Summary of Analytical Results from Samples Supporting Tank Closure Cesium Removal (TCCR) Batch 3 and Modeling Results for Cs Loading on CST

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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. Some results from analysis of those samples have been previously reported. This report documents the remaining analyses of the in-tank batch contact samples as well as the results of ZAM (Zheng, Anthony, Miller) isotherm modeling performed for comparison to the measured results.

Results of the additional analyses include analysis of the loading of other radionuclides besides ^{137}Cs on the crystalline silicotitanate (CST) contained within the in-tank batch contact test samples. Results from those analyses revealed the next highest contributor to the activity on the CST was ^{90}Sr with an average loading of 2.76E+08 dpm/g_{CST} compared to 3.56E+10 dpm/g_{CST} for the ^{137}Cs. Isotopes of plutonium were also detected on the samples. ZAM modeling was performed using the measured composition of the Tank 10H Batch 3 qualification samples. The modeling predicted a maximum Cs loading approximately 2.2x higher than the measured result. This is a slightly lower ratio (expected/measured) compared to what was observed for the prior TCCR in-tank batch contact testing performed for Batches 1A and 2 where the ZAM results were 2.7-2.8x higher than the measured values.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

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
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
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). Details of the preparation of TCCR Batch 3 were described in an earlier report along with characterization of the supernate samples and select results from the in-tank CST batch contact equilibrium samples.[1] Analytical results from the in-tank batch equilibrium samples (HTF-10-20-54 and -55) not previously reported are summarized herein. In addition to the analytical results, modeling was performed using the ZAM (Zheng, Anthony, Miller) isotherm model to determine the expected Cs loading on CST based on the measured Tank 10H Batch 3 composition and those results are also discussed in this report.

2.0 Experimental Procedure

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

Two modified sample vials containing the CST teabags (original holder design) 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 processed according to the established procedure.[2] 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 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 Analytical Development (AD) for analysis. A portion of those results were documented in a previous report[1] and the remaining results are summarized herein. In addition to the digested CST results, samples of the soak solutions generated during processing of the teabags were also submitted for analysis. The gamma spectroscopy results from those samples were previously reported;[1] the inductively coupled plasma – emission spectroscopy (ICP-ES) results are documented here.

2.2 Modeling Approach

Cesium loading on the CST was predicted using the ZAM isotherm model developed by the research group of Professor Rayford G. Anthony of Texas A&M University. The ZAM program, named after its developers (i.e., Zheng, Anthony, and Miller), was described in detail in a previous ion exchange study at SRNL.[3] In addition, the OLI Studio™ software (Version 10) from OLI Systems, Inc., was used to estimate feed solution density required as input data to the ZAM program.

There are two options to predict cesium loading.
- Use of an isotherm: An isotherm provides the equilibrium relation between the concentration of cesium loaded on the CST surface to the concentration of cesium in the solution. The isotherm covers a wide range of liquid-phase cesium concentrations. ZAM can generate equilibrium cesium loading data at a given temperature. Generally, an excellent fit for the ZAM data would be achieved by use of the Freundlich/Langmuir isotherm model.
- Variation of ZAM phase ratio: A phase ratio, ϕ, is defined as the ratio of total liquid volume (mL) processed to the mass of CST resin (g_{CST}). To simulate the saturation of cesium loading on CST for a specified feed, ZAM calculations are performed at increasing phase ratios until the calculated equilibrium liquid cesium concentration approximates (usually accurate up to 4 digits) the feed

cesium concentration. The corresponding cesium concentration on CST represents the maximum cesium loading.

The two approaches should deliver practically identical results. The methods were discussed in detail in a previous report.[4]

Note that IE-911 and R9120-B (engineered forms of CST) contain inert binder while ZAM data are based on IE-910 (powdered form) and the true-dry mass of CST (i.e., mass remaining at 460 °C). To compare ZAM predictions with test data using engineered CST, a recommended dilution factor of 0.68[3] is frequently applied to account for the binder effect. Additionally, since test data are based on the air-dried mass of CST, an F-factor of 0.8191, determined in the current work for this CST batch,[5] is needed for conversion to the true-dry mass basis.

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.[6] This work was performed following the applicable Task Technical and Quality Assurance Plan (TTQAP).[7] The Task Technical Request (TTR) associated with this work[8] 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.[9] Data are recorded in the Electronic Laboratory Notebook (ELN) system.[10]

OLI modeling is controlled under Software Quality Assurance Plan X-SQP-A-00001, Rev. 0.[11] ZAM is currently classified as Level D software[12] and the functional requirements placed on ZAM were verified and validated.[3]

3.0 Results and Discussion

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

^{137}Cs activities as well as the full suite of inductively coupled plasma – mass spectrometry (ICP-MS) and ICP-ES results for the digested CST from the teabags and the CST digestion standards were reported in a prior document.[1] A summary of other radionuclide data for the in-tank batch contact samples as well as the CST digestion standards is provided in Table 3-1. The gamma emitting isotopes were measured by gamma spectroscopy after Cs removal, the ^{90}Sr and Pu isotopes were measured after separation, and the total alpha and beta were measured by liquid scintillation counting (LSC) both with and without Cs removal. For comparison, the average ^{137}Cs activity on the teabag CST was 3.56E+10 dpm/g$_{CST}$.[1] Based on the cesium results, it is assumed that radionuclide (Sr and Pu) losses to the wash solutions are small relative to the amounts recovered from the teabags.

Table 3-1. Other Radionuclide Activities in the In-Tank Batch Contact Samples and CST Digestion Standards (Not Corrected for the F-Factor)

Isotope	Avg. Activity in Teabag CST (dpm/g$_{CST}$)	%RSD[a]	Avg. Analytical 1-sigma Unc.	Avg. Activity in Standard CST (dpm/g$_{CST}$)	%RSD[a]	Avg. Analytical 1-sigma Unc.
^{60}Co	< 9.76E+03	n/a	MDA	< 8.75E+03	n/a	MDA
^{90}Sr	2.76E+08	2.56%	14.1%	1.48E+05[b]	n/a	29.3%
^{106}Ru	< 8.02E+04	n/a	MDA	< 4.56E+04	n/a	MDA
^{125}Sb	< 5.08E+04	n/a	MDA	< 2.15E+04	n/a	MDA
^{126}Sb	< 1.42E+04	n/a	MDA	< 7.48E+03	n/a	MDA
^{126}Sn	< 8.94E+04	n/a	MDA	< 1.92E+04	n/a	MDA
^{135}Cs	8.74E-01	11.8%	20.0%	< 5.50E-02	n/a	Upper Limit
^{144}Ce	< 1.50E+05	n/a	MDA	< 4.58E+04	n/a	MDA
^{154}Eu	< 2.83E+04	n/a	MDA	< 1.28E+04	n/a	MDA
^{238}Pu	8.62E+06	5.91%	7.86%	3.61E+03[c]	n/a	22.9%
$^{239/240}$Pu	1.07E+05	3.97%	11.2%	< 1.91E+03	n/a	MDA
^{241}Pu	2.78E+06	31.0%	16.3%	< 1.05E+04	n/a	MDA
^{241}Am	< 2.71E+05	n/a	MDA	< 7.05E+04	n/a	MDA
Alpha Count[d]	< 1.11E+09	n/a	MDA	< 5.26E+06	n/a	MDA
Beta Count[d]	4.34E+10	0.98%	10.0%	< 7.60E+06	n/a	MDA
Alpha Count[e]	< 3.79E+07	n/a	Upper Limit	< 1.65E+05	n/a	MDA
Beta Count[e]	4.97E+08	13.5%	10.0%	< 2.10E+05	n/a	MDA/Upper Limit

[a]The %RSD is based on the standard deviation of duplicate samples. [b]Result from a single standard sample rather than the average. The other sample was below the detection limit (< 1.22E+05 dpm/g). [c]Result from a single standard sample rather than the average. The other sample was below the detection limit (< 2.17E+03 dpm/g). [d]From LSC without Cs removal. [e]From LSC after Cs removal.

In addition to analyzing the digested CST, the rinse solutions generated from rinsing the teabags prior to disassembly were analyzed. As reported in the prior document, the amount of ^{137}Cs found in the soak solutions was very low.[1] 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. Duplicate aliquots of each soak solution were also analyzed by ICP-ES and those results are provided in Table 3-2. As can be seen in the table, the soak solutions showed very low concentrations of a few elements, with a composition that is consistent with residual salt solution being retained in the teabags. The average measured sodium concentration of the 0.01 M NaOH rinses was 0.061 M, which is equivalent to ~ 1 mL of the Tank 10H supernate being diluted into the 65 mL of 0.01 M NaOH rinse solution. The DI water rinses contained ~ 40 mg/L Na, which is equivalent to ~ 2 mL of the first rinse (0.01 M NaOH) being carried over into the water rinse.

Table 3-2. Full ICP-ES Results for the Soak Solutions

Element	Avg. Concentration in 0.01 M NaOH rinse solutions (mg/L)	%RSD[a]	Avg. Concentration in DI water rinse solutions (mg/L)	%RSD[a]
Ag	< 0.035	n/a	< 0.035	n/a
Al	9.51	8.46%	0.819	1.03%
B	< 0.028	n/a	< 0.028	n/a
Ba	< 0.017	n/a	< 0.017	n/a
Be	< 0.002	n/a	< 0.002	n/a
Ca	0.090[b]	10.2%[b]	< 0.034	n/a
Cd	< 0.029	n/a	< 0.029	n/a
Ce	< 0.143	n/a	< 0.143	n/a
Co	< 0.036	n/a	< 0.036	n/a
Cr	0.198	44.8%	< 0.029	n/a
Cu	< 0.009	n/a	< 0.009	n/a
Fe	0.057	5.76%	< 0.030	n/a
Gd	< 0.029	n/a	< 0.029	n/a
K	< 0.747	n/a	< 0.747	n/a
La	< 0.017	n/a	< 0.017	n/a
Li	< 0.037	n/a	< 0.037	n/a
Mg	0.003	17.5%	0.002[b]	7.44%[b]
Mn	< 0.027	n/a	< 0.027	n/a
Mo	< 0.114	n/a	< 0.114	n/a
Na	1413	8.02%	42.0	17.2%
Ni	< 0.176	n/a	< 0.176	n/a
P	< 0.613	n/a	< 0.613	n/a
Pb	< 0.242	n/a	< 0.242	n/a
S	127	5.81%	< 7.53	n/a
Sb	< 0.750	n/a	< 0.750	n/a
Si	< 0.134	n/a	< 0.134	n/a
Sn	< 0.474	n/a	< 0.474	n/a
Sr	< 0.003	n/a	< 0.003	n/a
Th	< 0.074	n/a	< 0.074	n/a
Ti	< 0.023	n/a	< 0.023	n/a
U	< 0.663	n/a	< 0.663	n/a
V	< 0.019	n/a	< 0.019	n/a
Zn	< 0.038	n/a	< 0.038	n/a
Zr	0.038	27.6%	< 0.011	n/a

[a]The %RSD is based on the standard deviation of four samples (duplicates for each teabag). [b]Average and %RSD of duplicate samples from one soak solution as the other was below the detection limit.

3.2 ZAM Modeling Results

The measured composition of the Tank 10H Batch 3 qualification samples was used as input for the ZAM modeling calculations. The composition was documented in a previous report,[1] but is also summarized in Table 3-3 for reference. The ionic charges of the measured concentrations shown in Table 3-3 are not balanced. The total positive charge is 3.54176 eq/L while the total negative charge is -4.03780 eq/L. In the past, the total charge of anions is usually less than the total charge of cations; and therefore, the common practice has been to increase the Cl⁻ concentration to achieve the charge balance, since the change in chloride concentration has a minimal impact on cesium loading. Since that is not possible for this case, two options were considered for balancing the charges in the Batch 3 solution.

1. Full adjustment: Both anions and cations (except Cs^+) were adjusted to have an absolute charge balance at 3.78978 eq/L. This charge value is the average of the non-balanced total charges for anions and cations.
2. Adjustment of anions: Since it is not clear which specific anions should be adjusted, all anion concentrations were equally decreased by multiplying by 0.87715 to have a complete charge balance.

Both options are shown in Table 3-4. As seen in Table 3-4, the second option (i.e., adjustment of anions) provides an OLI calculated density of 1.1737 g/cm³ at 26 °C, which is nearly identical to the measured value. Therefore, Option 2 (anion adjustment) was selected as the adjusted composition for modeling purposes.

Table 3-3. Batch 3 Measured Concentrations

Component	Measured Concentration (M)
Na^+	3.54
K^+	1.74×10^{-3}
Cs^+	1.16×10^{-5}
Rb^+	4.31×10^{-6}
Total Sr	1.12×10^{-7}
Pb^{2+}	5.63×10^{-7}
OH^-	0.114
NO_3^-	2.08
NO_2^-	0.540
$Al(OH)_4^-$	0.0346
CO_3^{2-}	0.380
SO_4^{2-}	0.245
PO_4^{3-}	7.56×10^{-4}
$C_2O_4^{2-}$	8.34×10^{-3}
CrO_4^{2-}	1.24×10^{-4}
Measured Density (g/cm³) at 26 °C	1.174

Table 3-4. Batch 3 Adjusted Concentrations

Component	Full Adjustment (M)	Anion Adjustment (M)
Na^+	3.79	3.54
K^+	1.86×10^{-3}	1.74×10^{-3}
Cs^+	1.16×10^{-5}	1.16×10^{-5}
Rb^+	4.61×10^{-6}	4.31×10^{-6}
Total Sr	1.20×10^{-7}	1.12×10^{-7}
Pb^{2+}	6.02×10^{-7}	5.63×10^{-7}
OH^-	0.107	0.100
NO_3^-	1.95	1.82
NO_2^-	0.507	0.474
$Al(OH)_4^-$	0.0325	0.0303
CO_3^{2-}	0.357	0.333
SO_4^{2-}	0.230	0.215
PO_4^{3-}	7.10×10^{-4}	6.63×10^{-4}
$C_2O_4^{2-}$	7.83×10^{-3}	7.32×10^{-3}
CrO_4^{2-}	1.16×10^{-4}	1.09×10^{-4}
OLI Calculated Density (g/cm³) at 26 °C	1.185	1.1737

The CST isotherm was determined by use of the Freundlich/Langmuir Hybrid model to fit the ZAM data. The isotherm parameters are listed in Table 3-5. With the parameters listed, the expression reduces to a Langmuir isotherm.

Freundlich/Langmuir Hybrid isotherm model:

$$q = \frac{\eta_{df} C_T \rho_{Bed} C_p^{M_a}}{\beta + b C_p^{M_b}}$$

q: Cesium loading on CST (mol$_{Cs}$/L$_{Bed}$)
C_p: Liquid-phase cesium concentration (mol$_{Cs}$/L)

Table 3-5. Isotherm Parameters

Feed	T (°C)	η_{df}	C_T (mmol$_{Cs}$/g$_{CST}$)	Dry ρ_{Bed} [a] (g$_{CST}$/mL)	M_a	M_b	β (mol$_{Cs}$/L)	b (-)
Batch 3 Anion Adjusted Sample	36.8	0.68	0.58	0.991	1	1	1.7485E-4	1

[a]Dry bed density calculated from bed density (1.2097 g/mL) and F-factor (0.8191).

The maximum cesium loading was calculated by the method of phase ratio variation. Cesium loadings at 36.8 °C are given in Table 3-6 and are on a true dry mass basis (i.e., accounting for the F-factor). The ZAM loading value (engineered form) is 2.2x larger than the measured teabag result. This is a slightly lower ratio compared to what was observed for the prior TCCR in-tank batch contact testing performed for Batches 1A and 2 where the ZAM results were 2.7-2.8x higher than the measured values.[13,14]

Table 3-6. Maximum Cesium Loading at 36.8 °C

Case	q (Powder Form)[a]		q (Engineered Form)[b]		Teabag	
	mmol$_{Cs}$/g$_{CST}$	Ci/kg$_{CST}$	mmol$_{Cs}$/g$_{CST}$	Ci/kg$_{CST}$	mmol$_{Cs}$/g$_{CST}$	Ci/kg$_{CST}$
Batch 3 Sample	0.03608	66.5	0.02453	45.3	0.0110	20.2

[a]Calculated from ZAM. [b]Applied dilution factor η_{df} of 0.68 to ZAM values.

Cesium loading and isotherms for both fully adjusted and anion only adjusted compositions are shown in Figure 3-1, along with the teabag result. A dilution factor (η_{df}) of 0.305 was required to match the experimental data with the calculated isotherm for the anion adjusted composition.
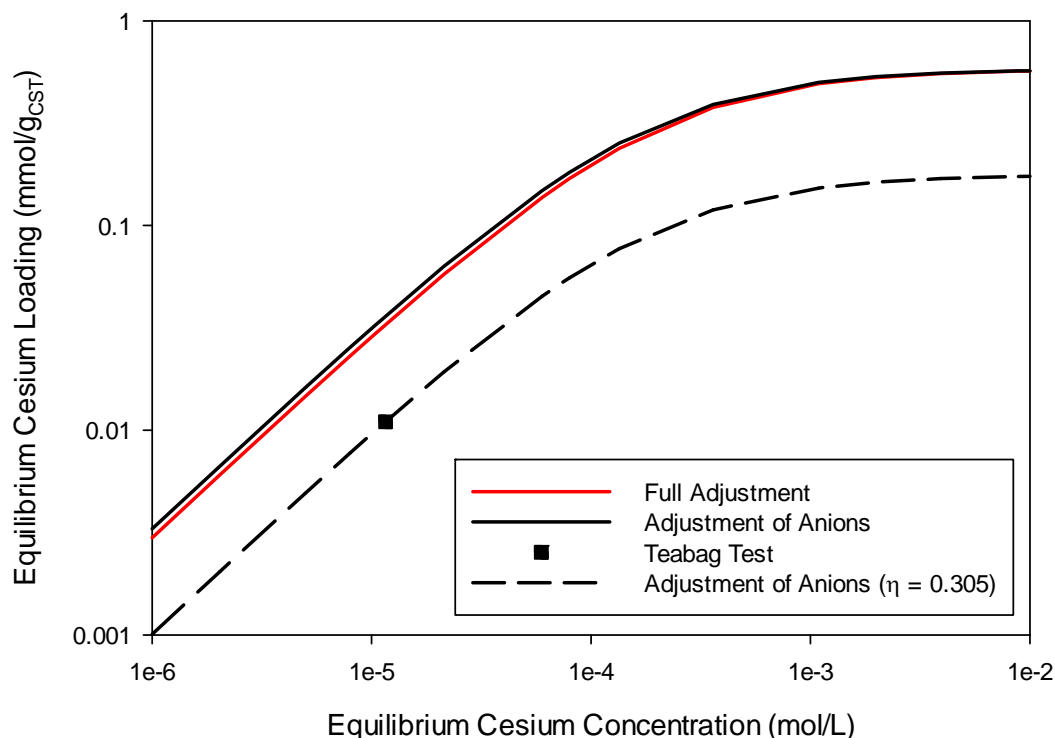
Figure 3-1. Batch 3 Isotherms at 36.8 °C.

The difference between the ZAM calculated loading and the loading results for the teabag samples are similar to the differences seen for the Batch 1A and Batch 2 teabags. One possible cause for the discrepancy is competition from other ions. Recently completed K_d experiments utilizing the actual Tank 10H surface samples from Batch 1A and Batch 2 that had been aged for several months in the Shielded Cells showed Cs loadings higher than observed on the teabags, but still lower than the ZAM prediction.[15] Dilution factors of 0.578 and 0.568 were required to match the testing results for the aged Batch 1A and Batch 2 supernates, respectively. The dilution factor calculated for the aged Batch 1A supernate was closer to the standard (0.68) dilution factor compared to what had been observed in testing several months earlier (15% low versus 32% low) indicating Cs loadings on the CST improved following supernate aging. Simulant testing to examine the impact of alkaline earth metals and their competition with Cs for binding sites on the CST was also recently performed, and although efficient removal of the alkaline earth elements (Ca, Ba) was observed, their removal did not impact the Cs removal under the conditions tested.[16] At an infinite phase ratio competitors may be more likely to have an impact on the Cs loading. Another potential reason for the discrepancy between the modeling and experimental data is mass transfer limited sorption during teabag loading in Tank 10H. Recent testing of a newly designed teabag holder meant to increase mass transfer to the CST showed improved loadings compared to the older design; however, loadings were still much lower than predicted by ZAM modeling.[17]

4.0 Conclusions

SRNL completed the analysis of the duplicate in-tank batch contact samples consisting of 0.1 g of CST contained within teabag devices as well as the associated rinse solutions. Results of these analyses revealed loadings of other radionuclides in addition to the ^{137}Cs loading previously reported. The most significant contributors to the activity on the CST after ^{137}Cs were ^{90}Sr (2.76E+08 dpm/g_{CST}) and the Pu isotopes (total of all isotopes: 1.15E+07 dpm/g_{CST}). For comparison the average ^{137}Cs activity on the teabag CST was 3.56E+10 dpm/g_{CST}.

ZAM modeling was also performed using the measured composition of the Batch 3 Tank 10H qualification sample. The maximum loading determined using ZAM modeling and the standard dilution factor of 0.68, was approximately 2.2x larger than the measured teabag result. Adjustment of the isotherm to match the teabag data requires a dilution factor of 0.305. This difference between the modeling and measured Cs loadings is similar in magnitude to the previously qualified TCCR batches.

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