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IMPACT OF URANIUM AND THORIUM ON HIGH TiO_2 CONCENTRATION NUCLEAR WASTE GLASSES

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ABSTRACT

This study focused on the potential impacts of the addition of Crystalline Silicotitanate (CST) and Monosodium Titanate (MST) from the Small Column Ion Exchange (SCIX) process on the Defense Waste Processing Facility (DWPF) glass waste form and the applicability of the DWPF process control models. MST from the Salt Waste Processing Facility (SWPF) is also considered in the study. The KT08-series of glasses was designed to evaluate any impacts of the inclusion of uranium and thorium in glasses containing the SCIX components. All but one of the study glasses were found to be amorphous by X-ray diffraction (XRD). One of the slowly cooled glasses contained a small amount of trevorite, which is typically found in DWPF-type glasses and had no practical impact on the durability of the glass. The measured Product Consistency Test (PCT) responses for the study glasses and the viscosities of the glasses were well predicted by the current DWPF models. No unexpected issues were encountered when uranium and thorium were added to the glasses with SCIX components.

INTRODUCTION

The Savannah River Site (SRS) Liquid Waste contractor had planned to begin a process referred to as Small Column Ion Exchange (SCIX) to disposition salt solution in fiscal year 2014 (these plans have been put on hold at present). In the first step of the process, salt solution retrieved from various waste tanks will be struck with Monosodium Titanate (MST) to remove key actinides and Sr. The salt solution will then be processed using Rotary Micro Filtration (RMF) to remove the MST and any insoluble solids. The MST and insoluble solids will accumulate on the bottom of the waste tank. The filtrate from RMF will be fed to ion exchange columns, also in the waste tank, to remove the ^{137}Cs using Crystalline Silicotitanate (CST) resin. The decontaminated salt solution from SCIX will be sent to the Saltstone Facility for immobilization in grout. The ^{137}Cs-laden CST resin will be sluiced and ground for particle size reduction, then sent to the Defense Waste Processing Facility (DWPF) for immobilization in glass. These processes mirror the current disposition paths for streams associated with the Salt Waste Processing Facility (SWPF), which is under construction and will run concurrently with SCIX.

The MST and CST from the SCIX process will significantly increase the concentrations of Nb_2O_5, TiO_2, and ZrO_2 in the DWPF feed. Other constituents of MST and CST – Na_2O and SiO_2 – are already present in high concentrations in DWPF glass; thus their influences are well understood. The increased concentrations of Nb_2O_5, TiO_2, and ZrO_2 will likely have some impact on the properties and performance of the DWPF glass product. Properties such as the liquidus temperature, viscosity, and rate of melting of the glass may be impacted. The performance of the glass, particularly its chemical durability as it pertains to repository acceptance requirements, may also be impacted. The DWPF uses a set of semi-empirical and first-principles models referred to as the Product Composition Control System (PCCS)[1] to predict the properties and performance of a glass based on its composition since it is not possible to measure these attributes during processing. The objective of this study is to evaluate the impacts of the SCIX streams on the properties and performance of the DWPF glass product and on the applicability of the current process control models.

EXPERIMENTAL PROCEDURE

The KT08-series of compositions was selected to evaluate any impacts of the inclusion of uranium and thorium in glasses with the SCIX components. While the composition projections for the sludge batches with SCIX additions included uranium and thorium,[2] these components are typically removed from the glasses fabricated for the experimental studies[3-5] in order to minimize exposure to radioactivity. Several variability studies performed at the Savannah River National Laboratory (SRNL) in support of frit optimization for DWPF processing have shown that the properties of glasses fabricated with uranium and thorium are unlikely to differ significantly from those of their non-radioactive counterparts.[6-9] The KT08-series glasses were selected to further confirm these findings when the SCIX components are included, as well as to determine whether changes in the amounts of the non-radioactive components in the glass (as a function of the total glass composition) have any significant impacts on the properties or performance of the glass.

The basis for the KT08-series compositions was a series of projections of individual sludge batches incorporating the SCIX streams. Composition projections for sludge batches 8 through 17 were used,[10] and CST additions to Tank 40 were projected at the accelerated DWPF processing rate of 75 Sludge Receipt and Adjustment Tank (SRAT) batches per year (including MST) with the SWPF streams added. The final SRAT batch composition for each sludge batch was used, since these cases represent the maximum concentrations of CST in the sludge. The resulting ten sludge composition projections are given in Table I. Each projection is identified by the relevant sludge batch and SRAT batch number.

Table I. Projected Compositions (wt %) of the Final SRAT Batches of Sludge Batches 8 through 17, Including SCIX Streams, Used to Develop the KT08 Glass Compositions.

Oxide	SB08-69	SB09-79	SB10-80	SB11-70	SB12-71	SB13-66	SB14-74	SB15-91	SB16-38	SB17-35
Al_2O_3	14.25	12.68	10.85	12.29	17.00	17.86	12.51	10.96	12.14	12.51
BaO	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17
CaO	2.30	2.31	2.32	2.12	2.32	2.43	1.98	1.76	2.11	2.16
Ce_2O_3	0.70	0.70	0.62	0.53	0.35	0.27	0.17	0.17	0.44	0.54
Cr_2O_3	0.22	0.22	0.22	0.22	0.33	0.33	0.33	0.22	0.22	0.23
CuO	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.09	0.10
Fe_2O_3	29.72	28.22	27.86	30.08	23.87	22.28	20.88	19.97	27.31	30.27
K_2O	0.09	0.09	0.09	0.09	0.18	0.27	0.18	0.18	0.18	0.09
La_2O_3	0.26	0.18	0.18	0.18	0.18	0.09	0.09	0.09	0.18	0.18
MgO	0.37	0.37	0.37	0.38	0.38	0.25	0.25	0.25	0.25	0.26
MnO	4.73	4.17	4.37	2.64	2.54	2.93	1.64	2.10	1.27	0.90
Na_2O	25.08	27.05	27.47	26.69	26.56	26.30	25.62	27.15	24.50	23.42
Nb_2O_5	2.54	2.68	2.66	2.61	2.67	2.53	2.66	2.83	1.88	1.75
NiO	0.86	0.48	0.77	0.39	0.29	0.39	1.42	1.32	1.15	1.08
PbO	0.40	0.32	0.32	0.24	0.16	0.16	0.16	0.16	0.33	0.33
SiO_2	3.43	4.68	5.15	6.74	8.08	7.96	6.55	5.72	3.04	2.31
ThO_2	0.43	1.54	2.14	0.60	0.00	0.00	0.00	0.00	0.00	0.00
TiO_2	10.67	10.69	10.64	10.91	11.03	10.80	10.79	10.79	10.72	10.04
U_3O_8	1.41	0.80	1.16	0.54	1.25	2.32	11.97	13.54	12.13	11.87
ZnO	0.00	0.09	0.09	0.19	0.09	0.19	0.19	0.09	0.09	0.10
ZrO_2	2.27	2.47	2.46	2.32	2.46	2.36	2.35	2.47	1.78	1.70

Note that the sludge projections did not include sulfate concentrations; therefore, a SO_4^{2-} concentration of 1.0 wt % was assumed for each sludge batch. Noble metals are not typically tracked in sludge batch projections, although they may play some role in determining the properties and performance of the glass. Therefore, the noble metals Ag, Pd, Rh, and Ru, along with SO_4^{2-}, were added to the sludge compositions, followed by a normalization of the remaining components to 100 wt %. The concentrations of the noble metals were obtained from recent measurements of Sludge Batch 6 (on a total solids basis), which was considered to contain a high concentration of noble metals.[11]

A single frit composition was identified that produced a PCCS Measurement Acceptability Region (MAR) acceptable glass at a targeted waste loading of 40 wt % with each of the sludge composition projections given in Table I. The composition of this frit, which was labeled Frit 0607, is 10 wt % B_2O_3, 6 wt % Li_2O, 5 wt % Na_2O, and 79 wt % SiO_2. Each of the sludge compositions with SO_4^{2-} and noble metal oxides added was then combined with Frit 0607 at a waste loading of 40 wt % to give the targeted glass compositions for the study, which were labeled as the KT08-series.

Each of the study glasses was prepared from the proper proportions of reagent-grade metal oxides, carbonates, and boric acid in 200 g batches. The raw materials were thoroughly mixed and placed into platinum/gold, 250 ml crucibles. The batch was placed into a high-temperature furnace at the melt temperature of 1150 °C. The crucible was removed from the furnace after an isothermal hold for 1 hour. The glass was poured onto a clean, stainless steel plate and allowed to air cool (quench). The glass pour patty was used as a sampling stock for the various property measurements described below.

Approximately 25 g of each glass was heat-treated to simulate cooling along the centerline of a DWPF-type canister[12] to gauge the effects of thermal history on the product performance. This cooling schedule is referred to as the canister centerline cooled (CCC) heat treatment. Visual observations of both quenched and CCC glasses were documented.

Representative samples of each quenched and CCC glass were analyzed by XRD to identify any measureable crystallization. Chemical analysis was performed on a representative sample from each quenched glass to confirm that the as-fabricated glasses met the targeted compositions. Two dissolution techniques, sodium peroxide fusion (PF) and cesium hydroxide fusion (CH), were used to prepare the glass samples, in duplicate, for analysis. Each of the samples was analyzed, twice for each element of interest, by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES). Glass standards were also intermittently measured to assess the performance of the ICP-AES instrument over the course of these analyses.

The PCT Method-A[13] was performed in triplicate on each quenched and CCC glass to assess chemical durability. Also included in the experimental test matrix was the EA benchmark glass,[14] the Approved Reference Material (ARM) glass,[15] and blanks from the sample cleaning batch. Samples were ground, washed, and prepared according to the standard procedure.[13] Fifteen milliliters of Type-I ASTM water were added to 1.5 g of glass in stainless steel vessels. The vessels were closed, sealed, and placed in an oven at 90 ± 2 °C where the samples were maintained at temperature for 7 days. Once cooled, the resulting solutions were sampled (filtered and acidified), then labeled and analyzed by ICP-AES. Samples of a multi-element, standard solution were also included in the analytical plans as a check on the accuracy of the ICP-AES instruments used for these measurements. Normalized release rates were calculated based on the measured compositions using the average of the common logarithms of the leachate concentrations.

The viscosity of the glasses was measured following Procedure A of the ASTM C 965 standard.[16] Harrop and Orton high temperature rotating spindle viscometers were used with platinum crucibles and spindles. The viscometers were specially designed to operate with small quantities of glass to support measurements of radioactive glasses when necessary.[17,18] A well characterized standard glass was used to determine the appropriate spindle constants.[18,19] Measurements were taken over a range of temperatures from 1050 to 1250 °C in 50 °C intervals. Measurements at 1150 °C were taken at three different times during the procedure to provide an opportunity to identify the effects of any crystallization or volatilization that may have occurred during the test. The data were fit to a Fulcher equation[20,21] to provide a measured viscosity value at the nominal DWPF melt temperature of 1150 °C.

RESULTS AND DISCUSSION

Crystallization

Visual observations of the quenched versions of the KT08-series glasses identified no visible crystallization. All of the quenched glasses were found to be amorphous by XRD. For the CCC versions of the KT08-series glasses, visual observations identified a small amount of surface crystallization on compositions KT08-01, -02, and -03. All of the CCC glasses were found to be amorphous by XRD with the exception of glass KT08-07. This indicates that the volume of surface crystallization in compositions KT08-01, -02, and -03 was below the XRD detection limit. Glass KT08-07 contained a small amount of trevorite, which may have been difficult to identify visually in the bulk of the glass. Spinels, including trevorite, are the crystalline phase typically found in DWPF-type glasses and have been shown to have no practical impact on durability.[22]

Measured Composition

The measured composition data for the study glasses were carefully reviewed. Minor issues with some of the measurements for the glasses prepared by CH were identified. There was likely a minor batching error for CuO in composition KT08-01. Overall, there were no indications of any significant issues in the batching of the KT08-series glasses. Decisions were made regarding which preparation method would be used for each oxide in determining the average measured composition. These decisions are summarized in Table II.

Table II. Preparation Methods Used in Determining the Concentration of Individual Oxides in the KT08-Series Glasses.

Oxide	Preparation Method(s)		Oxide	Preparation Method(s)
Al_2O_3	CH and PF		MnO	CH and PF
B_2O_3	CH		Na_2O	CH
BaO	CH and PF		Nb_2O_5	PF
CaO	CH		NiO	PF
Ce_2O_3	CH		PbO	CH
Cr_2O_3	CH		SO_4^{2-}	CH
CuO	CH and PF		SiO_2	PF
Fe_2O_3	CH		ThO_2	CH
K_2O	CH		TiO_2	CH
La_2O_3	CH and PF		U_3O_8	CH
Li_2O	CH and PF		ZnO	CH and PF
MgO	CH and PF		ZrO_2	CH

The data resulting from the preparation methods listed for each oxide in Table II were averaged to determine a representative chemical composition for each glass. A sum of oxides was also computed for each glass based upon the measured values. Glasses KT08-01, -02, and -07 each had one measured value for SiO_2 that was an outlier. These values were omitted as the average SiO_2 concentrations were determined for these glasses. All of the sums of oxides for the KT08 glasses fall within the PCCS acceptable interval of 95 to 105 wt%. A statistical review of the measured versus targeted compositions, which is detailed elsewhere,[23] suggested only minor difficulties in meeting the targeted compositions for the KT08-series glasses, none of which should impact the outcome of the study. The measured composition of each of the KT08-series glasses is reported in Table III.

Table III. Measured Compositions (wt %) of the KT08-series glasses.

Oxide	KT08-01	KT08-02	KT08-03	KT08-04	KT08-05	KT08-06	KT08-07	KT08-08	KT08-09	KT08-10
Al_2O_3	6.06	5.36	4.66	5.28	7.22	7.59	5.35	4.59	5.21	5.34
B_2O_3	6.01	5.98	6.01	5.98	6.01	6.01	6.06	5.89	6.02	5.98
BaO	0.07	0.07	0.07	0.07	0.07	0.07	0.07	0.06	0.07	0.07
CaO	0.98	0.98	0.96	0.92	0.98	1.04	0.84	0.74	0.92	0.93
Ce_2O_3	0.29	0.30	0.27	0.24	0.17	0.13	0.11	0.10	0.20	0.23
Cr_2O_3	0.09	0.09	0.08	0.09	0.13	0.13	0.13	0.09	0.09	0.09
CuO	0.08	0.05	0.05	0.05	0.05	0.05	0.04	0.05	0.04	0.04
Fe_2O_3	11.82	11.03	10.85	11.79	9.65	8.94	8.32	7.94	10.73	12.11
K_2O	0.21	0.23	0.23	0.22	0.24	0.27	0.27	0.25	0.28	0.22
La_2O_3	0.09	0.06	0.06	0.07	0.07	0.04	0.04	0.03	0.07	0.07
Li_2O	3.53	3.51	3.47	3.44	3.46	3.43	3.55	3.47	3.51	3.45
MgO	0.15	0.14	0.15	0.14	0.15	0.11	0.10	0.09	0.10	0.11
MnO	1.88	1.62	1.74	1.04	1.02	1.17	0.65	0.82	0.50	0.36
Na_2O	13.32	13.92	14.19	13.88	13.99	13.75	13.58	14.02	13.01	12.60
Nb_2O_5	0.86	0.92	0.97	0.92	0.98	0.94	0.89	1.11	0.65	0.64
NiO	0.34	0.20	0.32	0.19	0.14	0.18	0.57	0.51	0.45	0.46
PbO	0.21	0.12	0.12	0.09	0.12	0.12	0.06	0.13	0.13	0.16
SiO_2	45.92	48.49	52.09	50.81	51.93	52.52	47.21	48.67	45.67	50.70
SO_4^{2-}	0.26	0.26	0.26	0.26	0.26	0.26	0.27	0.27	0.26	0.27
ThO_2	0.17	0.62	0.89	0.24	0.02	0.02	0.02	0.02	0.02	0.02
TiO_2	4.12	4.16	4.13	4.26	4.35	4.21	4.27	4.18	4.20	3.93
U_3O_8	0.60	0.44	0.63	0.24	0.50	0.77	3.73	5.22	4.86	4.54
ZnO	0.01	0.04	0.04	0.08	0.04	0.08	0.07	0.04	0.04	0.05
ZrO_2	0.82	0.81	0.82	0.74	0.91	0.86	0.77	0.90	0.57	0.62

Durability

One of the quality control checkpoints for the PCT procedure is solution mass loss over the course of the seven day test. Water loss was in the acceptable range for all of the KT08 PCT vessels. One of the vessels, the first replicate of the quenched version of glass KT08-04, had an insufficient amount of glass to meet the required ratio of leachant volume to mass of ground glass. Data for this vessel were omitted from further analyses. This omission does not impact the outcome of the study since each glass was tested in triplicate. All of the measurements of the ARM reference glass fell within the control ranges.[15] A statistical review of the ICP-AES measurements of the PCT leachates, which is detailed elsewhere,[23] suggested only minor scatter in the triplicate values for some analytes for some of the glasses.

The PCT leachate concentrations were normalized using the measured cation compositions of the glasses to obtain g/L leachate concentrations following the ASTM procedure.[13] The resulting values are given in Table IV. The KT08-series glasses all had normalized release for boron (NL [B]) values that were well below the 16.695 g/L value of the benchmark EA glass. The highest NL [B] was for glass KT08-03, with values of 0.65 g/L and 0.60 g/L for the quenched and CCC versions of this glass, respectively.

Table IV. Normalized PCT Results for the KT08-Series Glasses.

Glass ID	Heat Treatment	NL B (g/L)	NL Li (g/L)	NL Na (g/L)	NL Si (g/L)
ARM	ref	0.43	0.51	0.49	0.27
EA	ref	14.42	8.01	11.28	3.64
KT08-01	CCC	0.52	0.61	0.55	0.41
KT08-02	CCC	0.55	0.64	0.60	0.41
KT08-03	CCC	0.60	0.69	0.65	0.41
KT08-04	CCC	0.54	0.66	0.59	0.41
KT08-05	CCC	0.45	0.59	0.52	0.39
KT08-06	CCC	0.45	0.62	0.53	0.39
KT08-07	CCC	0.50	0.63	0.57	0.46
KT08-08	CCC	0.58	0.70	0.64	0.48
KT08-09	CCC	0.56	0.71	0.59	0.48
KT08-10	CCC	0.55	0.68	0.54	0.41
KT08-01	Quenched	0.55	0.68	0.61	0.44
KT08-02	Quenched	0.57	0.67	0.66	0.42
KT08-03	Quenched	0.65	0.73	0.74	0.42
KT08-04	Quenched	0.56	0.68	0.65	0.42
KT08-05	Quenched	0.47	0.61	0.55	0.39
KT08-06	Quenched	0.47	0.64	0.58	0.39
KT08-07	Quenched	0.53	0.67	0.61	0.48
KT08-08	Quenched	0.59	0.72	0.69	0.49
KT08-09	Quenched	0.57	0.71	0.61	0.48
KT08-10	Quenched	0.58	0.72	0.57	0.42

The predictability of the PCT responses was evaluated using the DWPF durability models. The predicted PCT values, determined using the measured compositions of the KT08 glasses, were compared with the normalized PCT responses. Figure 1 provides plots of the DWPF models for B, Li, Na, and Si that relate the logarithm of the normalized PCT value (for each of the four elements of interest) to a linear function of a free energy of hydration term (ΔG_p or del Gp, in kcal/100 g glass) derived from both of the heat treatments of the KT08-series glasses. Prediction limits at a 95% confidence for an individual PCT result are also plotted along with the linear fit. The EA and ARM results are indicated on these plots as well. As shown in the plots, the measured PCT responses for the KT08-series glasses are well predicted by the DWPF models.

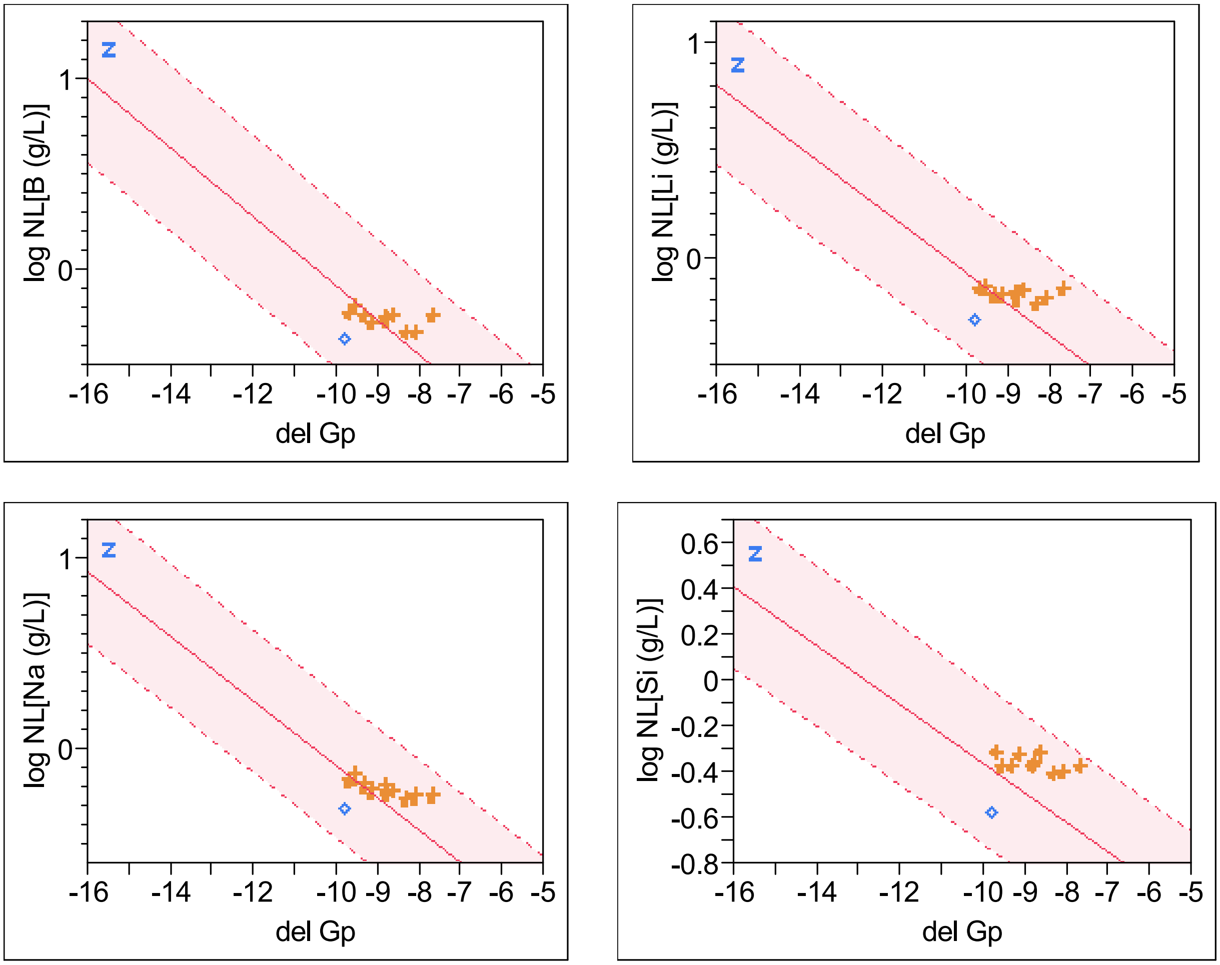

Figure 1. Plots of the DWPF durability models for B, Li, Na, and Si, showing the PCT responses for the study glasses (+), the ARM reference glass (◊), and the EA reference glass (**z**).

Viscosity

The measured viscosity at 1150 °C was determined by fitting the data for each glass to the Fulcher equation.[20,21] The results of the Fulcher fits were used to calculate a measured viscosity value for each glass at 1150 °C. These values are given in Table V. The measured values are displayed graphically versus the model predicted values in Figure 2. Figure 2 shows that all but one of the KT08-series glasses had measured viscosities that were predictable using the current DWPF viscosity model, based on the measured compositions. Composition KT08-03 had a measured viscosity that fell below the lower confidence interval for the model prediction. However, the difference between the lower confidence interval value and the measured value for this glass is only 2 poise (see Table V), which represents a difference with no practical impact. Overall, the measured viscosity values of the KT08-series glasses are well predicted by the current DWPF viscosity model.

Table V. Predicted and Measured Viscosity Values for the KT08-Series Glasses.

Glass ID	Viscosity Prediction (P)	Lower Confidence Interval for Prediction (P)	Upper Confidence Interval for Prediction (P)	Measured Viscosity (Fulcher Fit at 1150 °C) (P)	PCCS Predictable
KT08-01	41	28	61	52	Yes
KT08-02	46	31	67	52	Yes
KT08-03	55	38	81	36	No
KT08-04	54	37	78	56	Yes
KT08-05	76	52	111	80	Yes
KT08-06	88	60	129	67	Yes
KT08-07	50	34	74	70	Yes
KT08-08	52	36	76	58	Yes
KT08-09	42	28	61	51	Yes
KT08-10	63	43	93	68	Yes

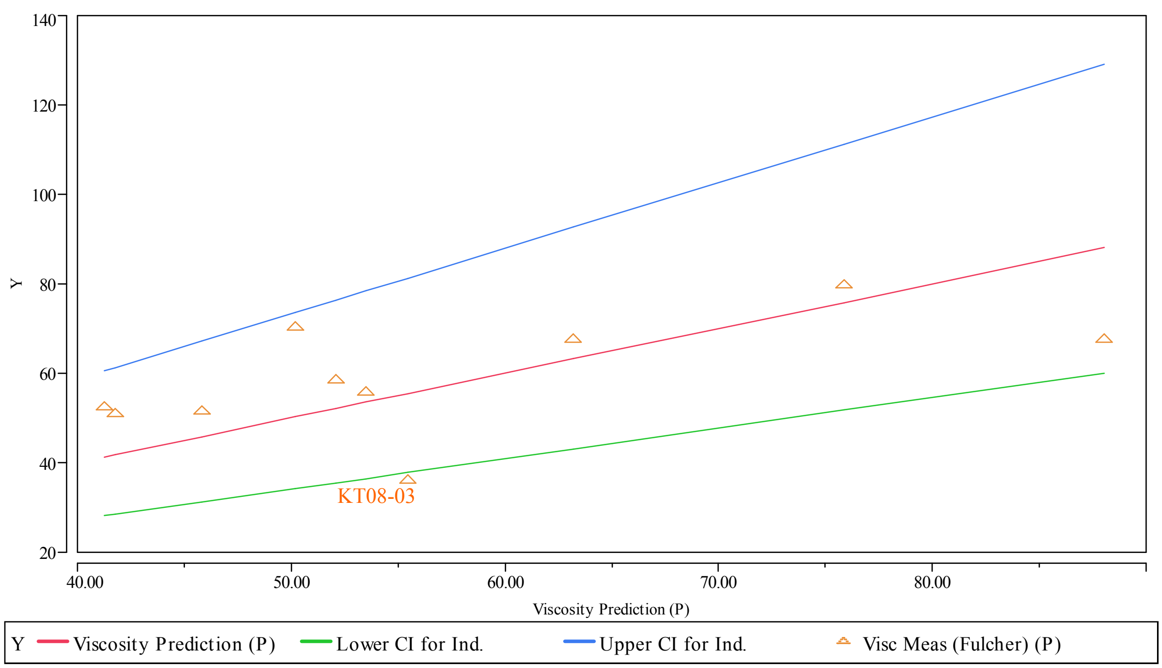

Figure 2. Predictability of the Viscosity Values at 1150 °C for the KT08-Series Glasses.

SUMMARY

A series of glass compositions was selected, fabricated, and characterized to determine the impacts of the addition of CST and MST from the SCIX process on the DWPF glass waste form and the applicability of the DWPF process control models. Specifically, the KT08-series of glasses was designed to evaluate any impacts of the inclusion of uranium and thorium in glasses containing the SCIX components. The glasses were fabricated in the laboratory and characterized using XRD to identify crystallization, ICP-AES to verify chemical compositions, and the PCT to measure durability. The viscosities of the glasses were also measured.

All but one of the KT08-series glasses were found to be amorphous by XRD. One of the slowly cooled glasses contained a small amount of trevorite, which is typically found in DWPF-type glasses

and had no practical impact on the durability of the glass. The measured PCT responses for the KT08-series glasses are well predicted by the current DWPF models. The viscosities of the KT08-series glasses were generally well predicted by the current DWPF model. No unexpected issues were encountered when uranium and thorium were added to the glasses with SCIX components. These results provide confidence that it will be possible to process high level waste with the addition of MST and CST from SCIX at the DWPF. Note however that liquidus temperature measurements remain in progress and may have an impact on the ability to process these feeds. Future work will determine the impact of these compositional changes on the applicability of the current DWPF liquidus temperature model.

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