Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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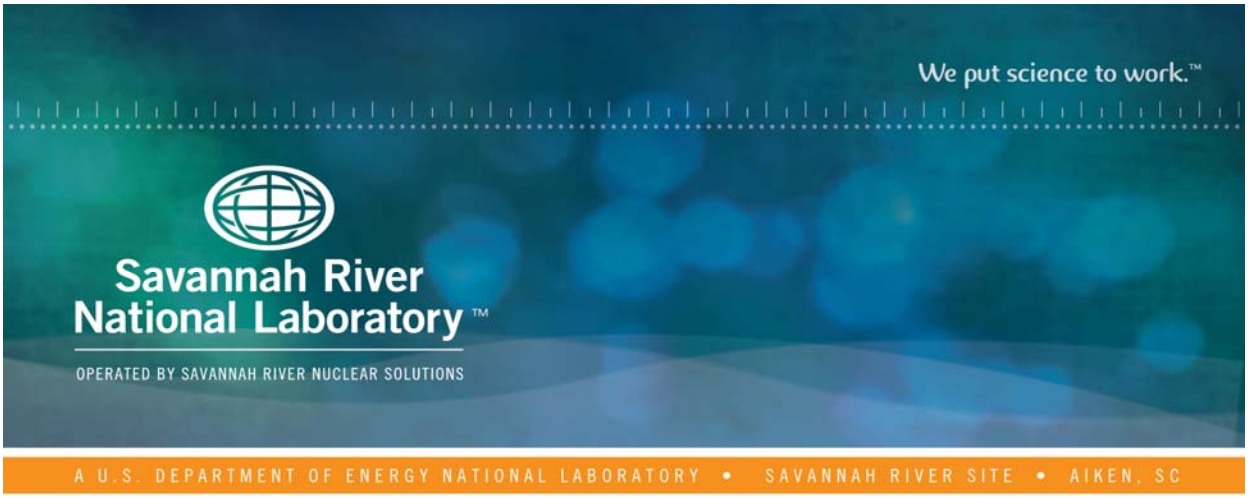

Pretreatment of Crystalline Silicotitanate (CST) and Development of a Digestion Standard to Support Tank Closure Cesium Removal (TCCR)

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March 2019

SRNL-STI-2019-00045, Revision 0

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Printed in the United States of America

Prepared for
U.S. Department of Energy

SRNL-STI-2019-00045
Revision 0

Keywords: *CST, pretreatment, standards*

Retention: *Permanent*

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EXECUTIVE SUMMARY

In support of Savannah River Remediation's (SRR) deployment of an ion exchange process to remove radioactive cesium from waste supernate, referred to as the Tank Closure Cesium Removal (TCCR) process, SRNL was tasked with validating the field protocol for pretreating the crystalline silicotitanate (CST) material to be used in the columns, preparing CST for use in in-tank batch contact testing, as well as developing a digestion standard to verify complete dissolution of the CST prior to characterization after each in-tank batch contact test. A procedure (SRNL L29 Manual, ITS-0229) was developed to document the protocol for pretreating CST utilizing conditions similar to what will be performed in the field. Analysis of material pretreated following the procedure confirmed similar conversion to the Na^+ form of the resin as had been obtained previously. Portions of the batch of material pretreated according to the developed protocol were also used for development of a digestion standard. Results from digestion and analysis of this standard were used to establish a set of control limits for the measured Ti, Zr, and Nb concentrations in digested CST material. An aliquot of this standard CST material will be digested and analyzed alongside each in-tank batch contact sample, and the measured concentrations of Ti, Zr, and Nb will be compared against the established control limits to confirm complete digestion has occurred.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

BV	bed volume
CST	crystalline silicotitanate
DCP	direct current plasma spectroscopy
DI	deionized
ICP-ES	inductively coupled plasma – emission spectroscopy
ICP-MS	inductively coupled plasma – mass spectrometry
IX	ion-exchange
LCL	lower confidence limit
SAC	Specific Administrative Control
SPC	statistical process control
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
TCCR	Tank Closure Cesium Removal
TGA	thermogravimetric analysis
UCL	upper confidence limit
XRF	X-ray fluorescence

1.0 Introduction

Savannah River Remediation (SRR) is deploying an ion exchange process to remove radioactive cesium from waste supernate which is referred to as the Tank Closure Cesium Removal (TCCR) system. The main unit operations of the TCCR system are salt retrieval, filtration, ion exchange (IX), ventilation, and Interim Safe Storage. The current plan is to deploy the TCCR demonstration project near Tank10H at the Savannah River Site H-Tank Farm. Prior to operation of TCCR, Tank 10H must undergo dissolution campaigns, dissolving the salt cake to form an aqueous salt solution (supernate). The contents of Tank 10H are planned to be recirculated via a new transfer pump with return lines to three different tank locations. The recirculation of the supernate will minimize potential liquid density gradients (stratification) within the tank. The same transfer pump and a new transfer line will provide a pathway for sending the supernate from Tank 10H to the TCCR system. Once inside the TCCR system, the salt solution stream will be fed to a set of pre-filters. The filtered salt solution will then pass through IX columns containing crystalline silicotitanate (CST) media commercially known as UOP IONSIV™ R9120-B to remove cesium as well as lesser amounts of strontium-90 and actinides. The effluent Decontaminated Salt Solution will be stored in Tank 11H until an evaluation has been performed to verify the waste complies with the Saltstone Processing Facility's Waste Acceptance Criteria, at which time it can be transferred to Tank 50H for final disposition.

The TCCR Safety Basis implements a Specific Administrative Control (SAC), which functions to prevent the column from reaching 100 °C.[1] Reaching or exceeding a temperature of 100 °C may result in over pressurization of the columns, which in turn could result in release of radionuclides from the IX column. The SAC ensures the ^{137}Cs loading on the CST does not exceed 293 curies of ^{137}Cs per kilogram of CST. To verify that the loading will remain in compliance with the limit established by the SAC, in-tank batch contact testing will be performed for each batch to be processed through TCCR. The in-tank batch contact tests will consist of duplicate samples for each batch. Each sample will consist of a mass of CST, which has been pre-treated according to a specified protocol, confined in a Swagelok fitting, referred to as a "teabag", which is then placed inside a sample vessel. The mass of CST will be confined between two pieces of stainless-steel mesh in the Swagelok fitting. Two sample vessels containing one teabag each will provide two separate masses of CST to serve as duplicate samples for a single in-tank batch contact test.

SRR requested SRNL to provide these prepared sample vessels for deployment in in-tank batch contact testing. As part of this request SRNL developed a procedure to be used for preparing the sample vessels, including pre-treatment of the CST, as well as a separate procedure for receipt and preparation for analysis of the samples after the in-tank batch contact testing. SRR provided the planned field protocol for pre-treating the CST, and SRNL conducted testing to confirm the effectiveness of the protocol for converting the CST to the Na^+ form compared to the lab pre-treatment process. This protocol was then used for preparing pre-treated CST to be used in the sample vessels for in-tank batch contact testing. After the in-tank batch contact testing, SRR will return the sample vessels to SRNL for determination and reporting of the Cs loadings on the CST. As part of this, SRNL prepared a digestion standard to confirm complete dissolution of the CST material under digestion conditions used for samples from the in-tank batch contact testing.

2.0 Experimental Procedure

2.1 CST Pretreatment

A total of three CST pretreatments were performed. Two were performed during the development of the procedure, SRNL Manual L29, ITS-0229, to confirm the protocol would successfully convert the resin to the sodium form, and the third pretreatment was performed executing the approved procedure to prepare the material to be used in the in-tank batch contact tests as well as the material to be used as the digestion standard. The developed procedure was based on the protocol to be used in the field for preparing the

TCCR columns, as documented in X-ESR-H-00959,[2] with minor adaptations. In the field, the columns will be flushed with filtered (0.5 µm) well water in an up-flow configuration to remove fines. Sodium hydroxide (3 M ± 0.16 M) will then be flowed down-flow at a rate of approximately 2 gallons per minute (gpm). The minimum volume of NaOH to be utilized is 561 gallons and the column will be held full of NaOH for a minimum of 72 hours prior to initiating feed of the waste solution to ensure complete exchange of H^+ and Na^+ occurred. Adaptations made to perform this protocol in the laboratory included the use of deionized (DI) water instead of well water for the fines elutriation and a final rinsing of the CST to remove NaOH after the 72-hour hold. The final rinse was performed with DI water in a down-flow configuration and was continued until the pH of the effluent was ≤ 11.5. This step was necessary to obtain an accurate weight of the dried CST after conversion. Justification of these deviations has been documented.[3] Details of each of the three pretreatments performed are provided in the following sections.

2.1.1 Equipment

All three pretreatments utilized the same equipment set-up, which included a column with an internal diameter of 19 mm fitted with a screen at the bottom to support the CST bed. Three different Fluid Metering, Inc. (FMI) pumps were used for the three stages of pretreatment: up-flow fines elutriation with DI water, down-flow NaOH treatment, and down-flow DI water rinsing. Each pump was fitted with an appropriately sized head, and the flow rate was set and measured using DI water. Table 2-1 provides a summary of the pump equipment and flow rates utilized for each step.

Table 2-1. FMI pumps and flow rates utilized for pretreatment steps.

Pump	Step	FMI pump motor	FMI pump head	Target flow rate (mL/min)	Target flow rate (BV/h)
1	Fines Elutriation	QG150	Q2	28.7	58.6
2	NaOH Pretreatment	QG50	RH00	0.45	0.91
3	Final Water Rinse	QG20	Q1	1.5	3.06

2.1.2 Test Pretreatment #1

The first test pretreatment was performed using a draft of ITS-0229 and CST from lot number 2099000035 (this production lot represents ~13 wt % of the CST in the TCCR columns). NaOH solution was prepared by diluting 50 wt % NaOH. The intention had been to prepare 3 M NaOH, but it was later determined (after the experiment had been performed) that the solution prepared was 3.75 M. The column, associated equipment, and pump 1 were set up for up-flow of DI water. 29.4016 g (~29.4 mL) of as-received CST was weighed into a beaker. A separate beaker was then filled with DI water, and the CST was transferred into the water. Sufficient water was added such that the CST was fully submerged with the water level slightly above the level of the CST. The CST was gently stirred with a stirring rod for approximately 30 seconds. Air bubbles were observed escaping from the CST. The top of the column was removed, and the CST slurry was sluiced into the column. Additional DI water was used to ensure all the CST had been transferred into the column. The lid of the column was then attached, and the feed container was filled with DI water. The effluent was collected from a side arm of the column head into a 500-mL graduated cylinder. Pump 1 was started to supply DI water up-flow into the column. The flow was continued until the fines visibly appeared flushed from the column. After approximately 200 mL of effluent had been collected the volume of water above the CST appeared mostly clear. Pumping was continued to clear the volume in the column head which still appeared slightly turbid. Once the fines elutriation was complete, the equipment was rearranged into the down-flow configuration and pump 1 was replaced with pump 2. The supply container was filled with 63 mL (2.14 BV) of NaOH solution and solution was pumped down-flow through

the column at a rate of ~0.45 mL/min. Just before reaching the end of the 63 mL in the feed container, the pH of the effluent was measured to be approximately 14. Once the 63 mL had been pumped from the supply container, the pump was stopped for approximately 9 minutes to simulate the changing out of totes in the field. The supply container was refilled with another 63 mL of NaOH solution (4.3 BV total) and the pumping resumed. Once the supply container was emptied pumping continued to empty the feed lines into the column. The column was then left in this configuration (filled with NaOH solution) for 72 hours. After the 72-hour hold, pump 2 was replaced with pump 3 and the feed container was filled with 2 L of DI water. Pumping of the rinse solution continued overnight, and the following morning the pumping was stopped after confirmation the pH was ≤ 11.5. Approximately 1.6 L (54.5 BV) of DI water had been used for this final rinse. The CST was then removed from the column and transferred into a disposable cup filter where the water was removed by vacuum filtration. The CST was then transferred to a pre-weighed crystallizing dish and placed into a 35 °C oven to dry. The dish was removed from the oven periodically and weighed until it remained constant (no additional weight loss) for three consecutive weighings. After removal from the oven, the CST immediately began adsorbing moisture from the air resulting in mass gain. Therefore, the CST was left on the benchtop loosely covered until a stable weight was obtained. At that time the dry CST was transferred to a capped vial.

2.1.3 Test Pretreatment #2

A second test pretreatment was performed after the error was discovered in the 3 M NaOH preparation in the first test pretreatment. The protocol for this second pretreatment was very similar to the first pretreatment. CST from lot number 2099000035 was again used. For loading the CST into the column, instead of pre-wetting the CST then transferring the slurry into the column, the column was partially filled with water and the dry CST was then slowly poured into the column. The total amount of as-received CST used in this test was 29.4040 g. Approximately 250 mL of effluent was collected during the fines elutriation. The pause time between the first and second aliquots of the NaOH was 5 minutes in this test, and similarly to Pretreatment #1, the pH of the effluent was observed to increase to approximately 14 just before the pause to refill the supply container (~58 mL of NaOH delivered to the column). Approximately 1.45 L of DI water was used for the final rinse. All other details are the same as the Test Pretreatment #1.

2.1.4 Pretreatment to prepare CST to be used for in-tank batch contact testing (Pretreatment #3)

This final pretreatment evolution was performed using the final approved version of ITS-0229 and utilized CST from lot number 2099000034 (this production lot represents ~87 wt % of the CST in the TCCR columns). The details were identical to those described for Test Pretreatment #2 above, utilizing 29.4501 g (~29 mL) of as-received CST. Approximately 330 mL of effluent was collected during the fines elutriation, and approximately 1.35 L of DI water was used for the final rinse. Again, the pH of the effluent was observed to increase to approximately 14 just before the pause to refill the supply container (~60 mL of NaOH delivered to the column).

2.2 Characterization of CST

Aliquots of the as-received and pretreated material from each of the above pretreatments were analyzed by both thermogravimetric analysis (TGA) and inductively coupled plasma – emission spectroscopy (ICP-ES) to determine the extent of Na^+ exchange, except for the material from Test Pretreatment #1 which was not submitted for ICP-ES analysis.

2.2.1 TGA Analysis

TGA analysis was performed using a TA instruments TGA 2050. The protocol consisted of ramping to 400 °C at a rate of 5 °C/min, holding at 400 °C for 240 minutes, and finally ramping to 700 °C at a rate of 5 °C/min. The mass remaining at 460 °C was considered the true dry mass for purposes of calculating the F-factor (water content correction factor).

2.2.2 ICP-ES Analysis

Samples of the air-dried CST from Pretreatments #2 and #3 were submitted for digestion followed by ICP-ES analysis. The digestion was performed on ~0.1 g samples using hot HNO_3/HF acid. The solution generated was then analyzed by ICP-ES, and results were reported on a mass basis using the amount of solid digested.

2.3 Preparation of CST Standard

The digestion standard was prepared from aliquots of the same batch of pretreated CST to be used in the in-tank batch contact tests. Five individual aliquots (0.1 g each) of this material were submitted for hot HNO_3/HF digestion followed by inductively coupled plasma – mass spectrometry (ICP-MS) analysis to measure the key components of the CST (i.e., Ti, Zr, and Nb). The five digestions were performed in the Shielded Cells to match the conditions that will be utilized for digesting the material from the in-tank batch contact testing. In addition, the five digestions were performed on different days to capture day-to-day variability that is likely to be experienced in future digestions of the pretreated CST. An aliquot of this batch of pretreated CST was also submitted for X-ray fluorescence (XRF) analysis.

2.4 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. The Technical Task Request[4] identifies this work as Safety Significant. Consistent with what was described in the Task Technical and Quality Assurance Plan[5] this report received a technical review by design verification (document review) in accordance with E7 2.60, Section 5.3. The reviews are documented in the attached design verification reports. JMP Pro Version 11.2.1 commercial software[6] was used for calculations of the control limits described below. This software is classified as Level D;[7] however, the output included in this report was independently verified using an alternate software package as part of the design verification meeting the requirements for safety significant work.

3.0 Results and Discussion

3.1 Pretreatment Results

Table 3-1 provides a summary of the three pretreatments performed on the CST, and Table 3-2 provides a summary of the results of these pretreatments. Two methods were used to calculate the millimoles of sodium per gram of CST after pretreatment. The first method is based on the mass gain during the pretreatment. Assuming the observed mass increase is due to an increase in the fraction of sites occupied by Na^+ ions, the mass gain can be converted to the adsorption of Na^+ in terms of mmol Na^+/g of Na-form CST. These results are shown in the 6th column of Table 3-2 and are consistent with the value of 4.4 mmol Na^+/g_{CST} obtained previously for the pretreatment performed using the more exhaustive lab protocol (i.e. 65 bed volumes of 3 M NaOH).[8] In addition to calculating the conversion from the weight gain, samples of pretreated material from Test #2 and the final pretreatment were digested and analyzed for sodium content by ICP-ES, along with samples of the as-received material. The final column of Table 3-2 shows the total Na content of the pretreated materials. These values are similar to the values obtained based on mass difference; however, they are not directly comparable as the ICP-ES results show the total Na content including Na already present in the as-received material. ICP-ES analysis of the as-received samples indicated a Na/Ti molar ratio of 0.21 – 0.23, which increased to 1.03 – 1.09 after pretreatment. See Appendix B for the full ICP-ES results. The Na/Ti molar ratio in the pretreated material is very similar to the value calculated based on the theoretical formula provided by Nyman et. al., for the "leached" (i.e., NaOH treated) material (Na/Ti molar ratio of 1.07 for the formula $Na_3Si_2(Nb_{0.3}Ti_{0.7})_4O_{13}(OH)\cdot 4H_2O$).[9] The value for the as-received is slightly lower than expected based on a theoretical formula of $H_2NaSi_2(Nb_{0.3}Ti_{0.7})_4O_{13}(OH)\cdot 4H_2O$ for the acid form of the material (Na/Ti molar ratio = 0.36). Based on these results, it is believed that the as-received CST media was mainly in the hydrogen ionic form. The TGA weight loss profiles for the as-received and pretreated CST from lot 2099000035 (Pretreatments #1

and #2) are shown in Figure 3-1. The TGA weight loss profiles for as-received and pretreated CST from lot 2099000034 are shown in Figure 3-2 (Pretreatment #3).

Table 3-1. Summary of CST Pretreatments

Pretreatment #	Lot of As-Received Material Used	Mass of As-Received Material Used (g)	Mass loss at 460 °C (As-Received Material)	True Dry Starting Mass (g)
1	2099000035	29.4016	19.93%	23.5419
2	2099000035	29.4040		23.5438
3 (Final)	2099000034	29.4501	21.06%	23.2479

Table 3-2. Summary of Results of CST Pretreatments

Pretreatment #	Mass of Air-Dried Product (g)	Mass loss at 460 °C (Pre-treated Material)	Final True Dry CST Mass (g)	CST Mass Gain (g)	mmol Na^+/g_{CST}* (based on mass gain)	mmol Na^+/g_{CST}* (ICP-ES results)
1	32.1588	18.60%	26.1773	2.6354	4.58	Not Measured
2	32.3350	18.84%	26.2431	2.6993	4.68	4.25
3 (Final)	31.4017	18.09%	25.7211	2.4732	4.37	4.34

*Based on the true dry pretreated CST mass basis.

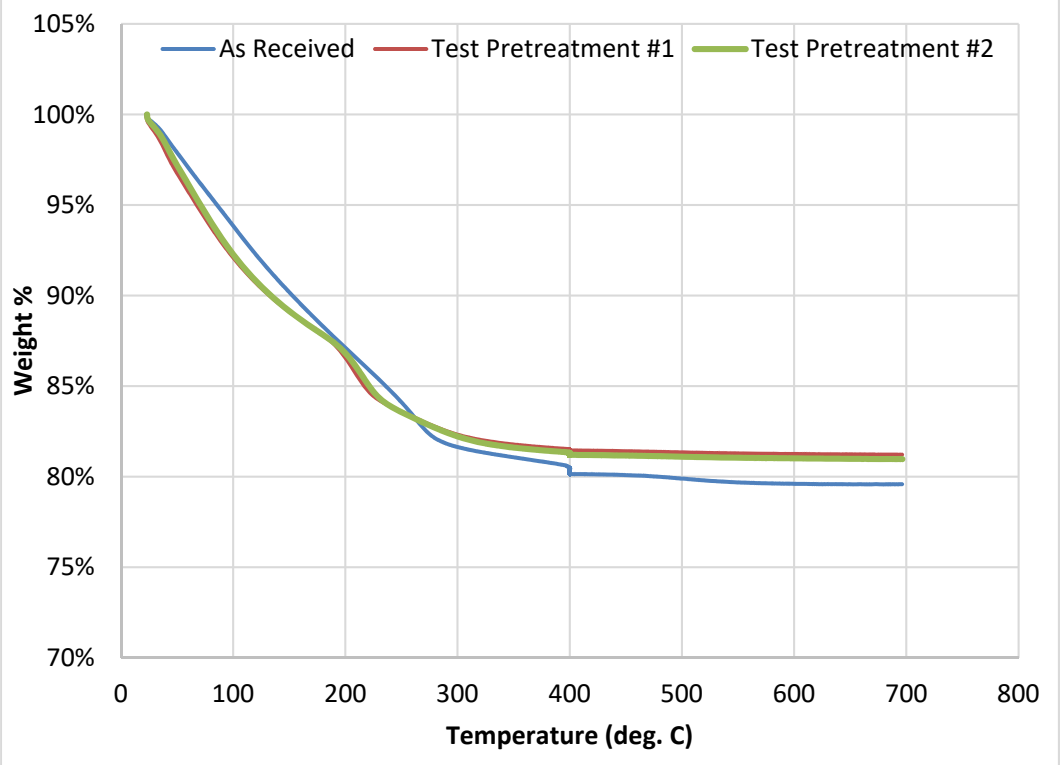

Figure 3-1. Thermogravimetric Analysis of As-Received and Pretreated CST Lot 2099000035.

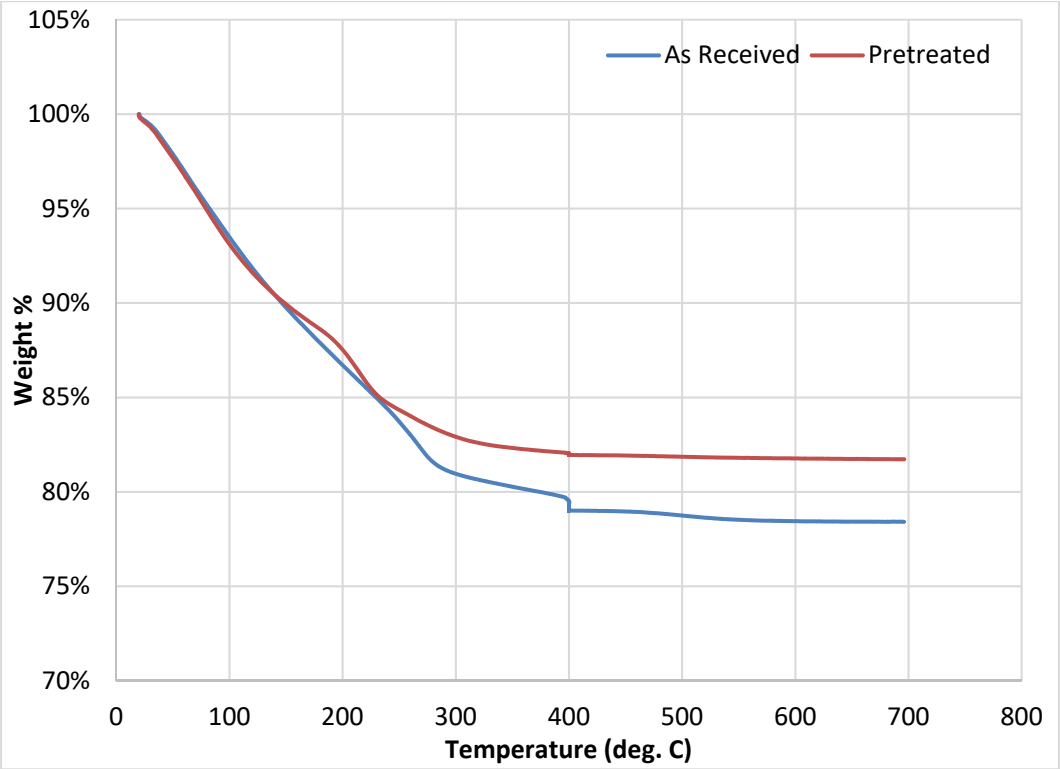

Figure 3-2. Thermogravimetric Analysis of CST Lot 2099000034 from Final Pretreatment (#3).

3.2 Evaluations of the Digestion Standard

Data obtained from the ICP-MS analysis described in Section 2.3 of the five sample digestions of the CST standard were used to develop a set of reference values and control limits for the concentrations of Ti, Zr, and Nb in future CST digestions. These data are provided in Table A-1 of Appendix A; JMP Pro Version 11.2.1[6] was used to conduct these evaluations. As stated previously, the five digestions were performed on different days to capture day-to-day variability that is likely to be experienced in future digestions of the pretreated CST. The ICP-MS analyses were conducted on the same day, leading to the within-day variation of the ICP-MS method to also be captured in the variation of the results of Table A-1. The following table provides descriptive statistics for these measurements.

Table 3-3. Summary Statistics for ICP-MS Measurement of Five Initial CST Digestions

Type of Data	Value
Number of Observations	5
Mean (Ti)	156000 (µg/g)
Mean (Zr)	100960 (µg/g)
Mean (Nb)	123200 (µg/g)
Standard Deviation (Ti)	6041.5 (µg/g)
Standard Deviation (Zr)	2628.3 (µg/g)
Standard Deviation (Nb)	3271.1 (µg/g)
% Relative Standard Deviation (%RSD) for Ti	3.87
%RSD (Zr)	2.60
%RSD (Nb)	2.66

* all concentrations provided on reference state (air dried) CST mass basis

The mean values for Ti, Zr, and Nb are consistent with prior analyses of CST.[9,10] A summary of this data is provided in Table 3-4, along with current data for comparison. As described in Section 2.3 a sample of this batch of CST was also submitted for XRF analysis, and those results are also presented in Table 3-4. The XRF analysis is performed on the solid material (i.e., no digestion is performed), and consistency between the XRF results and the ICP-MS results provides further confidence the material is being fully digested prior to analysis.

Table 3-4. Comparison of CST Component Concentrations

	Ti (wt %)	Zr (wt %)	Nb (wt %)
ICP-MS of Standards (mean data from Table 3-3)	15.60	10.10	12.32
XRF analysis (current work)	16.11	10.11	12.01
Walker, averages for IE-911 CST (ICP-ES)[10]	16.28	9.82	12.78
Nyman, Baseline CST (DCP*)[9]	17.78	11.24	15.03
Nyman, Leached CST (DCP*)[9]	16.27	10.21	13.17

*DCP = Direct Current Plasma Spectroscopy

While the summary statistics seen in Table 3-3 provide estimates of day-to-day variation in the digestion process, estimates of day-to-day variation of the ICP-MS method in the measurement of Ti, Zr, and Nb are needed to construct limits for statistical process control (SPC) charts. Historical measurements of laboratory standards utilized as part of routine ICP-MS analyses were used to address this issue. These data, which include measurements of opening and closing standards grouped by "originating file," are provided in Table A-2 of Appendix A. Measurements of Cs, Nb, Ti, and Zr are provided in this table, and these data are analyzed in Exhibit A-1 and Exhibit A-2 of Appendix A♣. This pair of exhibits provides an analysis of variance of a random effects model for each element. Included in the exhibits is an estimate of the variance of the measurements across the originating files, which is assumed to represent a day-to-day effect on the ICP-MS measurements. These results may be expressed as a percent day-to-day relative standard deviation (%RSD) for each of the elements: 1.541% for Cs, 1.360% for Nb, 1.689% for Ti, and 1.570% for Zr.

The results in Table 3-3 and in the exhibits of Appendix A are utilized to establish the centerline and 3-sigma limits for the SPC chart for each element. The centerline is the mean value from the table: 123200 for Nb, 156000 for Ti, and 100960 for Zr (all values in µg/g). The estimate of "sigma" (i.e., the total standard deviation) underlying each of these charts is determined as the square root of the sum of the day-to-day variance and the within-day variance for each element. These variances are expressed as %RSDs in the following table with the Lower Confidence Limit (LCL) and Upper Confidence Limit (UCL) determined as 3×sigma (99.7%) below and above the centerline, respectively.

Table 3-5. Determining Parameters for SPC Charts

Element	Day-to-Day %RSD	CST Digestion & Within Day %RSD	CST Avg µg/g (centerline)	Total %RSD for CST	99.7% LCL µg/g	99.7% UCL µg/g
Nb	1.360	2.655	123200	2.983	112170	134230
Ti	1.689	3.873	156000	4.225	136230	175770
Zr	1.570	2.603	100960	3.040	91750	110170

For each in-tank batch contact test sample, an aliquot of the CST digestion standard will be processed alongside and Ti, Zr, and Nb concentrations as measured by ICP-MS will be compared against the control limits (LCL to UCL range) established here to confirm complete digestion of the sample.

♣ The Cs results are provided as a point of comparison only. The CST standard does not contain Cs.

4.0 Conclusions

A procedure documenting the protocol for pretreating CST utilizing conditions similar to what will be performed in the field was developed and validated, confirming similar conversion to the Na^+ form of the media was obtained as seen in prior, more exhaustive pretreatments. The pretreated CST material to be used in the in-tank batch contact testing was determined to have a sodium loading of 4.34 mmol Na^+/g_{CST} as measured by ICP-ES analysis of the digested material. While similar conversion to the Na^+ form was demonstrated with the field pretreatment protocol, leaching of Nb and Si from the material during the pretreatment was not evaluated. ICP-MS analysis of the digested pretreated material does show Nb concentrations more similar to the leached versus baseline IE-911 material characterized by Nyman[9] however.

In addition, control limits have been established for the measured Ti, Zr, and Nb concentrations in digested CST material. For each in-tank batch contact sample received, an aliquot of the CST standard material will be digested alongside, and the ICP-MS measured concentrations of Ti, Zr, and Nb will be compared against the control limits established here to ensure complete digestion has occurred.

5.0 Future Work

The potential for refinement of these control limits by inclusion of additional data from future digestions will be evaluated as more data is obtained.

6.0 References

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Appendix A. Supporting Tables and Exhibits

Table A-1. ICP-MS Analytical Data for Samples of the CST Digestion Standard.

Sample	Nb (µg/g)	Ti (µg/g)	Zr (µg/g)
LW12103 CST St	125000	161000	102000
LW12104 CST St	122000	155000	100000
LW12105 CST St	126000	158000	103000
LW12106 CST St	125000	160000	103000
LW12107 CST St	118000	146000	96800

Table A-2. Historical Measurements of ICP-MS Opening and Closing Standards.

Upload Date	Analysis Date	Originating File	Opening (O)/ Closing (C)	mass	Analyte	Measurement	Reference Value	% relative difference
07/09/2018 1:52 PM	07/03/2018	Wells 10039,10643.xlsm	O	m/z = 47	Ti	0.7632	0.744	2.580
07/09/2018 1:52 PM	07/03/2018	Wells 10039,10643.xlsm	O	m/z = 90	Zr	5.1561	5.145	0.216
07/09/2018 1:52 PM	07/03/2018	Wells 10039,10643.xlsm	O	m/z = 93	Nb	10.1424	10	1.424
07/09/2018 1:52 PM	07/03/2018	Wells 10039,10643.xlsm	O	m/z = 133	Cs	10.2474	10	2.474
07/09/2018 1:52 PM	07/03/2018	Wells 10039,10643.xlsm	C	m/z = 47	Ti	0.7981	0.744	7.273
07/09/2018 1:52 PM	07/03/2018	Wells 10039,10643.xlsm	C	m/z = 90	Zr	5.2345	5.145	1.739
07/09/2018 1:52 PM	07/03/2018	Wells 10039,10643.xlsm	C	m/z = 93	Nb	10.0960	10	0.960
07/09/2018 1:52 PM	07/03/2018	Wells 10039,10643.xlsm	C	m/z = 133	Cs	10.1485	10	1.485
07/10/2018 5:00 PM	07/09/2018	King 10777,79,81,83,85,87.xlsm	O	m/z = 47	Ti	0.7247	0.744	-2.598
07/10/2018 5:00 PM	07/09/2018	King 10777,79,81,83,85,87.xlsm	O	m/z = 90	Zr	5.0785	5.145	-1.293
07/10/2018 5:00 PM	07/09/2018	King 10777,79,81,83,85,87.xlsm	O	m/z = 93	Nb	9.9734	10	-0.266
07/10/2018 5:00 PM	07/09/2018	King 10777,79,81,83,85,87.xlsm	O	m/z = 133	Cs	10.2198	10	2.198
07/10/2018 5:00 PM	07/09/2018	King 10777,79,81,83,85,87.xlsm	C	m/z = 47	Ti	0.7489	0.744	0.654
07/10/2018 5:00 PM	07/09/2018	King 10777,79,81,83,85,87.xlsm	C	m/z = 90	Zr	5.1031	5.145	-0.814
07/10/2018 5:00 PM	07/09/2018	King 10777,79,81,83,85,87.xlsm	C	m/z = 93	Nb	10.0327	10	0.327
07/10/2018 5:00 PM	07/09/2018	King 10777,79,81,83,85,87.xlsm	C	m/z = 133	Cs	10.3251	10	3.251
07/11/2018 3:14 PM	07/10/2018	King 10571,73,75,77,79,81,83,84.xlsm	O	m/z = 47	Ti	0.7767	0.744	4.391
07/11/2018 3:14 PM	07/10/2018	King 10571,73,75,77,79,81,83,84.xlsm	O	m/z = 90	Zr	5.1908	5.145	0.889
07/11/2018 3:14 PM	07/10/2018	King 10571,73,75,77,79,81,83,84.xlsm	O	m/z = 93	Nb	10.0580	10	0.580
07/11/2018 3:14 PM	07/10/2018	King 10571,73,75,77,79,81,83,84.xlsm	O	m/z = 133	Cs	10.0269	10	0.269
07/11/2018 3:14 PM	07/10/2018	King 10571,73,75,77,79,81,83,84.xlsm	C	m/z = 47	Ti	0.7877	0.744	5.867
07/11/2018 3:14 PM	07/10/2018	King 10571,73,75,77,79,81,83,84.xlsm	C	m/z = 90	Zr	5.1830	5.145	0.739
07/11/2018 3:14 PM	07/10/2018	King 10571,73,75,77,79,81,83,84.xlsm	C	m/z = 93	Nb	10.1529	10	1.529
07/11/2018 3:14 PM	07/10/2018	King 10571,73,75,77,79,81,83,84.xlsm	C	m/z = 133	Cs	10.0496	10	0.496
07/19/2018 5:40 PM	07/13/2018	Doman 10687,10688.xlsm	O	m/z = 47	Ti	0.7535	0.744	1.278
07/19/2018 5:40 PM	07/13/2018	Doman 10687,10688.xlsm	O	m/z = 90	Zr	5.0801	5.145	-1.262
07/19/2018 5:40 PM	07/13/2018	Doman 10687,10688.xlsm	O	m/z = 93	Nb	9.8804	10	-1.196
07/19/2018 5:40 PM	07/13/2018	Doman 10687,10688.xlsm	O	m/z = 133	Cs	10.0653	10	0.653
07/19/2018 5:40 PM	07/13/2018	Doman 10687,10688.xlsm	C	m/z = 47	Ti	0.7371	0.744	-0.931
07/19/2018 5:40 PM	07/13/2018	Doman 10687,10688.xlsm	C	m/z = 90	Zr	5.0637	5.145	-1.581
07/19/2018 5:40 PM	07/13/2018	Doman 10687,10688.xlsm	C	m/z = 93	Nb	9.9579	10	-0.421
07/19/2018 5:40 PM	07/13/2018	Doman 10687,10688.xlsm	C	m/z = 133	Cs	10.1042	10	1.042
07/26/2018 12:28 PM	07/23/2018	King 10870,72,74,76,78,80,82,83.xlsm	O	m/z = 47	Ti	0.7317	0.744	-1.657
07/26/2018 12:28 PM	07/23/2018	King 10870,72,74,76,78,80,82,83.xlsm	O	m/z = 90	Zr	5.1218	5.145	-0.450
07/26/2018 12:28 PM	07/23/2018	King 10870,72,74,76,78,80,82,83.xlsm	O	m/z = 93	Nb	9.8677	10	-1.323
07/26/2018 12:28 PM	07/23/2018	King 10870,72,74,76,78,80,82,83.xlsm	O	m/z = 133	Cs	10.1365	10	1.365
07/26/2018 12:28 PM	07/23/2018	King 10870,72,74,76,78,80,82,83.xlsm	C	m/z = 47	Ti	0.7450	0.744	0.137
07/26/2018 12:28 PM	07/23/2018	King 10870,72,74,76,78,80,82,83.xlsm	C	m/z = 90	Zr	5.1002	5.145	-0.871
07/26/2018 12:28 PM	07/23/2018	King 10870,72,74,76,78,80,82,83.xlsm	C	m/z = 93	Nb	9.7510	10	-2.490
07/26/2018 12:28 PM	07/23/2018	King 10870,72,74,76,78,80,82,83.xlsm	C	m/z = 133	Cs	10.0954	10	0.954
07/27/2018 3:11 PM	07/26/2018	Crawford 10341,44,47,50.xlsm	O	m/z = 47	Ti	0.7503	0.744	0.844
07/27/2018 3:11 PM	07/26/2018	Crawford 10341,44,47,50.xlsm	O	m/z = 90	Zr	5.0004	5.145	-2.811
07/27/2018 3:11 PM	07/26/2018	Crawford 10341,44,47,50.xlsm	O	m/z = 93	Nb	9.8160	10	-1.840
07/27/2018 3:11 PM	07/26/2018	Crawford 10341,44,47,50.xlsm	O	m/z = 133	Cs	10.0254	10	0.254
07/27/2018 3:11 PM	07/26/2018	Crawford 10341,44,47,50.xlsm	C	m/z = 47	Ti	0.7526	0.744	1.153
07/27/2018 3:11 PM	07/26/2018	Crawford 10341,44,47,50.xlsm	C	m/z = 90	Zr	4.9508	5.145	-3.774
07/27/2018 3:11 PM	07/26/2018	Crawford 10341,44,47,50.xlsm	C	m/z = 93	Nb	9.7743	10	-2.257

Upload Date	Analysis Date	Originating File	Opening (O)/ Closing (C)	mass	Analyte	Measurement	Reference Value	% relative difference
07/27/2018 3:11 PM	07/26/2018	Crawford 10341,44,47,50.xlsm	C	m/z = 133	Cs	10.0251	10	0.251

Table A-2. Historical Measurements of ICP-MS Opening and Closing Standards *(continued)*

Upload Date	Analysis Date	Originating File	Opening (O)/ Closing (C)	mass	Analyte	Measurement	Reference Value	% relative difference
07/29/2018 11:07 AM	07/27/2018	King 11001,03,05,07,09,11,13.xlsm	O	m/z = 47	Ti	0.7614	0.744	2.337
07/29/2018 11:07 AM	07/27/2018	King 11001,03,05,07,09,11,13.xlsm	O	m/z = 90	Zr	5.0453	5.145	-1.939
07/29/2018 11:07 AM	07/27/2018	King 11001,03,05,07,09,11,13.xlsm	O	m/z = 93	Nb	9.9422	10	-0.578
07/29/2018 11:07 AM	07/27/2018	King 11001,03,05,07,09,11,13.xlsm	O	m/z = 133	Cs	10.0889	10	0.889
07/29/2018 11:07 AM	07/27/2018	King 11001,03,05,07,09,11,13.xlsm	C	m/z = 47	Ti	0.7716	0.744	3.711
07/29/2018 11:07 AM	07/27/2018	King 11001,03,05,07,09,11,13.xlsm	C	m/z = 90	Zr	5.1321	5.145	-0.251
07/29/2018 11:07 AM	07/27/2018	King 11001,03,05,07,09,11,13.xlsm	C	m/z = 93	Nb	10.0079	10	0.079
07/29/2018 11:07 AM	07/27/2018	King 11001,03,05,07,09,11,13.xlsm	C	m/z = 133	Cs	9.9412	10	-0.588
08/01/2018 12:31 PM	07/31/2018	King 10828,30,32,34,36,38,40.xlsm	O	m/z = 47	Ti	0.7602	0.744	2.175
08/01/2018 12:31 PM	07/31/2018	King 10828,30,32,34,36,38,40.xlsm	O	m/z = 90	Zr	5.0482	5.145	-1.882
08/01/2018 12:31 PM	07/31/2018	King 10828,30,32,34,36,38,40.xlsm	O	m/z = 93	Nb	9.8359	10	-1.641
08/01/2018 12:31 PM	07/31/2018	King 10828,30,32,34,36,38,40.xlsm	O	m/z = 133	Cs	9.8221	10	-1.779
08/01/2018 12:31 PM	07/31/2018	King 10828,30,32,34,36,38,40.xlsm	C	m/z = 47	Ti	0.7619	0.744	2.400
08/01/2018 12:31 PM	07/31/2018	King 10828,30,32,34,36,38,40.xlsm	C	m/z = 90	Zr	5.0667	5.145	-1.522
08/01/2018 12:31 PM	07/31/2018	King 10828,30,32,34,36,38,40.xlsm	C	m/z = 93	Nb	9.9234	10	-0.766
08/01/2018 12:31 PM	07/31/2018	King 10828,30,32,34,36,38,40.xlsm	C	m/z = 133	Cs	9.7741	10	-2.259
08/03/2018 11:22 AM	08/01/2018	King 11035,37,39.xlsm	O	m/z = 47	Ti	0.7597	0.744	2.110
08/03/2018 11:22 AM	08/01/2018	King 11035,37,39.xlsm	O	m/z = 90	Zr	5.0559	5.145	-1.732
08/03/2018 11:22 AM	08/01/2018	King 11035,37,39.xlsm	O	m/z = 93	Nb	9.7225	10	-2.775
08/03/2018 11:22 AM	08/01/2018	King 11035,37,39.xlsm	O	m/z = 133	Cs	9.8450	10	-1.550
08/03/2018 11:22 AM	08/01/2018	King 11035,37,39.xlsm	C	m/z = 47	Ti	0.7528	0.744	1.176
08/03/2018 11:22 AM	08/01/2018	King 11035,37,39.xlsm	C	m/z = 90	Zr	5.0771	5.145	-1.319
08/03/2018 11:22 AM	08/01/2018	King 11035,37,39.xlsm	C	m/z = 93	Nb	9.8375	10	-1.625
08/03/2018 11:22 AM	08/01/2018	King 11035,37,39.xlsm	C	m/z = 133	Cs	9.8520	10	-1.480
08/10/2018 9:56 AM	08/01/2018	King 11134.xlsm	O	m/z = 47	Ti	0.7410	0.744	-0.409
08/10/2018 9:56 AM	08/01/2018	King 11134.xlsm	O	m/z = 90	Zr	4.9713	5.145	-3.376
08/10/2018 9:56 AM	08/01/2018	King 11134.xlsm	O	m/z = 93	Nb	9.7432	10	-2.568
08/10/2018 9:56 AM	08/01/2018	King 11134.xlsm	O	m/z = 133	Cs	9.7447	10	-2.553
08/10/2018 9:56 AM	08/01/2018	King 11134.xlsm	C	m/z = 47	Ti	0.7603	0.744	2.193
08/10/2018 9:56 AM	08/01/2018	King 11134.xlsm	C	m/z = 90	Zr	5.0905	5.145	-1.060
08/10/2018 9:56 AM	08/01/2018	King 11134.xlsm	C	m/z = 93	Nb	9.8936	10	-1.064
08/10/2018 9:56 AM	08/01/2018	King 11134.xlsm	C	m/z = 133	Cs	9.7426	10	-2.574
08/16/2018 11:13 AM	08/13/2018	King 11144,46,48,50.xlsm	O	m/z = 47	Ti	0.7425	0.744	-0.198
08/16/2018 11:13 AM	08/13/2018	King 11144,46,48,50.xlsm	O	m/z = 90	Zr	4.9565	5.145	-3.663
08/16/2018 11:13 AM	08/13/2018	King 11144,46,48,50.xlsm	O	m/z = 93	Nb	10.0676	10	0.676
08/16/2018 11:13 AM	08/13/2018	King 11144,46,48,50.xlsm	O	m/z = 133	Cs	10.1837	10	1.837
08/16/2018 11:13 AM	08/13/2018	King 11144,46,48,50.xlsm	C	m/z = 47	Ti	0.7157	0.744	-3.806
08/16/2018 11:13 AM	08/13/2018	King 11144,46,48,50.xlsm	C	m/z = 90	Zr	4.8780	5.145	-5.189
08/16/2018 11:13 AM	08/13/2018	King 11144,46,48,50.xlsm	C	m/z = 93	Nb	9.8697	10	-1.303
08/16/2018 11:13 AM	08/13/2018	King 11144,46,48,50.xlsm	C	m/z = 133	Cs	10.0195	10	0.195
08/23/2018 11:46 AM	08/16/2018	Rudisill 10808,20,11089,92.xlsm	O	m/z = 47	Ti	0.7438	0.744	-0.027
08/23/2018 11:46 AM	08/16/2018	Rudisill 10808,20,11089,92.xlsm	O	m/z = 90	Zr	4.9924	5.145	-2.966
08/23/2018 11:46 AM	08/16/2018	Rudisill 10808,20,11089,92.xlsm	O	m/z = 93	Nb	9.9917	10	-0.083
08/23/2018 11:46 AM	08/16/2018	Rudisill 10808,20,11089,92.xlsm	O	m/z = 133	Cs	10.2029	10	2.029
08/23/2018 11:46 AM	08/16/2018	Rudisill 10808,20,11089,92.xlsm	C	m/z = 47	Ti	0.7161	0.744	-3.747

08/23/2018 11:46 AM	08/16/2018	Rudisill 10808,20,11089,92.xlsm	C	m/z = 90	Zr	4.8224	5.145	-6.270
08/23/2018 11:46 AM	08/16/2018	Rudisill 10808,20,11089,92.xlsm	C	m/z = 93	Nb	9.5272	10	-4.728
08/23/2018 11:46 AM	08/16/2018	Rudisill 10808,20,11089,92.xlsm	C	m/z = 133	Cs	10.0590	10	0.590

Table A-2. Historical Measurements of ICP-MS Opening and Closing Standards *(continued)*

Upload Date	Analysis Date	Originating File	Opening (O)/ Closing (C)	mass	Analyte	Measurement	Reference Value	% relative difference
08/28/2018 5:13 PM	08/27/2018	Stevenson 10929,30,10936-39.xlsm	O	m/z = 47	Ti	0.7599	0.744	2.142
08/28/2018 5:13 PM	08/27/2018	Stevenson 10929,30,10936-39.xlsm	O	m/z = 90	Zr	4.9096	5.145	-4.576
08/28/2018 5:13 PM	08/27/2018	Stevenson 10929,30,10936-39.xlsm	O	m/z = 93	Nb	10.1971	10	1.971
08/28/2018 5:13 PM	08/27/2018	Stevenson 10929,30,10936-39.xlsm	O	m/z = 133	Cs	10.2160	10	2.160
08/28/2018 5:13 PM	08/27/2018	Stevenson 10929,30,10936-39.xlsm	C	m/z = 47	Ti	0.7414	0.744	-0.353
08/28/2018 5:13 PM	08/27/2018	Stevenson 10929,30,10936-39.xlsm	C	m/z = 90	Zr	5.3495	5.145	3.975
08/28/2018 5:13 PM	08/27/2018	Stevenson 10929,30,10936-39.xlsm	C	m/z = 93	Nb	10.1701	10	1.701
08/28/2018 5:13 PM	08/27/2018	Stevenson 10929,30,10936-39.xlsm	C	m/z = 133	Cs	10.1631	10	1.631
09/06/2018 12:38 PM	09/04/2018	Bennett 10970,10971.xlsm	O	m/z = 47	Ti	0.7566	0.744	1.697
09/06/2018 12:38 PM	09/04/2018	Bennett 10970,10971.xlsm	O	m/z = 90	Zr	5.3071	5.145	3.151
09/06/2018 12:38 PM	09/04/2018	Bennett 10970,10971.xlsm	O	m/z = 93	Nb	9.7045	10	-2.955
09/06/2018 12:38 PM	09/04/2018	Bennett 10970,10971.xlsm	O	m/z = 133	Cs	10.0023	10	0.023
09/06/2018 12:38 PM	09/04/2018	Bennett 10970,10971.xlsm	C	m/z = 47	Ti	0.7570	0.744	1.744
09/06/2018 12:38 PM	09/04/2018	Bennett 10970,10971.xlsm	C	m/z = 90	Zr	5.3690	5.145	4.354
09/06/2018 12:38 PM	09/04/2018	Bennett 10970,10971.xlsm	C	m/z = 93	Nb	9.6542	10	-3.458
09/06/2018 12:38 PM	09/04/2018	Bennett 10970,10971.xlsm	C	m/z = 133	Cs	9.9550	10	-0.450
09/13/2018 12:47 PM	09/11/2018	Diprete 11658-667,681.xlsm	O	m/z = 47	Ti	0.7461	0.744	0.278
09/13/2018 12:47 PM	09/11/2018	Diprete 11658-667,681.xlsm	O	m/z = 90	Zr	5.0962	5.145	-0.949
09/13/2018 12:47 PM	09/11/2018	Diprete 11658-667,681.xlsm	O	m/z = 93	Nb	10.2148	10	2.148
09/13/2018 12:47 PM	09/11/2018	Diprete 11658-667,681.xlsm	O	m/z = 133	Cs	10.2159	10	2.159
09/13/2018 12:47 PM	09/11/2018	Diprete 11658-667,681.xlsm	C	m/z = 47	Ti	0.7297	0.744	-1.923
09/13/2018 12:47 PM	09/11/2018	Diprete 11658-667,681.xlsm	C	m/z = 90	Zr	5.0916	5.145	-1.037
09/13/2018 12:47 PM	09/11/2018	Diprete 11658-667,681.xlsm	C	m/z = 93	Nb	10.0178	10	0.178
09/13/2018 12:47 PM	09/11/2018	Diprete 11658-667,681.xlsm	C	m/z = 133	Cs	10.1334	10	1.334
09/20/2018 3:06 PM	09/10/2018	Rudisill 11362,363,578.xlsm	O	m/z = 47	Ti	0.7579	0.744	1.863
09/20/2018 3:06 PM	09/10/2018	Rudisill 11362,363,578.xlsm	O	m/z = 90	Zr	5.1796	5.145	0.672
09/20/2018 3:06 PM	09/10/2018	Rudisill 11362,363,578.xlsm	O	m/z = 93	Nb	10.1941	10	1.941
09/20/2018 3:06 PM	09/10/2018	Rudisill 11362,363,578.xlsm	O	m/z = 133	Cs	10.1981	10	1.981
09/20/2018 3:06 PM	09/10/2018	Rudisill 11362,363,578.xlsm	C	m/z = 47	Ti	0.7594	0.744	2.068
09/20/2018 3:06 PM	09/10/2018	Rudisill 11362,363,578.xlsm	C	m/z = 90	Zr	5.0486	5.145	-1.875
09/20/2018 3:06 PM	09/10/2018	Rudisill 11362,363,578.xlsm	C	m/z = 93	Nb	9.8969	10	-1.031
09/20/2018 3:06 PM	09/10/2018	Rudisill 11362,363,578.xlsm	C	m/z = 133	Cs	10.0935	10	0.935
09/20/2018 7:05 PM	09/13/2018	Rudisill 11358-361,577.xlsm	O	m/z = 47	Ti	0.7565	0.744	1.681
09/20/2018 7:05 PM	09/13/2018	Rudisill 11358-361,577.xlsm	O	m/z = 90	Zr	5.1991	5.145	1.051
09/20/2018 7:05 PM	09/13/2018	Rudisill 11358-361,577.xlsm	O	m/z = 93	Nb	10.1881	10	1.881
09/20/2018 7:05 PM	09/13/2018	Rudisill 11358-361,577.xlsm	O	m/z = 133	Cs	10.3682	10	3.682
09/20/2018 7:05 PM	09/13/2018	Rudisill 11358-361,577.xlsm	C	m/z = 47	Ti	0.7337	0.744	-1.387
09/20/2018 7:05 PM	09/13/2018	Rudisill 11358-361,577.xlsm	C	m/z = 90	Zr	5.1307	5.145	-0.278
09/20/2018 7:05 PM	09/13/2018	Rudisill 11358-361,577.xlsm	C	m/z = 93	Nb	10.1733	10	1.733
09/20/2018 7:05 PM	09/13/2018	Rudisill 11358-361,577.xlsm	C	m/z = 133	Cs	10.3004	10	3.004

Exhibit A-1. Random Effects Analysis of ICP-MS Opening and Closing Standards for Cs and Ti.

Response % rel diff mass=m/z = 133, Analyte=Cs
Whole Model

Summary of Fit

RSquare	0.924938
RSquare Adj	0.924938
Root Mean Square Error	0.603489
Mean of Response	0.703083
Observations (or Sum Wgts)	34

Parameter Estimates

Term	Estimate	Std Error	DFDen	t Ratio	Prob>|t|	Lower 95%	Upper 95%
Intercept	0.7030831	0.387823	16	1.81	0.0887	-0.119065	1.5252316

REML Variance Component Estimates

Random Effect	Var Ratio	Var Component	Std Error	95% Lower	95% Upper	Pct of Total
Originating File	6.5206586	2.3748171	0.9061617	0.5987728	4.1508614	86.703
Residual		0.364199	0.1249192	0.2050737	0.8185127	13.297
Total		2.7390161	0.9061617	1.5694932	5.9481903	100.000

%RSD for day-to-day effect = 1.541%

Response % rel diff mass=m/z = 47, Analyte=Ti
Whole Model

Summary of Fit

RSquare	0.649715
RSquare Adj	0.649715
Root Mean Square Error	1.71444
Mean of Response	1.021123
Observations (or Sum Wgts)	34

Parameter Estimates

Term	Estimate	Std Error	DFDen	t Ratio	Prob>|t|	Lower 95%	Upper 95%
Intercept	1.0211226	0.50426	16	2.02	0.0599	-0.047862	2.0901069

REML Variance Component Estimates

Random Effect	Var Ratio	Var Component	Std Error	95% Lower	95% Upper	Pct of Total
Originating File	0.9706652	2.8530821	1.6093037	-0.301095	6.0072593	49.256
Residual		2.939306	1.0081736	1.655069	6.6058924	50.744
Total		5.792388	1.6093037	3.5897232	10.892366	100.000

%RSD for day-to-day effect = 1.689%

Exhibit A-2. Random Effects Analysis of ICP-MS Opening and Closing Standards for Zr and Nb.

Response % rel diff mass=m/z = 90, Analyte=Zr
Whole Model

Summary of Fit

RSquare	0.596865
RSquare Adj	0.596865
Root Mean Square Error	1.785285
Mean of Response	-1.0574
Observations (or Sum Wgts)	34

Parameter Estimates

Term	Estimate	Std Error	DFDen	t Ratio	Prob>|t|	Lower 95%	Upper 95%
Intercept	-1.057403	0.488633	16	-2.16	0.0459*	-2.09326	-0.021547

REML Variance Component Estimates

Random Effect	Var Ratio	Var Component	Std Error	95% Lower	95% Upper	Pct of Total
Originating File	0.7735036	2.4653432	1.5356362	-0.544448	5.4751348	43.614
Residual		3.1872419	1.093215	1.7946771	7.1631117	56.386
Total		5.6525851	1.5356362	3.53598	10.458924	100.000

%RSD for day-to-day effect = 1.570%

Response % rel diff mass=m/z = 93, Analyte=Nb
Whole Model

Summary of Fit

RSquare	0.718787
RSquare Adj	0.718787
Root Mean Square Error	1.174905
Mean of Response	-0.50704
Observations (or Sum Wgts)	34

Parameter Estimates

Term	Estimate	Std Error	DFDen	t Ratio	Prob>|t|	Lower 95%	Upper 95%
Intercept	-0.507044	0.386621	16	-1.31	0.2082	-1.326643	0.3125553

REML Variance Component Estimates

Random Effect	Var Ratio	Var Component	Std Error	95% Lower	95% Upper	Pct of Total
Originating File	1.3408289	1.8508831	0.9290764	0.0299267	3.6718394	57.280
Residual		1.3804022	0.473474	0.777279	3.1023609	42.720
Total		3.2312852	0.9290764	1.9734914	6.2343838	100.000

%RSD for day-to-day effect = 1.360%

Appendix B. ICP-ES Results

Table B-1. ICP-ES Results for As-Received and Pretreated CST from Lot # 2099000035

Element	Concentration in As-Received (µg/g)	One Sigma Uncertainty	Concentration in Pretreated* (µg/g)	One Sigma Uncertainty
Ag	< 850	n/a	< 850	n/a
Al	2340	10.1%	1790	10%
B	< 101	n/a	< 29.7	n/a
Ba	< 29.4	n/a	< 70.6	n/a
Be	< 0.504	n/a	< 3.81	n/a
Ca	889	10%	809	10%
Cd	< 4.35	n/a	< 2.67	n/a
Ce	< 49.8	n/a	< 39.2	n/a
Co	< 8.95	n/a	< 4.48	n/a
Cr	< 10.8	n/a	< 5.41	n/a
Cu	< 664	n/a	< 560	n/a
Fe	134	10.4%	130	10.3%
K	< 274	n/a	< 504	n/a
La	< 11.2	n/a	< 5.62	n/a
Li	< 15.7	n/a	< 7.86	n/a
Mg	250	10%	227	10%
Mn	< 1.46	n/a	< 0.733	n/a
Mo	< 40.4	n/a	< 20.3	n/a
Na	18100	10%	79300	10%
Ni	< 50.2	n/a	< 25.2	n/a
P	< 219	n/a	< 110	n/a
Pb	< 491	n/a	< 246	n/a
S	< 3470	n/a	< 2270	n/a
Sb	< 762	n/a	< 432	n/a
Sn	< 27	n/a	< 376	n/a
Sr	< 88.5	n/a	< 44.3	n/a
Ti	164000	10%	151000	10%
V	< 123	n/a	< 55.1	n/a
Zn	< 4.38	n/a	< 1.62	n/a
Zr	108000	10%	103000	10%

*Material from Pretreatment #2.

Table B-2. ICP-ES Results for As-Received and Pretreated CST from Lot # 2099000034

Element	Concentration in As-Received (µg/g)	One Sigma Uncertainty	Concentration in Pretreated* (µg/g)	One Sigma Uncertainty
Ag	< 16.2	n/a	< 16.1	n/a
Al	4300	10.1%	3140	10%
B	< 44.6	n/a	< 442	n/a
Ba	177	10%	166	10%
Be	< 0.505	n/a	< 0.5	n/a
Ca	1040	10%	978	10%
Cd	< 5.33	n/a	< 5.28	n/a
Ce	< 49.9	n/a	< 49.4	n/a
Co	< 8.96	n/a	< 8.87	n/a
Cr	< 10.8	n/a	< 10.7	n/a
Cu	< 344	n/a	< 332	n/a
Fe	188	10.6%	192	10.3%
K	< 600	n/a	< 492	n/a
La	< 11.2	n/a	< 11.1	n/a
Li	< 15.7	n/a	< 15.6	n/a
Mg	355	10.1%	324	10%
Mn	< 1.47	n/a	< 1.45	n/a
Mo	< 40.5	n/a	< 40.1	n/a
Na	17300	10.1%	81700	10.5%
Ni	< 50.3	n/a	< 49.8	n/a
P	< 220	n/a	< 218	n/a
Pb	< 491	n/a	< 487	n/a
S	< 4550	n/a	< 4500	n/a
Sb	< 963	n/a	< 963	n/a
Sn	< 278	n/a	< 275	n/a
Sr	< 88.6	n/a	< 87.8	n/a
Th	< 40.9	n/a	< 40.5	n/a
Ti	176000	10%	166000	10%
U	< 732	n/a	< 725	n/a
V	< 207.2	n/a	< 192	n/a
Zn	< 3.25	n/a	< 3.22	n/a
Zr	119000	10%	113000	10%

*Material from Pretreatment #3.

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