48	9.77	4.87	0.0482
Cs-137	3.71	4.53	12.2	0.119

^a Concentrations after subtracting the mass attributed to Ba. ^b Percent relative standard deviation from duplicate samples. The reported method uncertainty is 20% at two sigma. ^c Single replicate above the method detection limit.

A summary of the other radionuclide results for the Tank 11H pre-production and post-production samples are provided in Tables 3-11 and 3-12, respectively. Comparison of the Tank 11H post-production surface sample activities to the Tank 10H Batch 1 qualification samples⁷ shows an increase in ⁹⁰Sr activity ranging from 210% to 1200% higher than measured in the Tank 10H qualification samples depending on which of the three qualification samples it is compared to. This is indicative of significant leaching of ⁹⁰Sr from the heel present in Tank 11H. Results of comparison of the Pu isotopes also varies widely depending on which of the qualification samples it is being compared to. When compared to HTF-10-21-125, all Pu isotopes were lower in the Tank 11H post-Batch 1 surface sample, ranging from 76% lower for ²³⁸Pu to 12% lower for ^{239/240}Pu. When compared to HTF-10-21-126 ²³⁸Pu was 54% lower in the Tank 11H surface sample, while the activity of ^{239/240}Pu was 58% higher. A similar trend was seen for the comparison to HTF-10-21-127 where ²³⁸Pu was 32% lower in the Tank 11H surface sample, but ^{239/240}Pu was 200% higher. Removal of Pu by the CST in the TCCR columns is expected based on the teabag loading data;⁷ however, the removal is not expected to be isotope dependent as all isotopes are in the same chemical form. Therefore, the variation seen in the isotope activities is most likely due to differing isotopic compositions in the Tank 10H feed and Tank 11H heel which is also known to be leaching. The results indicate the residual Tank 11H material is enriched in ^{239/240}Pu compared to ²³⁸Pu. Comparison of the Tank 11H post-Batch 1 surface and VDS samples indicates higher activities of all measurable isotopes which would be expected due to leaching of the heel present at the bottom of the tank.

Table 3-11. Radionuclide Activities in Tank 11H Pre-Batch 1 Surface Sample (HTF-11-22-5)

Radionuclide	Avg. (dpm/mL)	%RSD ^a	Avg. Method Uncertainty
⁶⁰ Co	< 1.33E+01	n/a	MDA
⁹⁰ Sr	4.37E+06	16.7%	16.2%
¹⁰⁶ Ru	< 9.02E+01	n/a	MDA
¹²⁵ Sb	< 5.89E+01	n/a	MDA
¹²⁶ Sb	< 1.78E+01	n/a	MDA
¹²⁶ Sn	< 5.44E+01	n/a	MDA
¹⁴⁴ Ce	< 1.23E+02	n/a	MDA
¹⁵⁴ Eu	< 3.42E+01	n/a	MDA
²³⁸ Pu	3.49E+04	1.62%	6.07%
^{239/240} Pu	1.84E+03	1.54%	6.51%
²⁴¹ Pu	6.89E+03	5.65%	15.4%
²⁴¹ Am	< 1.67E+02	n/a	MDA
Alpha Count	< 3.36E+04	n/a	Upper Limit
Beta Count	1.02E+07	6.28%	10.0%

^a The %RSD is based on the standard deviation of duplicate samples.

Table 3-12. Radionuclide Activities in Tank 11H Post-Batch 1 Samples

Radionuclide	HTF-11-22-27 (Surface)			HTF-11-22-29 (VDS)		
	Avg. (dpm/mL)	%RSD ^a	Avg. Method Uncertainty	Avg. (dpm/mL)	%RSD ^a	Avg. Method Uncertainty
⁶⁰ Co	< 1.13E+01	n/a	MDA	< 3.33E+01	n/a	MDA
⁹⁰ Sr	1.11E+06	9.60%	19.4%	2.21E+06	14.4%	23.0%
¹⁰⁶ Ru	< 1.37E+02	n/a	MDA	< 1.78E+02	n/a	MDA
¹²⁵ Sb	< 7.79E+01	n/a	MDA	< 1.47E+02	n/a	MDA
¹²⁶ Sb	8.96E+02	2.53%	5.00%	< 2.94E+02	n/a	MDA
¹²⁶ Sn	8.96E+02	2.53%	5.00%	< 5.85E+01	n/a	MDA
¹⁴⁴ Ce	< 3.83E+02	n/a	MDA	< 2.27E+02	n/a	MDA
¹⁵⁴ Eu	< 4.67E+01	n/a	MDA	< 5.82E+01	n/a	MDA
²³⁸ Pu	1.04E+04	5.94%	6.01%	2.70E+04	3.94%	7.62%
^{239/240} Pu	5.60E+02	7.71%	6.79%	1.41E+03	1.51%	8.02%
²⁴¹ Pu	2.18E+03	2.93%	15.4%	5.26E+03	7.26%	16.0%
²⁴¹ Am	< 3.60E+02	n/a	MDA	< 1.08E+02	n/a	MDA
Alpha Count	< 2.70E+04	n/a	MDA	< 2.70E+04	n/a	MDA
Beta Count	3.57E+06	0.198%	10.4%	5.40E+06	2.23%	10.3%
Alpha Count				< 3.41E+05 ^b	n/a	MDA
Beta Count				2.95E+07 ^b	0.290%	10.6%

^a The %RSD is based on the standard deviation of duplicate samples. ^b Results for the digested slurry, all other results are for the filtrate.

4.0 Conclusions

A total of eight samples from Tank 11H collected before, during, and after processing of Batch 1 through TCCR 1A have been analyzed. An initial pre-production sample was collected from the tank prior to processing and was characterized for a suite of analyses. Five surface samples were collected at various times during the processing of Batch 1, including periods where processing was paused for repairs and there were no additions being made to the tank between samples. The density and gamma activity of each of the interim samples were determined. Finally, both a surface and a variable depth sample were collected from the tank at the conclusion of processing (VDS collected ~12 days after surface sample) and were analyzed

for a suite of analytes. The density of the pre-production sample was the highest of all samples measured and beginning with the second interim sample the densities were fairly consistent for the remainder of the samples (~1.3 g/mL). The ^{137}Cs activity was found to decrease as additional decontaminated effluent from the TCCR columns was added to Tank 11H during processing; however, an increase in activity was observed during periods of no processing which is attributed to leaching of ^{137}Cs from the known solids in Tank 11H. The Cs isotope concentrations in the Tank 11H post-production surface sample were determined to be 99.7-99.8% lower than the concentrations measured in the Tank 10H feed as measured by ICP-MS.

5.0 References

¹ K. M. L. Taylor-Pashow and W. D. King, “Task Technical and Quality Assurance Plan for Analysis of Tank 10H and Tank 11H Samples in Support of Tank Closure Cesium Removal (TCCR) 1A”, SRNL-RP-2021-04002, Rev. 0, July 2021.

² “Savannah River National Laboratory Technical Report Design Check Guidelines” WSRC-IM-2002-00011, Rev. 2, August 2004.

³ J. B. Goldblatt and K. R. Miklia, “TCCR Sampling for Tank 10 Post-Recirculation, Tank 11 Production for Batch Qualification, and DWPF Waste Qualification”, X-TTR-H-00119, Rev. 0, June 2021.

⁴ Savannah River Site Manual E7 “Conduct of Engineering”, Procedure 2.60 Rev. 20 “Technical Reviews”, November 9, 2021.

⁵ SRNL Electronic Laboratory Notebook #E7518-00472-07.

⁶ Corrosion control sample results report for HTF-11-21-43, LW-AD-PROJ-210525-5, LIMS sample ID 22249.

⁷ K. M. L. Taylor-Pashow, T. Hang, “Summary of Results from Batch 1 Qualification Samples for Tank Closure Cesium Removal 1A (TCCR 1A)”, SRNL-STI-2022-00058, Rev. 0, March 2022.

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