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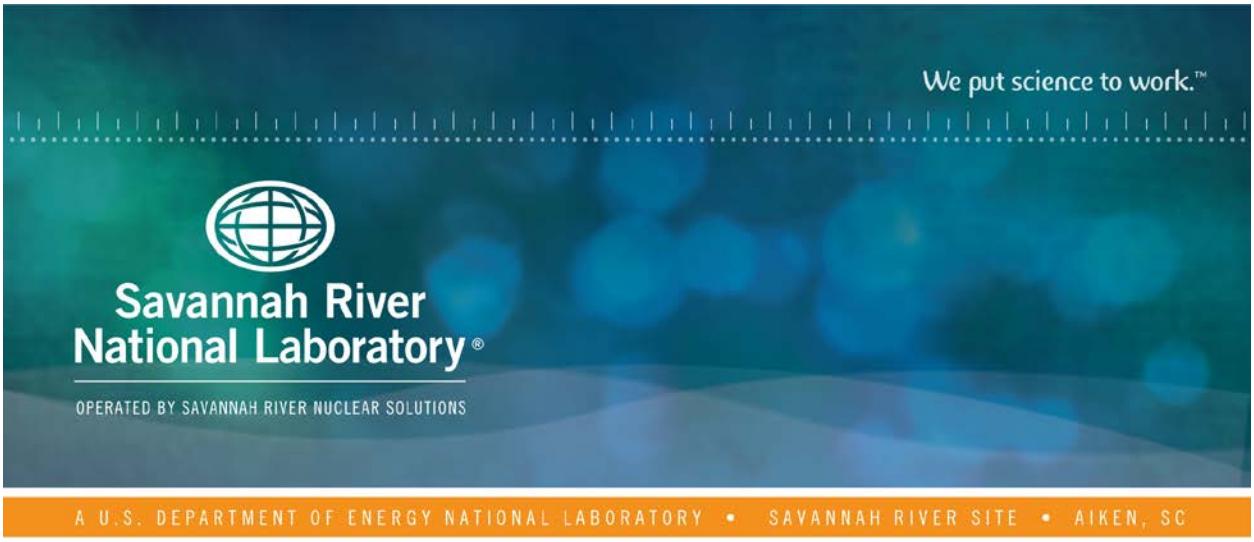

Summary of Expedited Results from Samples Supporting Tank Closure Cesium Removal (TCCR) Batch 3

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July 2020

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EXECUTIVE SUMMARY

Savannah River Remediation (SRR) is currently operating the Tank Closure Cesium Removal (TCCR) process to remove ^{137}Cs from tank waste supernate using an ion exchange process. As part of that process, Savannah River National Laboratory (SRNL) receives and analyzes samples in support of the qualification of each batch to be processed. SRNL recently received supernate samples retrieved from Tank 10H as well as in-tank batch contact samples for characterization in support of qualifying Batch 3 for processing through the TCCR unit.

SRNL received and characterized a set of dip samples collected from Tank 10H (one surface and one variable depth sample). No solids were observed in either sample and therefore the samples were combined, and a suite of analyses were performed on the composite sample. The density of the combined sample was 1.174 g/mL (0.18 %RSD), which was consistent with the measured sodium concentration of 3.54 M (0.08 %RSD). The ^{137}Cs activity in the sample was 4.75E+07 dpm/mL (4.28 %RSD) and the total Cs concentration was calculated to be 1.54 mg/L (1.16E-05 M).

The in-tank batch contact samples consist of 0.1 g of crystalline silicotitanate (CST) contained within a teabag device. Duplicate samples were submerged in Tank 10H supernate for a period of about 13 days, after which time they were retrieved and transferred to SRNL for analysis. At SRNL the CST was rinsed to remove excess salt solution and the CST was then air dried before being digested for analysis. Results of the analysis indicated a ^{137}Cs loading of 3.66E+10 ± 1.40E+09 dpm/g or 16.5 ± 0.63 Ci/kg_{CST}. This value represents a bounding upper limit as it includes the addition of two sigma uncertainty from replicate analysis of the individual teabag samples as well as the addition of the small amount of ^{137}Cs activity measured in the rinse solutions. The above values are based on the air-dried mass of CST. Correcting to the true dry mass using a F-factor of 0.8191 results in a maximum loading of 20.2 ± 0.77 Ci/kg_{CST}.

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LIST OF ABBREVIATIONS

AD	Analytical Development
CST	crystalline silicotitanate
ELN	Electronic Laboratory Notebook
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
SRNL	Savannah River National Laboratory
TCCR	Tank Closure Cesium Removal
TTQAP	Task Technical and Quality Assurance Plan
TTR	Technical Task Request
ZAM	ZAM (Zheng, Anthony, and Miller) Isotherm Model

1.0 Introduction

In support of the Tank Closure Cesium Removal (TCCR) program, SRNL analyzed several samples from Tank 10H, including the in-tank crystalline silicotitanate (CST) batch contact equilibrium (or "teabag") samples deployed in that tank. Tank 10H serves dual functions as both the salt dissolution tank as well as the feed tank for the TCCR system. Prior to operation of TCCR, Tank 10H must undergo dissolution campaigns, dissolving the salt cake to form an aqueous salt solution (supernate). After completion of Batch 2 processing, hydrolancing was performed in Tank 10H to aid salt dissolution. The first hydrolancing was performed in July 2019 adding 1,626 gallons of water. Following hydrolancing, the contents of the tank was recirculated for approximately one week. Following this period of recirculation ~50,000 gallons of domestic water were added in two ~25,000-gallon additions in late July 2019. The contents were then recirculated via the transfer pump with return lines to three different riser locations in the tank for a period of ~8 days after which time the submersible transfer pump failed and had to be replaced. A second hydrolancing was performed in December 2019. Recirculation of the tank contents began again on March 10, 2020. Recirculation was stopped and an intermediate sample (HTF-10-20-18) was removed from the tank on March 16, 2020 and sent to SRNL for characterization. Recirculation resumed after the sample was removed from the tank and continued on and off until April 1, 2020 when recirculation was stopped for another water addition. Based on results of the intermediate sample an additional ~24,000 gallons of domestic water were added to the tank on April 1, 2020. Recirculation resumed on April 2, 2020 and continued on and off until April 9, 2020 when it was suspended for a period due to the reduction in on-site work due to COVID-19. A 33-hour recirculation was performed during this interim period starting on May 4, 2020. Recirculation resumed again on May 30, 2020 and a second intermediate sample was collected from the tank on June 3, 2020 (HTF-10-20-51). An additional ~20,000 gallons of domestic water was then added to the tank on June 9, 2020, followed by recirculation. On June 14, 2020 recirculation was stopped and the following day two samples, one surface and one variable depth (HTF-10-20-52 and -53) were collected from the tank and sent to SRNL for characterization. The following day the CST batch contact equilibrium test vials were lowered into the tank for a 13-day contact. After the 13-day contact, the vials (HTF-10-20-54 and -55) were retrieved and sent to SRNL for analysis.

Analytical results from key components of the Tank 10H intermediate, qualification, and in-tank batch equilibrium samples are summarized below. Results from the remaining analyses described in the Task Technical and Quality Assurance Plan (TTQAP)[1] will be documented in a future report.

2.0 Experimental Procedure

2.1 Tank 10H Intermediate Surface Samples (HTF-10-20-18 and HTF-10-20-51)

A single 82-mL dip sample was received from Tank 10H on March 16, 2020 (HTF-10-20-18). The sample was placed into the Shielded Cells on March 18, 2020. The sample was then opened and transferred to a clear polymethylpentene (PMP) beaker for observation. No solids were observed in the sample. A photograph of the sample is provided in Figure 3-1. The density of the sample was measured using a measurement and test equipment (M&TE) balance in duplicate using 2-mL density tubes at ambient temperature (22 °C). Samples used for density measurements were returned to the sample bottle. Aliquots of the sample were then diluted with 3 M nitric acid and submitted to Analytical Development (AD) for gamma spectroscopy and inductively coupled plasma – emission spectroscopy (ICP-ES) analyses as requested by the customer.[2]

A second 82-mL dip sample was received from Tank 10H on June 3, 2020 (HTF-10-20-51). The sample was placed into the Shielded Cells the following day. The sample was then opened and transferred to a clear PMP beaker for observation. No solids were observed in the sample. The density of the sample was measured using an M&TE balance in triplicate using 2-mL density tubes at ambient temperature (27 °C).

Samples used for density measurements were returned to the sample bottle. Aliquots of the sample were diluted with 3 M nitric acid and were submitted to AD for gamma spectroscopy as requested by the SRR customer.[3]

2.2 Tank 10H Batch 3 Qualification Samples (HTF-10-20-52 and HTF-10-20-53)

Two 200-mL dip samples were received from Tank 10H on June 15, 2020. One sample was collected from the surface and the other sample was collected from a depth of 70 inches. The samples were placed into the Shielded Cells the following day. The samples were then opened and transferred to clear PMP beakers for observation. No solids were observed in either sample. Photographs of the samples are provided in Figure 3-2. The samples were combined, and the density was measured in duplicate using 2-mL density tubes at ambient temperature (26 °C). Samples used for density measurements were returned to the sample bottle. Following the density measurements, aliquots of the sample were diluted with either deionized water or 3 M nitric acid and were submitted to AD for a suite of analysis as described in the TTQAP.[1]

2.3 Tank 10H In-Tank Batch Contact Samples (HTF-10-20-54 and HTF-10-20-55)

Two modified sample vials containing the CST teabags which had been suspended in Tank 10H supernate for a total of ~13 days were received at SRNL on June 29, 2020. The teabags were removed from the sample vials in the Shielded Cells and were processed according to the established procedure.[4] Duplicate aliquots of the CST standard were processed alongside the pair of teabag samples and were submitted for identical analyses. The CST standard is from the same batch of pre-treated CST that was used in the teabags. After completion of air drying (~2 days), the CST was weighed and subjected to hot HF-HNO_3 digestion. Aliquots of the digestion solutions from the teabag samples and the standard samples were then submitted to AD for analysis. The gamma spectroscopy and inductively coupled plasma – mass spectrometry (ICP-MS) results are reported below, and the complete set of analytical results will be documented in a future report. In addition to the digested CST results, samples of the soak solutions generated during processing of the teabags were also submitted for analysis and the gamma spectroscopy results from those samples are also reported here.

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.[5] This work was performed following the applicable TTQAP.[1] The Task Technical Request (TTR) associated with this work[6] requested a functional classification of Safety Significant (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.[7] Data are recorded in the Electronic Laboratory Notebook (ELN) system.[8]

3.0 Results and Discussion

3.1 Tank 10H Intermediate Samples

A photograph of the first Tank 10H Batch 3 intermediate sample is provided in Figure 3-1. The second intermediate sample was similar in appearance. Both samples appeared clear and colorless, with no evidence of significant solids.

Figure 3-1. Photograph of Tank 10H Intermediate Sample HTF-10-20-18.

The densities of the Tank 10H intermediate surface samples are reported in Table 3-1. The density of the second intermediate sample was slightly lower than the first, due to dilution from additional water added to the tank between the two sample retrievals; however, the density of the second intermediate sample is higher than would be expected from pure dilution with water indicating additional salt was also dissolved from the tank.

Table 3-1. Densities of Tank 10H Batch 3 Intermediate Samples

Sample	Date Sample Collected	Mean Density (g/mL)	%RSD	Temperature During Measurement
HTF-10-20-18	March 16, 2020	1.228	0.58	22 °C
HTF-10-20-51	June 3, 2020	1.214	0.53	27 °C

The ^{137}Cs activity of both intermediate samples was measured by gamma spectroscopy and the results are provided in Table 3-2. Similar to the density measurements, the ^{137}Cs activity was slightly lower in the second sample when compared to the first, consistent with additional dilution from the added water.

Table 3-2. ^{137}Cs Activities in the Tank 10H Batch 3 Intermediate Samples

Sample	Date Sample Collected	^{137}Cs Activity (dpm/mL)	%RSD
HTF-10-20-18	March 16, 2020	5.67E+07	2.11
HTF-10-20-51	June 3, 2020	5.23E+07	5.68

The first intermediate sample (HTF-10-20-18) was also submitted for ICP-ES analysis and those results are provided in Table 3-3. The sodium concentration in this sample measured 4.62 M. The only other elements present above the detection limit were Al (present as $Al(OH)_4^-$) with a concentration of 0.041 M and S (present as SO_4^{2-}) with a concentration of 0.35 M.

Table 3-3. ICP-ES Results for the Tank 10H Batch 3 First Intermediate Sample (HTF-10-20-18)

Element	Average Concentration (mg/L)	%RSD[a]
Ag	< 2.76	n/a
Al	1106	0.89
B	< 74.4	n/a
Ba	< 1.58	n/a
Be	< 0.401	n/a
Ca	< 2.30	n/a
Cd	< 3.91	n/a
Ce	< 27.1	n/a
Co	< 6.52	n/a
Cr	< 9.2	n/a
Cu	< 8.20	n/a
Fe	< 3.10	n/a
Gd	< 5.98	n/a
K	< 184	n/a
La	< 3.12	n/a
Li	< 11.0	n/a
Mg	< 0.215	n/a
Mn	< 0.722	n/a
Mo	< 11.6	n/a
Na	106170	0.77
Ni	< 21.3	n/a
P	< 72.2	n/a
Pb	< 31.2	n/a
S	11205	5.49
Sb	< 56.3	n/a
Si	< 44.2	n/a
Sn	< 43.2	n/a
Sr	< 0.369	n/a
Th	< 37.5	n/a
Ti	< 1.26	n/a
U	< 68.7	n/a
V	< 2.59	n/a
Zn	< 1.88	n/a
Zr	< 1.82	n/a

[a]The %RSD is based on the standard deviation of duplicate samples. The reported analytical method uncertainties (at two sigma) are 10% for Al and Na and 15% for S.

3.2 Tank 10H Batch 3 Qualification Samples

Photographs of the surface and variable depth Tank 10H Batch 3 qualification samples collected on June 15, 2020 are provided in Figure 3-2. Both samples appeared clear and colorless, with no evidence of solids. The two samples were combined for analysis. The density of the combined sample measured 1.174 g/mL (0.18 %RSD) at 26 °C. The measured density indicates minimal salt dissolution occurred after the final water addition of ~20,000 gallons was made on June 9, 2020.

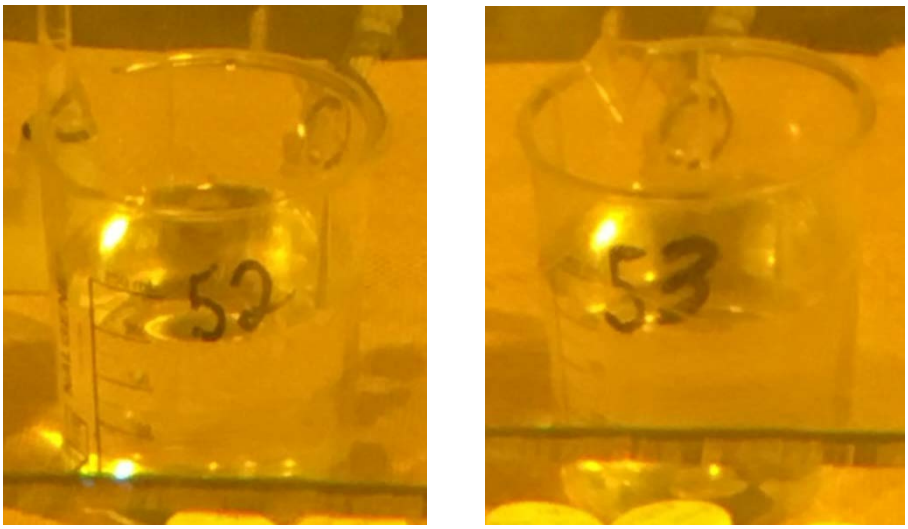

Figure 3-2. Photographs of Tank 10H Qualification Samples HTF-10-20-52 (left) and HTF-10-20-53 (right).

The ^{137}Cs activity in the sample measured 4.75E+07 dpm/mL (4.28 %RSD). This is equivalent to 0.246 mg/L ^{137}Cs. Based on the ICP-MS results (see Table 3-4) the isotopic abundance of ^{137}Cs in this sample is 15.9%; therefore, the total Cs in the sample is calculated to be 1.54 mg/L (1.16E-05 M). This isotopic abundance of ^{137}Cs is lower than seen in the previous TCCR batch (16.8% in Batch 2).[9] The full suite of ICP-MS results is provided in Table 3-9.

Table 3-4. Cs Isotopes from ICP-MS for the Composite Tank 10H Batch 3 Qualification Sample (HTF-10-20-52 and -53)

Isotope	Mean Concentration (mg/L)	%RSD[a]	Isotopic Distribution, Mass %	Mole Fraction
Cs-133	8.69E-01	0.41	76.4	0.768
Cs-134	< 1.17E-02	n/a	< 1.02	< 0.010
Cs-135	8.78E-02	0.56	7.71	0.076
Cs-137	1.81E-01	1.80	15.9	0.156

[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 composite Tank 10H Batch 3 qualification sample as measured by liquid scintillation counting (LSC) are shown in Table 3-5. 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 in the table. A summary of other radionuclide activities in the sample is provided in Table 3-6.

Table 3-5. Alpha and Beta Activity in the Composite Tank 10H Batch 3 Qualification Sample (HTF-10-20-52 and -53)

	Cs Removed (dpm/mL)	%RSD[a]	Without Cs Removal (dpm/mL)	%RSD[a]
Alpha count	< 3.09E+04[b,c]	n/a	< 1.81E+05[b,c]	n/a
Beta Count	6.10E+05	6.19	5.80E+07	4.01

[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. [c]Both samples were below the method detection limit and only the lower value is reported.

Table 3-6. Summary of Radionuclides Measured in the Composite Tank 10H Batch 3 Qualification Sample (HTF-10-20-52 and -53)

Radionuclide	Average Activity[a] (dpm/mL)	%RSD[b]	Average Reported Analytical Uncertainty
^{60}Co	< 4.08E+01	n/a	MDA
^{90}Sr	3.19E+05	18.8	14.0
^{99}Tc	1.66E+04	5.87	6.19
^{106}Ru	< 2.82E+02	n/a	MDA
^{125}Sb	< 1.71E+01	n/a	MDA
^{126}Sb	1.38E+02	9.24	6.21
^{126}Sn	1.38E+02	9.24	6.21
^{129}I	6.62E+00	6.62	8.71
^{135}Cs	2.36E+02	2.67	20.0
^{137}Cs	4.75E+07	4.28	5.00
^{144}Ce	< 4.14E+02	n/a	MDA
^{154}Eu	< 1.20E+04	n/a	MDA
^{238}Pu	3.61E+04	29.8	13.8
$^{239/240}$Pu	5.53E+02	23.7	19.0
^{241}Pu	1.70E+03	29.6	20.6
^{241}Am	< 6.03E+02	n/a	MDA

[a]Mean activity except when both samples were below the method detection limit, and then only the lower value is reported. [b]The %RSD is based on the standard deviation of duplicate samples.

The ICP-ES results for the qualification sample are provided in Table 3-7. The sodium concentration in this sample measured 3.54 M. The anion and carbon analysis results for this sample are provided in Table 3-8. The sample was found to be relatively low in hydroxide and high in nitrate. The anion/cation balance showed a difference of 13% with the anions being high.

Table 3-7. ICP-ES Results for the Composite Tank 10H Batch 3 Qualification Sample (HTF-10-20-52 and -53)

Element	Average Concentration (mg/L)	%RSD[a]
Ag	< 0.50	n/a
Al	933	0.36
B	< 0.71	n/a
Ba	< 0.25	n/a
Be	< 0.02	n/a
Ca	< 0.14	n/a
Cd	< 0.41	n/a
Ce	< 2.06	n/a
Co	< 0.51	n/a
Cr	6.45	3.41
Cu	< 0.13	n/a
Fe	< 0.49	n/a
Gd	< 0.41	n/a
K	68.0	2.07
La	< 0.24	n/a
Li	< 1.25	n/a
Mg	< 0.02	n/a
Mn	< 0.04	n/a
Mo	< 2.87	n/a
Na	81340	0.08
Ni	< 2.53	n/a
P	22.8	3.25
Pb	< 3.48	n/a
S	8464	2.55
Sb	< 10.8	n/a
Si	< 3.54	n/a
Sn	< 3.05	n/a
Sr	< 0.04	n/a
Th	< 1.07	n/a
Ti	< 0.11	n/a
U	< 7.21	n/a
V	< 0.28	n/a
Zn	< 0.54	n/a
Zr	< 0.16	n/a

[a]The %RSD is based on the standard deviation of duplicate samples. The reported analytical method uncertainties (at two sigma) are 10% except for S which had a 20% reported uncertainty.

Table 3-8. Anion and Carbon Results for the Composite Tank 10H Batch 3 Qualification Sample (HTF-10-20-52 and -53).

Analyte	Average Concentration	%RSD[a]
Free OH^- (M)	0.114	3.28
NO_3^- (M)	2.08	1.45
SO_4^{2-} (M)	0.245	0.96
NO_2^- (M)	0.540	1.29
Br^- (M)	< 1.81E-03	n/a
$C_2O_4^{2-}$ (M)	8.34E-03	0.79
F^- (M)	< 1.52E-03	n/a
Cl^- (M)	< 8.17E-04	n/a
CHO_2^- (M)	< 6.43E-04	n/a
PO_4^{3-} (M)	7.56E-04	5.17
CO_3^{2-} [b] (M)	0.380	8.72
TOC[c] (mg/L)	224	4.79

[a]The %RSD is based on the standard deviation of duplicate samples. The reported analytical method 1-sigma uncertainties were 10%. [b]Calculated from total inorganic carbon (TIC) result. [c]Total organic carbon.

Table 3-9. ICP-MS Results (Including As and Se) for the Composite Tank 10H Batch 3 Qualification Sample (HTF-10-20-52 and -53).

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	< 5.83E+00	n/a	128	< 2.91E+00	n/a	177	< 2.91E+00	n/a
As	< 1.46E+01	n/a	130	< 2.91E+01	n/a	178	< 2.91E+00	n/a
Se	< 1.84E+02	n/a	133	8.69E+02	0.41%	179	< 2.91E+00	n/a
82	2.27E+02	0.43%	134	< 1.17E+01	n/a	180	< 2.91E+00	n/a
84	< 5.83E+00	n/a	135	8.78E+01	0.56%	181	< 2.91E+00	n/a
85	1.18E+02	2.53%	136	< 2.91E+00	n/a	182	7.23E+00	0.67%
86	< 2.91E+00	n/a	137	1.81E+02	1.80%	183	4.03E+00	0.05%
87	2.50E+02	0.06%	138	3.07E+00	3.81%	184	8.79E+00	1.18%
88	8.08E+00	1.44%	139	< 2.91E+00	n/a	185	< 2.91E+00	n/a
89	< 5.83E+00	n/a	140	3.32E+00	11.59%	186	8.01E+00	0.68%
90	< 2.91E+00	n/a	141	< 2.91E+00	n/a	187	< 2.91E+00	n/a
91	< 2.91E+00	n/a	142	< 2.91E+00	n/a	191	< 2.91E+00	n/a
92	1.62E+01	3.39%	143	< 2.91E+00	n/a	193	< 2.91E+00	n/a
93	< 2.91E+00	n/a	144	< 2.91E+00	n/a	194	< 2.91E+00	n/a
94	1.10E+01	1.52%	145	< 2.91E+00	n/a	195	< 2.91E+00	n/a
95	6.97E+02	1.36%	146	< 2.91E+00	n/a	196	5.71E+00	8.13%
96	1.99E+01	6.29%	147	< 2.91E+00	n/a	198	2.85E+02	3.47%
97	6.38E+02	0.61%	148	< 2.91E+00	n/a	203	< 2.91E+00	n/a
98	6.32E+02	0.83%	149	< 2.91E+00	n/a	204	1.78E+02	3.89%
99	3.28E+02	0.41%	150	< 2.91E+00	n/a	205	2.94E+00[b]	n/a
100	6.51E+02	0.44%	151	< 2.91E+00	n/a	206	2.80E+01	3.09%
101	1.47E+02	3.29%	152	< 2.91E+00	n/a	207	2.58E+01	0.72%
102	1.27E+02	2.94%	153	< 2.91E+00	n/a	208	6.12E+01	0.05%
103	1.01E+02	1.37%	154	< 2.91E+00	n/a	229	< 2.91E+00	n/a
104	6.92E+01	7.16%	155	< 2.91E+00	n/a	230	< 2.91E+00	n/a
105	1.94E+01	13.3%	156	< 2.91E+00	n/a	232	1.17E+01	5.87%
106	1.61E+01	16.2%	157	< 2.91E+00	n/a	233	7.72E+00	0.48%
107	6.65E+00	7.59%	158	< 2.91E+00	n/a	234	2.20E+03	5.54%
108	< 2.91E+00	n/a	159	< 2.91E+00	n/a	235	5.94E+01	1.03%
109	< 2.91E+00	n/a	160	< 2.91E+00	n/a	236	1.65E+01	1.04%
110	< 5.83E+00	n/a	161	< 2.91E+00	n/a	237	1.37E+01	1.27%
111	< 2.91E+00	n/a	162	< 2.91E+00	n/a	238	3.51E+02	0.09%
112	< 2.91E+00	n/a	163	< 2.91E+00	n/a	239	< 2.91E+00	n/a
113	< 1.46E+01	n/a	164	< 2.91E+00	n/a	240	< 2.91E+00	n/a
114	< 2.91E+00	n/a	165	< 2.91E+00	n/a	241	< 2.91E+00	n/a
116	1.23E+01	5.04%	166	< 2.91E+00	n/a	242	< 2.91E+00	n/a
117	8.99E+00	2.74%	167	< 2.91E+00	n/a	243	< 2.91E+00	n/a
118	2.42E+01	5.21%	168	< 2.91E+00	n/a	244	< 2.91E+00	n/a
119	1.35E+01	11.94%	169	< 2.91E+00	n/a	245	< 2.91E+00	n/a
120	3.32E+01	8.47%	170	< 2.91E+00	n/a	246	< 2.91E+00	n/a
121	< 2.91E+00	n/a	171	< 2.91E+00	n/a	247	< 2.91E+00	n/a
122	9.25E+00	5.78%	172	< 2.91E+00	n/a	248	< 2.91E+00	n/a
123	< 2.91E+00	n/a	173	< 2.91E+00	n/a	249	< 2.91E+00	n/a
124	1.38E+01	3.73%	174	< 2.91E+00	n/a	250	< 2.91E+00	n/a
125	< 2.91E+00	n/a	175	< 2.91E+00	n/a	251	< 2.91E+00	n/a
126	5.58E+01	0.75%	176	< 2.91E+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 as the duplicate was below the method detection limit (< 2.93E+00 µg/L).

3.3 Tank 10H In-Tank Batch Contact Samples (HTF-10-20-54 and HTF-10-20-55)

Duplicate aliquots of the solutions generated from digestion of the CST contained in the teabags and the CST digestion standards, as well as the soak solutions, were submitted for replicate gamma spectroscopy analysis. Soak solutions were all 65 mL in volume. The individual results of these analyses, as well as the average and standard deviations of the replicates are shown in Table 3-10. The activity per gram is calculated based on the air-dried mass of CST that was used for the digestion.

Table 3-10. ^{137}Cs Activity from Gamma Counting for In-Tank Batch Contact Samples, Digestion Standards, and Soak Solutions

	Replicate 1	Replicate 2	Average	St. Dev.	%RSD[a]
Teabag A7	3.66E+10 dpm/g	3.56E+10 dpm/g	3.61E+10 dpm/g	7.07E+08 dpm/g	1.96%
Teabag A8	3.52E+10 dpm/g	3.49E+10 dpm/g	3.51E+10 dpm/g	2.12E+08 dpm/g	0.61%
CST Standard A7	< 3.73E+06 dpm/g	< 4.01E+06 dpm/g	< 3.87E+06 dpm/g	n/a	n/a
CST Standard A8	< 3.30E+06 dpm/g	< 4.43E+06 dpm/g	< 3.87E+06 dpm/g	n/a	n/a
Teabag A7 – 0.01 M NaOH soak	1.64E+05 dpm/mL	1.59E+05 dpm/mL	1.62E+05 dpm/mL	3.54E+03 dpm/mL	2.19%
Teabag A7 – DI Water soak	8.89E+03 dpm/mL	7.60E+03 dpm/mL	8.25E+03 dpm/mL	9.12E+02 dpm/mL	11.1%
Teabag A8 – 0.01 M NaOH soak	2.12E+05 dpm/mL	2.51E+05 dpm/mL	2.32E+05 dpm/mL	2.76E+04 dpm/mL	11.9%
Teabag A8 – DI Water soak	1.50E+04 dpm/mL	1.35E+04 dpm/mL	1.43E+04 dpm/mL	1.06E+03 dpm/mL	7.44%

Units: Cs-137 dpm/g air-dried CST; Cs-137 dpm/mL of soak solution

[a]The %RSD is based on the standard deviation of the replicate samples. The reported method uncertainty is 5% at one sigma for all samples except for the DI water soak samples which had reported method uncertainties ranging between 5.70 and 6.47%.

Two aliquots of the CST standard were digested alongside the CST from the teabags in the Shielded Cells, and solutions from these digestions were analyzed by gamma spectroscopy as well as ICP-MS. The main purpose of these standards is to confirm complete digestion of the CST; however, they also serve as a blank to detect contamination of the samples during handling and digestion in the Shielded Cells. As can be seen in Table 3-10, both standards had ^{137}Cs activities below the method detection limit and, therefore, indicate no significant level of contamination that would have affected the results. The amount of ^{137}Cs found in the soak solutions used for rinsing the teabags was also low. The amount of ^{137}Cs in the combined 0.01 M NaOH and deionized water solutions for each teabag amounted to 0.3% and 0.5% of the ^{137}Cs found on CST in teabags A7 and A8, respectively. The ICP-MS analysis of the standards showed Ti and Zr concentrations within the control limits established during the development of the standard.[10] The Nb concentration for one sample was within the control limits, while the other was slightly low. The Ti concentrations measured for the teabag samples were within the standard control limits indicating complete dissolution. Comparison of the Nb and Zr concentrations for the teabag samples to the standard control limits is not useful due to the known leaching of these elements when contacted with tank waste.[11] While within the established control limits the Ti and Zr concentrations in the standard samples were towards the lower end. One possible cause of this could have been a difference in humidity and therefore the relative amount of moisture on the CST samples at the completion of air drying. Additional moisture would have biased the measured concentrations low. The full suite of ICP-MS results for the in-tank batch contact samples and digestion standards are provided in Tables 3-12 and 3-13, respectively. The masses from 106 to 112 are due to the presence of mono oxides of Zr and Nb. The masses from 176-180 are from hafnium (Hf) which is a known contaminant of the Zr in the CST.

As described in Task 6 of the TTR,[6] the ^{137}Cs loading for a single in-tank batch contact teabag is calculated by adding the ^{137}Cs found in the two associated soak solutions (average plus 2 sigma uncertainty[a]) to the ^{137}Cs activity calculated from the average of replicate analyses of a single teabag sample (plus 2 sigma uncertainty[a]). Those results, along with the average and standard deviation of the two individual teabags, are provided in Table 3-11.

Table 3-11. Calculated ^{137}Cs Loadings on the Individual In-Tank Batch Contact Samples.

	^{137}Cs Loading (dpm/g)	^{137}Cs Loading (Ci/kg$_{CST}$)
Teabag A7	3.76E+10	1.71E+01
Teabag A8	3.57E+10	1.61E+01
Average	3.66E+10	1.65E+01
Standard Deviation	1.40E+09	6.28E-01

[a]Values include the 2 sigma uncertainties of the CST and soak solution measurements.

As stated above the ^{137}Cs loadings reported in Tables 3-10 and 3-11 are based on the air-dried mass of CST used for the digestions. To compare these results directly with the ZAM (Zheng, Anthony, Miller) modeling results (to be documented in a later report), the loadings should be corrected for the true dry mass (i.e., mass remaining at 460 °C). For this batch of pretreated CST, the mass remaining at 460 °C was 81.91 wt %, which gives an F-factor of 0.8191.[10] The average ^{137}Cs loading from the duplicate teabags, corrected to the true dry mass, is 20.2 ± 0.77[b] Ci/kg$_{CST}$.

[a] The 2-sigma uncertainty refers to 2 standard deviations of the replicate analyses.
[b] Standard deviation from Table 3-11 corrected to the true dry mass.

Table 3-12. ICP-MS Results for In-Tank Batch Contact Samples (Teabags A7 and A8)

m/z	Avg. Conc. (µg/g)	%RSD[a]	m/z	Avg. Conc. (µg/g)	%RSD[a]	m/z	Avg. Conc. (µg/g)	%RSD[a]
Ti	1.39E+05	1.54%	133	7.02E+02	1.24%	178	5.25E+02	1.23%
59	6.81E-01	15.7%	134	< 5.17E-01	n/a	179	2.63E+02	1.50%
82	< 5.17E-01	n/a	135	7.16E+01	0.97%	180	6.85E+02	1.39%
84	< 5.17E-01	n/a	136	< 5.17E-01	n/a	181	1.40E+02	1.05%
85	5.91E+00	3.98%	137	1.46E+02	1.52%	182	< 7.75E-01	n/a
86	1.29E+00	1.12%	138	4.16E+00	3.72%	183	< 7.75E-01	n/a
87	1.37E+01	0.46%	139	4.13E+00	7.91%	184	< 7.75E-01	n/a
88	1.38E+01	6.97%	140	1.35E+00	7.57%	185	< 5.17E-01	n/a
89	1.60E+00	3.77%	141	< 5.17E-01	n/a	186	< 7.75E-01	n/a
Zr	9.22E+04	1.31%	142	< 5.17E-01	n/a	187	< 5.17E-01	n/a
92	1.38E+04	1.47%	143	< 5.17E-01	n/a	191	< 5.17E-01	n/a
Nb	1.08E+05	1.20%	144	< 5.17E-01	n/a	193	5.58E+00	3.44%
94	1.45E+04	1.55%	145	< 5.17E-01	n/a	194	1.59E+01	2.33%
95	4.68E+00	9.02%	146	< 5.17E-01	n/a	195	1.52E+01	3.89%
96	2.13E+03	1.93%	147	< 5.17E-01	n/a	196	1.77E+01	1.09%
97	1.13E+00	22.5%	148	< 5.17E-01	n/a	198	6.02E+01	11.3%
98	1.19E+00	63.4%	149	< 5.17E-01	n/a	203	< 5.17E-01	n/a
99	< 5.17E-01	n/a	150	< 5.17E-01	n/a	204	3.07E+01	2.50%
100	< 1.03E+00	n/a	151	< 5.17E-01	n/a	205	< 5.17E-01	n/a
101	< 7.75E-01	n/a	152	< 5.17E-01	n/a	206	2.61E+01	8.57%
102	< 1.03E+00	n/a	153	< 5.17E-01	n/a	207	2.34E+01	8.05%
103	< 5.17E-01	n/a	154	< 5.17E-01	n/a	208	5.63E+01	8.47%
104	< 5.17E-01	n/a	155	< 5.17E-01	n/a	229	< 5.17E-01	n/a
105	< 5.17E-01	n/a	156	< 5.17E-01	n/a	230	< 5.17E-01	n/a
106	9.26E+01	2.11%	157	< 5.17E-01	n/a	232	1.87E+00	4.47%
107	8.92E+01	0.73%	158	< 5.17E-01	n/a	233	2.47E+00	3.41%
108	4.24E+01	1.32%	159	< 5.17E-01	n/a	234	7.31E-01	2.17%
109	1.63E+02	1.35%	160	< 5.17E-01	n/a	235	1.93E+01	4.13%
110	7.72E+01	2.20%	161	< 5.17E-01	n/a	236	5.54E+00	3.57%
111	4.03E+01	1.90%	162	< 5.17E-01	n/a	237	3.64E+00	2.05%
112	1.06E+01	0.20%	163	< 5.17E-01	n/a	238	1.13E+02	4.46%
113	6.83E+00	6.89%	164	< 5.17E-01	n/a	239	< 7.75E-01	n/a
114	< 5.17E-01	n/a	165	< 5.17E-01	n/a	240	< 7.75E-01	n/a
116	< 5.17E-01	n/a	166	< 5.17E-01	n/a	241	< 7.75E-01	n/a
117	< 5.17E-01	n/a	167	< 5.17E-01	n/a	242	< 7.75E-01	n/a
118	5.82E-01	5.26%	168	< 5.17E-01	n/a	243	< 7.75E-01	n/a
119	2.02E+00	2.33%	169	< 5.17E-01	n/a	244	< 7.75E-01	n/a
120	< 5.17E-01	n/a	170	< 5.17E-01	n/a	245	< 7.75E-01	n/a
121	< 5.17E-01	n/a	171	< 5.17E-01	n/a	246	< 7.75E-01	n/a
122	< 5.17E-01	n/a	172	< 5.17E-01	n/a	247	< 7.75E-01	n/a
123	< 7.75E-01	n/a	173	< 5.17E-01	n/a	248	< 7.75E-01	n/a
124	< 5.17E-01	n/a	174	2.71E+00	0.90%	249	< 7.75E-01	n/a
125	< 1.03E+00	n/a	175	< 5.17E-01	n/a	250	< 7.75E-01	n/a
126	1.66E+00	14.0%	176	9.15E+01	1.24%	251	< 7.75E-01	n/a
128	< 5.17E-01	n/a	177	3.51E+02	1.27%	252	< 7.75E-01	n/a
130	< 5.17E-01	n/a						

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

Table 3-13. ICP-MS Results for CST Digestion Standards

m/z	Avg. Conc. (µg/g)	%RSD[a]	m/z	Avg. Conc. (µg/g)	%RSD[a]	m/z	Avg. Conc. (µg/g)	%RSD[a]
Ti	1.47E+05	1.44%	133	< 4.88E-01	n/a	178	5.47E+02	2.20%
59	< 4.88E-01	n/a	134	< 4.88E-01	n/a	179	2.71E+02	2.57%
82	< 4.88E-01	n/a	135	< 4.88E-01	n/a	180	7.08E+02	2.93%
84	< 4.88E-01	n/a	136	< 4.88E-01	n/a	181	1.47E+02	2.06%
85	< 4.88E-01	n/a	137	4.98E-01[b]	n/a	182	< 7.32E-01	n/a
86	7.28E-01	1.50%	138	2.42E+00	29.9%	183	< 7.32E-01	n/a
87	5.82E-01	10.9%	139	4.05E+00	5.17%	184	< 7.32E-01	n/a
88	6.48E+00	2.70%	140	5.37E-01	0.00%	185	< 4.88E-01	n/a
89	1.27E+00	4.93%	141	< 4.88E-01	n/a	186	< 7.32E-01	n/a
Zr	9.63E+04	2.59%	142	< 4.88E-01	n/a	187	< 4.88E-01	n/a
92	1.45E+04	1.96%	143	< 4.88E-01	n/a	191	< 4.88E-01	n/a
Nb	1.12E+05	1.91%	144	< 4.88E-01	n/a	193	5.98E+00	3.05%
94	1.52E+04	1.62%	145	< 4.88E-01	n/a	194	1.71E+01	3.77%
95	4.48E+00	0.65%	146	< 4.88E-01	n/a	195	1.62E+01	1.70%
96	2.22E+03	2.54%	147	< 4.88E-01	n/a	196	1.80E+01	0.64%
97	8.56E-01	6.11%	148	< 4.88E-01	n/a	198	1.39E+00	3.75%
98	5.01E-01	1.34%	149	< 4.88E-01	n/a	203	< 4.88E-01	n/a
99	< 4.88E-01	n/a	150	< 4.88E-01	n/a	204	1.58E+00	49.6%
100	< 9.76E-01	n/a	151	< 4.88E-01	n/a	205	< 4.88E-01	n/a
101	< 7.32E-01	n/a	152	< 4.88E-01	n/a	206	1.16E+00	40.7%
102	< 9.76E-01	n/a	153	< 4.88E-01	n/a	207	9.65E-01	26.8%
103	< 4.88E-01	n/a	154	< 4.88E-01	n/a	208	2.12E+00	44.5%
104	< 4.88E-01	n/a	155	< 4.88E-01	n/a	229	< 4.88E-01	n/a
105	< 4.88E-01	n/a	156	< 4.88E-01	n/a	230	< 4.88E-01	n/a
106	9.51E+01	2.10%	157	< 4.88E-01	n/a	232	9.15E-01	10.3%
107	9.07E+01	2.02%	158	< 4.88E-01	n/a	233	< 4.88E-01	n/a
108	4.43E+01	2.95%	159	< 4.88E-01	n/a	234	< 4.88E-01	n/a
109	1.74E+02	3.89%	160	< 4.88E-01	n/a	235	< 9.76E-01	n/a
110	8.14E+01	1.70%	161	< 4.88E-01	n/a	236	< 4.88E-01	n/a
111	4.16E+01	2.92%	162	< 4.88E-01	n/a	237	< 4.88E-01	n/a
112	1.13E+01	1.26%	163	< 4.88E-01	n/a	238	< 9.76E-01	n/a
113	7.41E+00	0.85%	164	< 4.88E-01	n/a	239	< 7.32E-01	n/a
114	< 4.88E-01	n/a	165	< 4.88E-01	n/a	240	< 7.32E-01	n/a
116	< 4.88E-01	n/a	166	< 4.88E-01	n/a	241	< 7.32E-01	n/a
117	< 4.88E-01	n/a	167	< 4.88E-01	n/a	242	< 7.32E-01	n/a
118	< 4.88E-01	n/a	168	< 4.88E-01	n/a	243	< 7.32E-01	n/a
119	< 4.88E-01	n/a	169	< 4.88E-01	n/a	244	< 7.32E-01	n/a
120	< 4.88E-01	n/a	170	< 4.88E-01	n/a	245	< 7.32E-01	n/a
121	< 4.88E-01	n/a	171	< 4.88E-01	n/a	246	< 7.32E-01	n/a
122	< 4.88E-01	n/a	172	< 4.88E-01	n/a	247	< 7.32E-01	n/a
123	< 7.32E-01	n/a	173	< 4.88E-01	n/a	248	< 7.32E-01	n/a
124	< 4.88E-01	n/a	174	2.82E+00	4.61%	249	< 7.32E-01	n/a
125	1.14E+00[b]	n/a	175	< 4.88E-01	n/a	250	< 7.32E-01	n/a
126	< 9.76E-01	n/a	176	9.59E+01	2.37%	251	< 7.32E-01	n/a
128	< 4.88E-01	n/a	177	3.65E+02	3.14%	252	< 7.32E-01	n/a
130	< 4.88E-01	n/a						

[a]The %RSD is based on the standard deviation of duplicate samples.
[b]One of the duplicates was above detection and one was below. The value shown in the table is not the average, but the single result for the replicate above detection.

Loading of several other elements was observed on the teabag CST based on the ICP-MS and ICP-ES data. A summary of loading of other elements on the CST is provided in Table 3-14 in order of decreasing loading. A number of these elements are also present in the standard samples, indicating they are trace components of the as-manufactured and pretreated CST. Therefore, concentrations in both the teabag CST and standard CST are shown. The full ICP-ES results for the teabag samples and standards are provided in Table 3-15.

Table 3-14. Comparison of Cs Loading With That of Other Elements on the CST.

Element	Avg. Amount in Teabags (mmol/g_{CST})	Avg. Amount in Standards (mmol/g_{CST})	Net Loading (Teabag-standard) (mmol/g_{CST})
Na	3.96E+00	3.19E+00	7.72E-01
Al	2.10E-01	1.12E-01	9.86E-02
Ca	5.02E-02	2.37E-02	2.65E-02
Fe	3.33E-02[a]	8.24E-03[b]	2.51E-02
Cs	8.69E-03	≤ 1.47E-05	≥ 8.67E-03
U	5.95E-04[c]	< 1.45E-05[c]	> 5.81E-04
Pb	5.17E-04	2.13E-05	4.96E-04
Rb	2.29E-04	≤ 1.25E-05	≥ 2.17E-04
Sr	1.90E-04	8.95E-05	1.01E-04
^{237}Np	1.54E-05	< 2.06E-06	> 1.33E-05
Co	1.15E-05	< 8.27E-06	> 3.28E-06

[a]Large discrepancy between the two teabag samples (104 %RSD), Teabag A7 had an Fe loading of 3230 µg/g while Teabag A8 had an Fe loading of 493 µg/g. [b]Large discrepancy between the two standard samples (84.9 %RSD), Standard A7 had an Fe loading of 184 µg/g and Standard A8 had an Fe loading of 736 µg/g. [c]Sum of isotopes 233, 234, 235, 236, and 238. All U isotopes were below the detection limit in the standard samples.

Table 3-15. ICP-ES Results for the In-Tank Batch Contact Samples (Teabags A7 and A8) and Associated Digestion Standards

Element	Avg. Conc. In Teabag Samples (µg/g)	%RSD[a]	Avg. Conc. In Standard Samples (µg/g)	%RSD[a]
Ag	< 89.2	n/a	< 84.2	n/a
Al	5670	1.75%	3010	1.41%
Ba	< 44.7	n/a	< 42.2	n/a
Be	< 4.40	n/a	< 4.15	n/a
Ca	2010	4.93%	948	2.39%
Cd	< 61.2	n/a	< 57.7	n/a
Ce	< 346	n/a	< 327	n/a
Co	< 77.8	n/a	< 73.5	n/a
Cr	< 73.7	n/a	< 69.6	n/a
Cu	< 414	n/a	< 390	n/a
Fe	1862[b]	104%	460[c]	84.9%
Gd	< 74.2	n/a	< 70.0	n/a
K	< 1435	n/a	< 1355	n/a
La	< 23.1	n/a	< 21.8	n/a
Li	< 51.0	n/a	< 48.2	n/a
Mg	283	1.00%	332	7.04%
Mn	< 62.0	n/a	< 58.6	n/a
Mo	< 295	n/a	< 279	n/a
Na	91050	6.29%	73300	1.16%
Ni	< 456	n/a	< 430	n/a
P	< 547	n/a	< 517	n/a
Pb	< 2445	n/a	< 2310	n/a
Sb	< 1190	n/a	< 1125	n/a
Sn	< 1535	n/a	< 1450	n/a
Sr	< 15.5	n/a	< 14.7	n/a
Th	< 259	n/a	< 244	n/a
Ti	139500	2.53%	148000	1.91%
U	< 1295	n/a	< 1225	n/a
V	< 104	n/a	97.6	n/a
Zn	< 155	n/a	< 147	n/a
Zr	90200	2.35%	95550	1.55%

[a]The %RSD is based on the standard deviation of duplicate samples. The reported analytical method uncertainties (at two sigma) are 10%. [b]Large discrepancy between the two teabag samples (3230 µg/g and 493 µg/g). [c]Large discrepancy between the two standard samples (184 µg/g and 736 µg/g).

4.0 Conclusions

SRNL received and characterized a set of dip samples collected from Tank 10H (one surface and one variable depth sample) for qualification of TCCR Batch 3. No solids were observed in either sample and therefore the samples were combined, and a suite of analyses were performed on the composite sample. The density of the combined sample was 1.174 g/mL (0.18 %RSD), which was consistent with the measured sodium concentration of 3.54 M (0.08 %RSD). The ^{137}Cs activity in the sample was 4.75E+07 dpm/mL (4.28 %RSD) and the total Cs concentration was calculated to be 1.54 mg/L (1.16E-05 M).

Duplicate in-tank batch contact samples consisting of 0.1 g of crystalline silicotitanate (CST) contained within teabag devices were also received and characterized by SRNL after being submerged in Tank 10H for a period of about 13 days. The CST was rinsed to remove excess salt solution and the CST was then

air dried before being digested for analysis. Results of the analysis indicated a ^{137}Cs loading of 3.66E+10 ± 1.40E+09 dpm/g or 16.5 ± 0.63 Ci/kg_{CST}. This value represents a bounding upper limit as it includes the addition of two sigma uncertainty from replicate analysis of the individual teabag samples as well as the addition of the small amount of ^{137}Cs activity measured in the rinse solutions. The above values are based on the air-dried mass of CST. Correcting to the true dry mass using a F-factor of 0.8191 results in a maximum loading of 20.2 ± 0.77 Ci/kg_{CST}.

5.0 Future Work

ZAM modeling will be performed using the measured Tank 10H Batch 3 composition. These results will then be compared to the measured Cs loading determined here. Results from additional analyses of the digested CST from the in-tank batch contact testing samples will also be documented in a future report.

6.0 References

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