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IMPACT OF COOLING RATE ON THE DURABILITY OF PHA GLASSES (U)

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SUMMARY AND CONCLUSION

This study was conducted to determine the effect, if any, on the PCT responses of glasses cooled at different rates. Two bounding cooling profiles were used in this study: rapidly quenched and a canister centerline cooling curve. Glasses were selected based on a number of criteria, but mainly to challenge the regions where amorphous phase separation is expected based upon current model predictions. The current DWPF homogeneity constraint, imposed to preclude regions of phase separation, predicted that most of the glasses selected would be phase separated. It was, therefore, important to ensure that deleterious phase separation does not occur at either cooling profile. In this case, deleterious phase separation is defined as the formation of an amorphous phase in the glass that significantly decreases the glass durability as measured by the PCT response.

X-ray diffraction and scanning electron microscopy were used to evaluate crystalline phases observed in the centerline cooled glasses. In one glass, pha17, a phase rich in copper was detected that had formed on the surface of Trevorite crystals. Trevorite crystals were also detected in several other clc glasses. A secondary phase precipitated out on the surface of Trevorite crystals in glasses that had low sludge loadings (~22 wt% oxide) and high PHA (~13% wt% oxides in the glass). This phase was rich in potassium and titanium.

The results showed that there was no practical difference between the PCT responses for glasses subjected to the two cooling profiles. In fact, although to a small extent, the centerline-cooled (clc) glasses were generally more durable than the rapidly quenched glasses in this study. These results reveal that no deleterious phase separation (amorphous or crystalline) occurred under either cooling regime.

All of the glasses readily satisfied the requirement that the PCT responses be at least two standard deviations below the PCT response of the EA glass. Furthermore, the PCT responses were almost entirely within the prediction intervals of the DWPF durability model. These results are detailed in the report.

INTRODUCTION

One of the Alternative Salt Disposition Flowsheets being considered would require that the Defense Waste Processing Facility (DWPF) vitrify a coupled feed containing High Level Waste (HLW) and Precipitate Hydrolysis Aqueous (PHA). A Technical Task Request (TTR) [1] was received by the Savannah River Technology Center (SRTC) requesting that a glass variability study be conducted to explore the impact of cooling rate on glass durability for this alternative under consideration as part of the Salt Processing Program. A Task Technical and Quality Assurance (TT&QA) plan [2] was issued by SRTC in response to the TTR. The objective of this task is to study the impact of cooling rate on the durability of glass with anticipated levels of PHA in DWPF glasses with nominal levels of monosodium titanate (MST).

The selection of targeted glass compositions for this task was provided in a separate memorandum.¹ The study glasses were selected from a set of candidate glasses that covered Purex, HM, and Blend sludge types at sludge loadings (in the glass) of 22 to 30 oxide weight percent (wt%), utilized PHA loadings (in the glass) of 7, 10, and 13 oxide wt%, and included MST concentrations (in the glass) mainly at 2.5 wt%. A $\pm 10\%$ variation in the composition of each sludge type and of the PHA was also utilized in the development of the set of candidate glass compositions. For each composition, the remainder of the glass consisted of Frit 202.

¹ The memorandum – Edwards, T. B., “Selecting Test Compositions for the FY01 Glass Studies of the Salt Processing Program,” SRT-SCS-2000-00069, December 15, 2000 – details the selection of targeted compositions for the PHA and CST studies. Some information contained in this memo is proprietary making the full details of the memorandum unavailable for discussion in this report.

Compositions were selected from the set of candidate points that increased the opportunity of seeing any significant cooling impact on glass durability as measured by the PCT. Several glasses were chosen that would also demonstrate reproducibility of the results from an earlier study [3]. The target compositions for the PHA glasses are provided in Table 1.

Table 1: Target Compositions (in wt%) for the PHA FY01 Study

Glass ID	Sludge																			
	Type	Sludge	MST	PHA	Frit	Al ₂ O ₃	B ₂ O ₃	CaO	CuO	Fe ₂ O ₃	K ₂ O	Li ₂ O	MgO	MnO	Na ₂ O					
pha01	Purex	26	2.50	13	58.50	2.856	9.247	0.992	1.033	11.613	6.180	4.037	1.334	2.037	8.503					
pha02	HM	26	1.25	10	62.75	7.040	8.520	0.470	0.790	7.450	4.750	4.330	1.380	2.310	8.000					
pha03	HM	22	2.50	13	62.50	5.450	9.100	0.360	0.910	6.030	6.710	4.310	1.350	2.150	8.220					
pha04	Blend	22	2.50	13	62.50	3.370	9.102	0.801	0.926	8.662	6.748	4.313	1.386	2.178	7.073					
pha05	Purex	22	2.50	13	62.50	2.707	10.025	0.807	1.119	8.844	5.553	4.313	1.388	1.896	8.567					
pha06	Purex	28	2.50	13	56.50	3.306	8.627	1.176	0.942	12.292	6.795	3.899	1.307	1.974	8.926					
pha07	Purex	24	2.50	13	60.50	2.907	9.867	1.008	0.932	10.536	5.942	4.175	1.361	1.692	8.532					
pha08	Purex	30	2.50	13	54.50	3.506	9.392	1.029	1.140	12.867	5.795	3.761	1.280	2.586	8.238					
pha09	Purex	28	2.50	13	56.50	2.766	8.627	1.176	0.942	12.832	6.795	3.899	1.307	1.974	8.926					
pha10	Purex	28	2.50	7	62.50	3.343	7.185	0.960	0.644	11.323	3.552	4.313	1.427	2.414	8.752					
pha11	Purex	30	2.50	7	60.50	3.542	7.027	1.260	0.545	12.372	3.770	4.175	1.400	2.115	8.799					
pha12	Purex	22	2.50	7	68.50	2.319	7.660	0.755	0.628	9.321	3.507	4.727	1.507	1.896	8.312					
pha13	Purex	22	2.50	13	62.50	2.283	9.102	0.755	1.119	9.321	6.372	4.313	1.388	1.896	8.671					
pha14	Purex	28	2.50	13	56.50	2.766	8.627	0.960	1.135	11.864	6.417	3.899	1.307	2.414	9.112					
pha15	Purex	22	2.50	7	68.50	2.319	8.157	0.924	0.628	10.756	3.164	4.727	1.507	1.551	7.619					
pha16	Purex	26	2.50	7	64.50	2.641	7.841	1.092	0.535	12.711	3.297	4.451	1.453	1.833	7.814					
pha17	Purex	30	2.50	13	54.50	2.927	9.392	1.029	1.140	13.446	5.795	3.761	1.280	2.586	8.238					
Glass ID	Sludge																			
	Type	Sludge	MST	PHA	Frit	NiO	P ₂ O ₅	SiO ₂	TiO ₂	U ₃ O ₈	ZnO	ZrO ₂	Cl	F	SO ₄					
pha01	Purex	26	2.50	13	58.50	1.097	0.046	45.523	2.197	2.363	0.104	0.169	0.308	0.053	0.310					
pha02	HM	26	1.25	10	62.75	0.400	0.030	51.940	1.100	0.800	0.080	0.130	0.230	0.040	0.230					
pha03	HM	22	2.50	13	62.50	0.370	0.030	51.470	2.200	0.740	0.070	0.110	0.190	0.030	0.190					
pha04	Blend	22	2.50	13	62.50	0.858	0.039	50.108	2.197	1.452	0.087	0.142	0.257	0.046	0.220					
pha05	Purex	22	2.50	13	62.50	1.021	0.039	48.526	2.197	2.200	0.088	0.143	0.261	0.046	0.260					
pha06	Purex	28	2.50	13	56.50	1.064	0.050	44.140	2.197	2.290	0.112	0.182	0.332	0.059	0.330					
pha07	Purex	24	2.50	13	60.50	0.912	0.043	47.064	2.197	1.963	0.096	0.156	0.284	0.050	0.280					
pha08	Purex	30	2.50	13	54.50	1.392	0.053	42.678	2.197	3.000	0.120	0.195	0.355	0.063	0.360					
pha09	Purex	28	2.50	13	56.50	1.064	0.050	44.140	2.197	2.290	0.112	0.182	0.332	0.059	0.330					
pha10	Purex	28	2.50	7	62.50	1.299	0.050	48.728	2.197	2.800	0.112	0.182	0.332	0.059	0.330					
pha11	Purex	30	2.50	7	60.50	1.392	0.053	47.266	2.197	3.000	0.120	0.195	0.355	0.063	0.360					
pha12	Purex	22	2.50	7	68.50	1.021	0.039	53.114	2.197	2.200	0.088	0.143	0.261	0.046	0.260					
pha13	Purex	22	2.50	13	62.50	1.021	0.039	48.526	2.197	2.200	0.088	0.143	0.261	0.046	0.260					
pha14	Purex	28	2.50	13	56.50	1.299	0.050	44.140	2.197	2.800	0.112	0.182	0.332	0.059	0.330					
pha15	Purex	22	2.50	7	68.50	0.836	0.039	52.979	2.197	1.800	0.088	0.143	0.261	0.046	0.260					
pha16	Purex	26	2.50	7	64.50	0.988	0.046	50.031	2.197	2.127	0.104	0.169	0.308	0.055	0.310					
pha17	Purex	30	2.50	13	54.50	1.392	0.053	42.678	2.197	3.000	0.120	0.195	0.369	0.063	0.360					

Property models [4] utilized by the DWPF for process and product quality control were used to help in the selection of the study glasses. The process properties of interest included viscosity and liquidus temperature. The product property of interest for the study glasses is durability (as measured by the 7-day Product Consistency Test (PCT) [5]). Predictions for the "Homogeneity" discriminator [6], used by the DWPF to avoid glass compositions that may undergo amorphous phase separation upon cooling, played an important role in the selection of the study glasses. The property predictions along with other key characteristics of the targeted compositions are provided in Table 2.

Table 2: Property Predictions for the PHA Study Glasses

Glass	Sludge	Loadings				Al ₂ O ₃	alkalis	Homo	del Gp	Normalized Releases				T _i
		S	MST	PHA	Frit					(wt%)	(wt%)	(wt%)	(g/L)	
pha01	Purex	26	2.50	13	58.50	2.856	18.72	205.2	-12.572	2.37	2.00	2.23	28.9	1009.7
pha02	HM	26	1.30	10	62.70	7.040	17.08	208.8	-9.652	0.70	0.75	0.71	97.6	933.7
pha03	HM	22	2.50	13	62.50	5.450	19.24	194.8	-11.948	1.83	1.62	1.75	71.5	904.9
pha04	Blend	22	2.50	13	62.50	3.370	18.13	196.5	-11.760	1.69	1.52	1.62	55.7	944.3
pha05	Purex	22	2.50	13	62.50	2.707	18.43	193.2	-12.480	2.28	1.94	2.15	39.6	949.4
pha06	Purex	28	2.50	13	56.50	3.306	19.62	210.8	-13.104	2.96	2.40	2.75	24.3	1032.7
pha07	Purex	24	2.50	13	60.50	2.907	18.65	202.8	-12.457	2.26	1.93	2.13	33.2	984.2
pha08	Purex	30	2.50	13	54.50	3.506	17.79	210.3	-11.880	1.77	1.59	1.70	25.4	1054.3
pha09	Purex	28	2.50	13	56.50	2.766	19.62	210.8	-13.255	3.15	2.53	2.92	22.4	1038.4
pha10	Purex	28	2.50	7	62.50	3.343	16.62	204.6	-10.320	0.93	0.94	0.92	52.0	993.0
pha11	Purex	30	2.50	7	60.50	3.542	16.74	210.9	-10.296	0.92	0.93	0.91	45.8	1018.9
pha12	Purex	22	2.50	7	68.50	2.319	16.55	194.0	-10.348	0.94	0.94	0.93	66.1	941.5
pha13	Purex	22	2.50	13	62.50	2.283	19.36	193.2	-13.196	3.07	2.48	2.85	36.0	955.3
pha14	Purex	28	2.50	13	56.50	2.766	19.43	203.8	-13.320	3.23	2.58	3.00	24.0	1020.7
pha15	Purex	22	2.50	7	68.50	2.319	15.51	202.0	-9.341	0.62	0.67	0.63	66.8	963.2
pha16	Purex	26	2.50	7	64.50	2.641	15.56	210.6	-9.378	0.63	0.68	0.63	53.8	1005.8
pha17	Purex	30	2.50	13	54.50	2.927	17.79	210.3	-12.041	1.90	1.67	1.81	23.1	1060.3

The purpose of this report is to provide and investigate comparisons between

- the measured and target compositions of the PHA glasses,
- the PCT measurements and durability model predictions, and
- the PCT measurements for quenched versus centerline-cooled glasses.

Evaluating the results from these comparisons provides the basis for assessing the impact of cooling rate on the durability of PHA glasses.

RESULTS AND DISCUSSION

The 17 glasses comprising the PHA study were designated as pha01 through pha17. Composition and PCT measurements of these glasses were conducted in accordance with analytical plans that were used to generate the measurements supporting the PHA study. These plans, which are identified in the discussion that follows, were prepared to support the overall Task Technical and QA plan [2] and the analytical study plan [7]. The results of these measurements (both composition and properties) are presented in this section.

Predictions for the properties of interest generated for these target compositions by the models utilized by the DWPF are also included in the discussion provided in Attachment I. These properties, for a given composition, relate to its processability and its product quality. For a given composition, acceptable property characteristics and reliable property predictions (using the current DWPF models) are of interest. The critical property for this study is glass durability, and comparisons between predictions and measurements for this property for both cooling treatments are provided in the discussion that follows.

Glass Batching and Cooling

The PHA glasses, corresponding to rows pha01 through pha17 of Table 1, were batched to the targeted compositions (combinations of sludge, glass formers, PHA, and MST) indicated in that table. Since the compositions of the simulated sludges were allowed to vary $\pm 10\%$ around their respective nominal compositions, their contributions to the PHA glasses had to be represented via dry chemicals (e.g., reagent grade nitrates, carbonates, and oxides). This was also true for the contribution of PHA in the glasses. The contributions of MST to the composition of the study glasses were also handled the same way. The contributions of the glass formers were represented by appropriate additions of Frit 202, Lot 14, available from the DWPF. The Frit 202 composition is given in Table 10 of the memorandum identified in footnote #1.

For each glass, the combined raw materials (~110 grams) were added to a 100 mL Pt-Au crucible and placed in a calibrated furnace, heated to 1150°C at a rate of 10°C/minute, and then held for four hours at 1150°C. The crucible was then removed and the glass immediately poured onto a clean stainless steel plate.

A portion of each glass cooled by quenching (as described in the preceding paragraph) was then remelted and allowed to cool following a centerline cooling profile. This was accomplished by taking a portion of each glass and placing it in a smaller crucible. Then, the crucible was introduced to the furnace at 1150°C, and the furnace was programmed to cool at a rate that simulated the centerline cooling profile of DWPF canisters.

Chemical Compositions

In addition to the PHA glasses, a standard glass (Batch 1) and a standard uranium-bearing glass were included in the planning of these analyses (for possible bias correction). An analytical plan (in the form of a memorandum [8]) was provided to assist the SRTC-Mobile Laboratory (SRTC-ML) in conducting these analyses (see Attachment I of this report).

Tables A.1 and A.2 in Appendix A provide the composition measurements obtained by the SRTC-ML for the plan covering these analyses. As indicated by these tables, two dissolution methods were used to perform these analyses: lithium metaborate and peroxide fusion dissolutions. The lithium metaborate method was used to generate measurements for Al, Ca, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, Si, Ti, and Zr. Peroxide fusion was used for B, Li, and U. The values are presented as weight percent (wt%) elemental concentrations. Table entries preceded by a "<" indicate measurements below the analytical detection limit (value shown). The elemental concentrations were converted to their corresponding oxide concentrations via multiplication by the appropriate gravimetric factor. Values below detection were replaced by 1/2 of the detection limit during the conversion process.

Exhibits A.1 and A.2 in Appendix A provide a plot of the measurements by oxide in analytical sequence for the two preparation methods. The analytical plan called for four measurements on each oxide of interest for each glass [8].

A review of the results from the standards was used to provide insight into the possibility that the ICP calibration contributes (in a systematic way) to the variation seen in the oxide measurements for the study glasses. Exhibits A.3 and A.4 in Appendix A provide plots of the oxide measurements per analytical block by oxide by preparation method. Statistical tests for significant differences among the analytical block averages are also included in these exhibits. Some statistically significant differences are indicated. Also, note that the pattern of behavior demonstrated by the measurements of one standard over a set of analytical blocks is frequently demonstrated by the measurements of the other standard.

Table 3 provides the average measured compositions by analytical block and the reference values for the two standards included in the analytical plan.

Table 3: Measurements from Glass Standards for Both Preparation Methods

Oxide	std (Batch 1)					Ustd (Uranium-bearing Standard)				
	Analytical Block				Reference Value	Analytical Block				Reference Value
	1-1	1-2	2-1	2-2		1-1	1-2	2-1	2-2	
Al ₂ O ₃	4.812	4.818	4.894	4.862	4.877	3.892	3.892	3.958	3.934	4.100
B ₂ O ₃	8.007	7.975	8.103	8.07	7.777	9.483	9.128	9.466	8.951	9.209
CaO	1.255	1.262	1.300	1.283	1.220	1.284	1.303	1.325	1.320	1.301
Cr ₂ O ₃	0.102	0.094	0.100	0.100	0.107	0.236	0.226	0.236	0.240	0.000
CuO	0.386	0.387	0.394	0.393	0.399	0.009	0.008	0.009	0.011	0.000
Fe ₂ O ₃	13.029	13.172	13.244	13.225	12.839	13.075	13.375	13.325	13.339	13.196
K ₂ O	3.670	3.650	3.734	3.662	3.327	2.861	2.897	2.933	2.891	2.999
Li ₂ O	4.406	4.449	4.600	4.485	4.429	3.079	3.003	3.143	2.928	3.057
MgO	1.394	1.389	1.401	1.454	1.419	1.171	1.155	1.180	1.222	1.210
MnO	1.648	1.636	1.666	1.709	1.726	2.628	2.582	2.653	2.712	2.892
Na ₂ O	8.847	8.811	8.982	8.852	9.003	10.211	10.225	10.353	10.252	11.795
NiO	0.649	0.649	0.660	0.671	0.751	0.936	0.924	0.947	0.966	1.120
SiO ₂	51.771	52.056	51.985	51.985	50.220	46.530	47.386	47.492	47.278	45.353
TiO ₂	0.643	0.634	0.656	0.657	0.677	0.930	0.918	0.949	0.957	1.049
U ₃ O ₈	0.059	0.059	0.059	0.059	0.000	2.364	2.323	2.423	2.258	2.406
ZrO ₂	0.074	0.066	0.077	0.071	0.098	0.008	0.008	0.008	0.008	0.000
Sum of Oxides	100.75	101.11	101.86	101.54	98.87	98.70	99.35	100.40	99.27	99.69

The measurements of the standards and the results from Exhibits A3 and A4 suggest bias correcting the measurements of the study glasses for potential analytical block effects. How was the bias correction conducted? For each dissolution method, let \bar{a}_{ij} be the average measurement for the i^{th} oxide at analytical block j for Batch 1 (or Ustd for uranium), and let t_i be the reference value for the i^{th} oxide. (The averages and reference values are provided in Table 3 for each oxide of interest.) Let \bar{c}_{ijk} be the average measurement for the i^{th} oxide at analytical block j for the k^{th} glass prepared by the given dissolution method. The bias adjustment was conducted as follows for each of the two dissolution methods

$$\bar{c}_{ijk} \cdot \left(1 - \frac{\bar{a}_{ij} - t_i}{\bar{a}_{ij}} \right) = \bar{c}_{ijk} \cdot \frac{t_i}{\bar{a}_{ij}}$$

Bias corrected measurements are indicated by additional “bc” suffix, and such adjustments were performed for all of the oxides and both of the dissolution methods of this study. This approach was used to bias correct the composition measurements of the PHA and standard glasses. Both measured and measured “bc” values are included in the discussion that follows.

Exhibit A.5 and A.6 provide plots of the measurements and bias-corrected (bc) measurements for each oxide by glass number (including the standards) by preparation method. The repeatability of the 4 measurements for each oxide for each glass is of interest in these plots.

Exhibit A.7 in Appendix A provides comparison plots for each oxide over all of the glasses (including the standards). These plots provide three views of the (average) chemical composition of the glasses: measured, measured bias-corrected, and targeted. Table 4 provides summary information for these compositional views. The sums of oxides for the targeted, measured, and measured bias-corrected compositions are also provided. A review of these sums shows that they all are within the interval of 95 to 105 weight percent with the smallest value being 95.3 wt% for the measured bias-corrected composition of pha13 and the largest being 101.3 wt% for the measured composition of Batch 1. One observation from this exhibit and table is that the K₂O measurements are consistently low for the PHA glasses (and bias-correction does not help). Also, the NiO and U₃O₈ measurements for pha07 and the U₃O₈ measurements for pha08 and pha17 indicate likely errors in the batching of these 3 glasses.²

² Additional samples of pha07, pha08, and pha17 were subsequently re-measured by the SRTC-ML yielding information supporting the conclusions regarding the batching of these glasses.

No other serious problems are seen in the comparisons of these compositional views of the PHA study glasses.

Table 4: Target, Measured and Bias-Corrected Compositions (in wt%) for the Study Glasses

Oxide	Batch 1			Uranium Standard (u-std)			pha01			pha02		
	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.
Al ₂ O ₃	4.877	4.847	4.877	4.100	3.921	3.945	2.856	2.924	2.962	7.040	6.916	6.914
B ₂ O ₃	7.777	8.039	7.777	9.209	9.257	8.956	9.247	8.831	8.491	8.520	8.404	8.179
CaO	1.220	1.275	1.220	1.301	1.308	1.252	0.992	1.055	1.023	0.470	0.561	0.530
Cr ₂ O ₃	0.107	0.099	0.107	0.000	0.234	0.253	0.000	0.007	0.008	0.000	0.007	0.008
CuO	0.399	0.390	0.399	0.000	0.009	0.009	1.033	1.035	1.069	0.790	0.757	0.767
Fe ₂ O ₃	12.839	13.168	12.839	13.196	13.278	12.947	11.613	11.541	11.311	7.450	7.270	7.053
K ₂ O	3.327	3.679	3.327	2.999	2.896	2.619	6.180	5.306	4.824	4.750	4.204	3.782
Li ₂ O	4.429	4.485	4.429	3.057	3.038	3.000	4.037	3.956	3.856	4.330	4.333	4.335
MgO	1.419	1.410	1.419	1.210	1.182	1.190	1.334	1.363	1.390	1.380	1.410	1.402
MnO	1.726	1.665	1.726	2.892	2.644	2.741	2.037	2.001	2.104	2.310	2.201	2.252
Na ₂ O	9.003	8.873	9.003	11.795	10.260	10.410	8.503	7.926	8.082	8.000	7.845	7.921
NiO	0.751	0.657	0.751	1.120	0.943	1.078	1.097	0.984	1.138	0.400	0.350	0.395
SiO ₂	50.220	51.949	50.220	45.353	47.172	45.601	45.523	47.011	45.478	51.940	53.429	51.615
TiO ₂	0.677	0.647	0.677	1.049	0.939	0.981	2.197	2.156	2.287	1.100	1.053	1.086
U ₃ O ₈	0.000	0.059	0.061	2.406	2.342	2.406	2.363	2.291	2.354	0.800	0.828	0.850
ZrO ₂	0.098	0.072	0.098	0.000	0.007	0.009	0.169	0.158	0.221	0.130	0.111	0.147
Sum of Oxides	98.869	101.314	98.930	99.687	99.430	97.398	99.181	98.546	96.596	99.410	99.680	97.235
Oxide	pha03			pha04			pha05			pha06		
	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.
Al ₂ O ₃	5.450	5.262	5.330	3.370	3.666	3.665	2.707	2.707	2.742	3.306	3.722	3.770
B ₂ O ₃	9.100	9.177	8.931	9.102	9.024	8.677	10.025	9.764	9.503	8.627	8.887	8.649
CaO	0.360	0.423	0.410	0.801	0.898	0.849	0.807	0.886	0.860	1.176	1.272	1.233
Cr ₂ O ₃	0.000	0.007	0.008	0.000	0.007	0.008	0.000	0.007	0.008	0.000	0.007	0.008
CuO	0.910	0.920	0.950	0.926	0.945	0.958	1.119	1.264	1.306	0.942	0.902	0.931
Fe ₂ O ₃	6.030	5.812	5.696	8.662	8.257	8.010	8.844	8.546	8.376	12.292	12.042	11.801
K ₂ O	6.710	5.776	5.251	6.748	5.776	5.197	5.553	4.897	4.451	6.795	5.860	5.327
Li ₂ O	4.310	4.370	4.372	4.313	4.257	4.150	4.313	4.317	4.318	3.899	4.064	4.066
MgO	1.350	1.396	1.423	1.386	1.413	1.405	1.388	1.394	1.422	1.307	1.310	1.336
MnO	2.150	2.134	2.243	2.178	2.160	2.210	1.896	1.853	1.948	1.974	1.905	2.002
Na ₂ O	8.220	7.781	7.934	7.073	6.801	6.866	8.567	7.957	8.113	8.926	8.300	8.463
NiO	0.370	0.345	0.399	0.858	0.780	0.880	1.021	0.889	1.029	1.064	0.945	1.094
SiO ₂	51.470	53.483	51.738	50.108	52.092	50.323	48.526	50.434	48.790	44.140	45.728	44.236
TiO ₂	2.200	2.118	2.247	2.197	2.139	2.206	2.197	2.056	2.181	2.197	2.114	2.242
U ₃ O ₈	0.740	0.759	0.779	1.452	1.386	1.424	2.200	2.223	2.282	2.290	2.385	2.448
ZrO ₂	0.110	0.098	0.136	0.142	0.123	0.162	0.143	0.125	0.174	0.182	0.161	0.225
Sum of Oxides	99.480	99.861	97.847	99.316	99.722	96.988	99.306	99.319	97.500	99.117	99.603	97.831
Oxide	pha07			pha08			pha09			pha10		
	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.
Al ₂ O ₃	2.907	3.085	3.124	3.506	3.566	3.566	2.766	2.985	3.024	3.343	3.335	3.334
B ₂ O ₃	9.867	10.223	9.949	9.392	9.499	9.244	8.627	8.235	7.918	7.185	7.156	6.964
CaO	1.008	1.124	1.090	1.029	1.165	1.100	1.176	1.221	1.184	0.960	1.064	1.005
Cr ₂ O ₃	0.000	0.007	0.008	0.000	0.007	0.008	0.000	0.007	0.008	0.000	0.010	0.010
CuO	0.932	0.946	0.977	1.140	1.267	1.285	0.942	0.903	0.932	0.644	0.665	0.674
Fe ₂ O ₃	10.536	10.805	10.589	12.867	12.857	12.473	12.832	12.649	12.397	11.323	11.141	10.808
K ₂ O	5.942	5.351	4.865	5.795	5.318	4.785	6.795	5.616	5.105	3.552	3.439	3.094
Li ₂ O	4.175	4.338	4.340	3.761	3.859	3.860	3.899	3.870	3.771	4.313	4.306	4.308
MgO	1.361	1.433	1.461	1.280	1.351	1.343	1.307	1.297	1.322	1.427	1.497	1.488
MnO	1.692	1.711	1.798	2.586	2.647	2.708	1.974	1.917	2.016	2.414	2.421	2.477
Na ₂ O	8.532	8.176	8.336	8.238	7.876	7.952	8.926	8.287	8.450	8.752	8.290	8.370
NiO	0.912	0.006	0.007	1.392	0.838	0.945	1.064	0.958	1.108	1.299	1.186	1.338
SiO ₂	47.064	49.953	48.324	42.678	45.407	43.865	44.140	45.728	44.236	48.728	49.953	48.257
TiO ₂	2.197	2.210	2.344	2.197	2.218	2.287	2.197	2.089	2.216	2.197	2.189	2.257
U ₃ O ₈	1.963	0.059	0.061	3.000	2.008	2.061	2.290	2.182	2.241	2.800	2.854	2.929
ZrO ₂	0.156	0.141	0.198	0.195	0.183	0.242	0.182	0.154	0.216	0.182	0.174	0.230
Sum of Oxides	99.244	99.569	97.471	99.056	100.066	97.724	99.117	98.098	96.143	99.119	99.679	97.544

**Table 4: Target, Measured and Bias-Corrected Compositions
(in wt%) for the Study Glasses (continued)**

Oxide	pha11			pha12			pha13			pha14		
	Target	Measured	Bias-cor.	Target	Measured	Bias-cor.	Target	Measured	Bias-cor.	Target	Measured	Bias-cor.
Al ₂ O ₃	3.542	3.434	3.433	2.319	2.352	2.383	2.283	2.485	2.484	2.766	2.721	2.756
B ₂ O ₃	7.027	6.858	6.595	7.660	7.615	7.411	9.102	8.927	8.585	8.627	8.573	8.343
CaO	1.260	1.309	1.237	0.755	0.844	0.819	0.755	0.856	0.809	0.960	1.054	1.022
Cr ₂ O ₃	0.000	0.007	0.008	0.000	0.007	0.008	0.000	0.007	0.008	0.000	0.007	0.008
CuO	0.545	0.551	0.559	0.628	0.634	0.655	1.119	1.122	1.137	1.135	1.139	1.177
Fe ₂ O ₃	12.372	11.863	11.509	9.321	9.175	8.992	9.321	9.200	8.925	11.864	11.902	11.665
K ₂ O	3.770	3.454	3.108	3.507	3.400	3.091	6.372	5.523	4.969	6.417	5.520	5.018
Li ₂ O	4.175	4.091	3.987	4.727	4.683	4.685	4.313	4.333	4.225	3.899	3.897	3.898
MgO	1.400	1.397	1.389	1.507	1.507	1.536	1.388	1.426	1.418	1.307	1.331	1.357
MnO	2.115	2.050	2.097	1.896	1.834	1.927	1.896	1.888	1.932	2.414	2.363	2.484
Na ₂ O	8.799	7.970	8.047	8.312	7.815	7.969	8.671	7.997	8.074	9.112	8.307	8.470
NiO	1.392	1.207	1.362	1.021	0.914	1.058	1.021	0.925	1.044	1.299	1.175	1.360
SiO ₂	47.266	50.274	48.567	53.114	54.606	52.825	48.526	48.830	47.172	44.140	45.621	44.133
TiO ₂	2.197	2.118	2.184	2.197	2.131	2.260	2.197	2.160	2.227	2.197	2.127	2.256
U ₃ O ₈	3.000	2.969	3.052	2.200	2.249	2.309	2.200	2.126	2.185	2.800	2.833	2.908
ZrO ₂	0.195	0.186	0.246	0.143	0.119	0.167	0.143	0.118	0.156	0.182	0.159	0.223
Sum of Oxides	99.055	99.739	97.379	99.307	99.886	98.094	99.307	97.923	95.348	99.119	98.730	97.077

Oxide	pha15			pha16			pha17		
	Target	Measured	Bias-cor.	Target	Measured	Bias-cor.	Target	Measured	Bias-cor.
Al ₂ O ₃	2.319	2.551	2.550	2.641	2.844	2.843	2.927	3.307	3.349
B ₂ O ₃	8.157	7.816	7.516	7.841	7.768	7.470	9.392	7.317	7.036
CaO	0.924	1.026	0.970	1.092	1.089	1.029	1.029	1.082	1.049
Cr ₂ O ₃	0.000	0.007	0.008	0.000	0.007	0.008	0.000	0.007	0.008
CuO	0.628	0.620	0.628	0.535	0.560	0.567	1.140	1.153	1.190
Fe ₂ O ₃	10.756	10.351	10.042	12.711	12.456	12.084	13.446	13.639	13.366
K ₂ O	3.164	3.075	2.766	3.297	3.126	2.812	5.795	3.192	2.902
Li ₂ O	4.727	4.596	4.480	4.451	4.500	4.386	3.761	4.021	3.919
MgO	1.507	1.548	1.539	1.453	1.531	1.522	1.280	1.379	1.405
MnO	1.551	1.569	1.605	1.833	1.885	1.928	2.586	2.673	2.809
Na ₂ O	7.619	7.202	7.271	7.814	7.171	7.241	8.238	7.037	7.175
NiO	0.836	0.760	0.858	0.988	0.911	1.028	1.392	1.292	1.495
SiO ₂	52.979	55.194	53.320	50.031	52.413	50.633	42.678	48.936	47.340
TiO ₂	2.197	2.127	2.193	2.197	2.164	2.231	2.197	2.239	2.375
U ₃ O ₈	1.800	1.745	1.793	2.127	1.781	1.831	3.000	1.854	1.906
ZrO ₂	0.143	0.120	0.159	0.169	0.174	0.230	0.195	0.187	0.261
Sum of Oxides	99.307	100.307	97.697	99.180	100.380	97.844	99.056	99.314	97.587

As stated earlier, two glasses for this study were repeats of glasses studied as part of the FY99 PHA work [3]. Table 5 provides the details of the chemical compositions of the glasses from the two studies. The two glasses for the current study are labeled FY01 pha01 and FY01 pha02. These glasses are repeats of pha12c and pha26, respectively, from the FY99 results.

The repeats are arranged together in Table 5, and they demonstrate good reproducibility between the two studies even though the FY01 sludge contribution to each glass was prepared individually from oxides in contrast to the FY99 approach where a large quantity of each sludge type was prepared for use in batching the glasses.

**Table 5: Target, Measured and Bias-Corrected Compositions
(in wt%) for the FY01 and FY99 Repeated Glasses**

Oxide	FY01 pha01			FY99 pha12c			FY01 pha02			FY99 pha26		
	Target	Measured	Bias-cor.	Target	Measured	Bias-cor.	Target	Measured	Bias-cor.	Target	Measured	Bias-cor.
Al ₂ O ₃	2.856	2.924	2.962	2.858	2.705	2.707	7.040	6.916	6.914	7.048	6.453	6.457
B ₂ O ₃	9.247	8.831	8.491	9.219	9.283	9.297	8.520	8.404	8.179	8.488	8.365	8.373
CaO	0.992	1.055	1.023	1.081	1.159	0.999	0.470	0.561	0.530	0.562	0.660	0.531
Cr ₂ O ₃	0.000	0.007	0.008	0.125	0.146	0.146	0.000	0.007	0.008	0.086	0.120	0.120
CuO	1.033	1.035	1.069	1.023	0.991	0.985	0.790	0.757	0.767	0.764	0.691	0.687
Fe ₂ O ₃	11.613	11.541	11.311	11.680	11.348	10.842	7.450	7.270	7.053	7.507	7.348	7.020
K ₂ O	6.180	5.306	4.824	6.125	5.397	5.661	4.750	4.204	3.782	4.736	4.445	4.662
Li ₂ O	4.037	3.956	3.856	4.013	4.280	4.126	4.330	4.333	4.335	4.305	4.391	4.233
MgO	1.334	1.363	1.390	1.237	1.248	1.224	1.380	1.410	1.402	1.409	1.286	1.262
MnO	2.037	2.001	2.104	2.041	1.984	1.969	2.310	2.201	2.252	2.311	2.320	2.303
Na ₂ O	8.503	7.926	8.082	8.487	8.510	8.289	8.000	7.845	7.921	7.980	7.952	7.745
NiO	1.097	0.984	1.138	1.099	0.980	0.940	0.400	0.350	0.395	0.397	0.377	0.362
SiO ₂	45.523	47.011	45.478	45.256	46.927	47.333	51.940	53.429	51.615	51.656	52.378	52.548
TiO ₂	2.197	2.156	2.287	2.222	2.222	2.143	1.100	1.053	1.086	1.125	1.173	1.131
U ₃ O ₈	2.363	2.291	2.354	2.367	2.328	2.328	0.800	0.828	0.850	0.800	0.954	0.954
ZrO ₂	0.169	0.158	0.221	0.129	0.155	0.125	0.130	0.111	0.147	0.141	0.171	0.138
Sum of Oxides	99.181	98.546	96.596	98.962	99.737	99.187	99.410	99.680	97.235	99.315	99.102	98.814

PCT Results

Samples of the 17 PHA glasses, after being batched and fabricated (via quenching), were subjected to a second heat treatment – they were cooled to simulate a centerline canister cooling profile. Differences in glass durability for these two cooling regimes (quenched versus centerline cooled) are of primary interest to this study. The investigation into this question required durability to be measured for the quenched and centerline cooled versions of each of the study glasses.

The 7-day Product Consistency Test (PCT) was used as the assessment of glass durability [5]. More specifically, Method A of the PCT (ASTM C1285) was used for these measurements. The PCTs were conducted in triplicate for the PHA glasses. In addition, PCTs were also conducted in triplicate for samples of the Environmental Assessment (EA) glass, the Approved Reference Material (ARM) glass, and a blank (ASTM Type I water). An analytical plan [9] supporting these tests was provided in the form of a memorandum (see Attachment II). This plan assisted the SRTC-ML in measuring the compositions of the solutions resulting from these PCTs. Of primary interest were the concentrations (in parts per million, ppm) of boron (B), lithium (Li), sodium (Na), and silicon (Si). Samples of a multi-element solution standard were also included in the analytical plan (as a check on the accuracy of the Inductively Coupled Plasma (ICP) – Emission Spectrometer used for these measurements).

The results from these tests are given in Table A.3 of Appendix A. The PCT results for the centerline-cooled version of each PHA study glass are indicated by the “clc” suffix on the glass ID. One of the quality control checkpoints for the PCT procedure is solution-weight loss over the course of the 7-day test. The shaded entries of Table A3 indicate those solutions that fell outside the weight-loss guidelines (weight loss must be less than 5%). Two successful solutions out of the 3 conducted for a glass are required to generate a representative PCT for that glass [5]. Although this criterion is not met for all of the PHA study glasses, the results are believed to provide meaningful and representative comparisons for assessing the impact of the cooling regimes since the impact must be larger than the measurement variability to be of consequence.

Any measurement in the “as reported” columns of Table A.3 proceeded by a “<” was below the detection limit for the ICP, and the measurement was replaced by ½ of the detection limit in the determination of the parts per million (ppm) columns of the table. The values in the ppm columns were also adjusted for the dilution factors by multiplying the “as-reported” values by 1.6667 for the PHA and ARM glasses and by 16.6667 for the EA glass. Thus, the concentrations in the ppm columns reflect detection and dilution adjustments.

Exhibit A.8 in the Appendix provides pairs of plots of the leachate concentrations and standards in the analytical sequence reported by the SRTC-ML. For each element of interest, one plot includes the values from the EA PCTs and the blanks. These values expand the scales of these plots, making it difficult to distinguish among the results of the other analyses. The other plot excludes the EA and blank results.

Exhibit A.9 in the Appendix provides pairs of plots of the leachate concentrations for each type of submitted solution: the standards, the blanks, EA, ARM, and the PHA glasses. One plot includes EA and the blank the other does not.

PCT leachate concentrations are typically normalized using the corresponding cation concentration (expressed as a weight percent) in the glass to obtain a grams-per-liter (g/L) leachate concentration. The normalization of the PCTs is usually conducted using the measured compositions of the glasses. This is the preferred normalization process for the PCTs. For completeness, the targeted cation and the bias-corrected cation concentrations will also be used to conduct this normalization.

As is the usual convention, the common logarithm of the normalized PCT (normalized leachate, NL) for each element of interest will be determined and used for comparison. To accomplish this computation, one must

1. Determine the common logarithm of the elemental parts per million (ppm) leachate concentration for each of the triplicates and each of the elements of interest (where these values are provided in Table A.3 of Appendix A),
2. Average the common logarithms over the triplicates for each element of interest, and then

Normalizing Using Measured Composition (preferred method)

3. Subtract a quantity equal to 1 plus the common logarithm of the average cation measured concentration (expressed as a weight percent of the glass) from the average computed in step 2.

Or Normalizing Using Target Composition

3. Subtract a quantity equal to 1 plus the common logarithm of the target cation concentration (expressed as a weight percent of the glass) from the average computed in step 2.

Or Normalizing Using Measured Bias-Corrected Composition

3. Subtract a quantity equal to 1 plus the common logarithm of the measured bias-corrected cation concentration (expressed as a weight percent of the glass) from the average computed in step 2.

As a preliminary step to completing these normalizations of the PCTs, statistical analyses were conducted of the results from the three analyses of the multi-element standard solution per analytical block. Exhibit A.10 in the Appendix provides these analyses. Although there appears to be statistical differences among the block averages, no bias correction of the PCT results for the study glasses was conducted. This approach was taken since the triplicate PCTs for a single study glass were placed in different ICP blocks. Averaging the ppm's for each set of triplicates helps to minimize the impact of the ICP effects.

The block averages are presented in Table 6, and they indicate consistent and reasonably accurate results (differences of overall averages versus reference values < 5%) from these analyses.

Table 6: Measurements of Standard Solution

Analytical	Avg	Avg	Avg	Avg
Block	B (ppm)	Li (ppm)	Na (ppm)	Si (ppm)
1	21.1	9.4	80.9	51.0
2	19.2	9.5	80.6	48.3
3	20.3	9.7	82.6	51.6
4	19.6	9.7	80.0	48.5
5	19.2	9.3	79.7	47.3
6	20.2	9.9	85.2	51.4
Grand Average	19.9	9.6	81.5	49.7
Reference Value	20	10	81	50
% difference	-0.3%	-4.3%	0.6%	-0.6%

Table 7 provides the results from the normalization process using the information in Table 4 and all of the data of Table A.3 (i.e., before screening the PCT results for solution-weight problems). Exhibit A.11 in Appendix A provides scatter plots for these results (both quenched and centerline cooled) offering an opportunity to investigate the consistency in the leaching across the elements for the glasses of this study. The consistency is typically demonstrated by a high degree of linear correlation among the values. PCT values normalized using targeted, measured, and bias-corrected compositions are investigated. A high degree of correlation is seen for these data for most pairs of elements. The smallest correlation (88%) is between B and Si for the PCTs normalized using the targeted compositions.

Table 7: Normalized PCTs before Screening for Solution-Weight Problems

Glass ID	Composition	Quenched								Centerline Cooled							
		log NL [B(g/L)]	log NL [Li(g/L)]	log NL [Na(g/L)]	log NL [Si(g/L)]	NL B(g/L)	NL Li(g/L)	NL Na(g/L)	NL Si(g/L)	log NL [B(g/L)]	log NL [Li(g/L)]	log NL [Na(g/L)]	log NL [Si(g/L)]	NL B(g/L)	NL Li(g/L)	NL Na(g/L)	NL Si(g/L)
ARM	see [10]	-0.2997	-0.2428	-0.3010	-0.5639	0.50	0.57	0.50	0.27								
EA	see [10]	1.1941	0.9326	1.0925	0.5577	15.64	8.56	12.37	3.61								
pha01	Meas.	0.1157	0.0745	0.0951	-0.2363	1.31	1.19	1.24	0.58	0.1225	0.0890	0.1079	-0.2253	1.33	1.23	1.28	0.60
	Meas. bc	0.1328	0.0856	0.0866	-0.2219	1.36	1.22	1.22	0.60	0.1395	0.1001	0.0995	-0.2109	1.38	1.26	1.26	0.62
	Target	0.0957	0.0657	0.0646	-0.2224	1.25	1.16	1.16	0.60	0.1025	0.0802	0.0774	-0.2113	1.27	1.20	1.20	0.61
pha02	Meas.	-0.3407	-0.2899	-0.3282	-0.5529	0.46	0.51	0.47	0.28	-0.3556	-0.3027	-0.3400	-0.5555	0.44	0.50	0.46	0.28
	Meas. bc	-0.3289	-0.2901	-0.3324	-0.5379	0.47	0.51	0.47	0.29	-0.3438	-0.3029	-0.3442	-0.5405	0.45	0.50	0.45	0.29
	Target	-0.3467	-0.2896	-0.3367	-0.5406	0.45	0.51	0.46	0.29	-0.3615	-0.3024	-0.3485	-0.5433	0.43	0.50	0.45	0.29
pha03	Meas.	-0.1017	-0.1062	-0.1023	-0.4604	0.79	0.78	0.79	0.35	-0.1806	-0.1811	-0.1919	-0.4761	0.66	0.66	0.64	0.33
	Meas. bc	-0.0899	-0.1064	-0.1108	-0.4460	0.81	0.78	0.77	0.36	-0.1688	-0.1813	-0.2003	-0.4617	0.68	0.66	0.63	0.35
	Target	-0.0981	-0.1001	-0.1262	-0.4438	0.80	0.79	0.75	0.36	-0.1770	-0.1751	-0.2157	-0.4595	0.67	0.67	0.61	0.35
pha04	Meas.	-0.0157	-0.0090	-0.0187	-0.3546	0.96	0.98	0.96	0.44	-0.0933	-0.0779	-0.0884	-0.3943	0.81	0.84	0.82	0.40
	Meas. bc	0.0013	0.0021	-0.0228	-0.3396	1.00	1.00	0.95	0.46	-0.0763	-0.0669	-0.0925	-0.3792	0.84	0.86	0.81	0.42
	Target	-0.0194	-0.0147	-0.0357	-0.3377	0.96	0.97	0.92	0.46	-0.0971	-0.0836	-0.1054	-0.3774	0.80	0.82	0.78	0.42
pha05	Meas.	0.2204	0.1835	0.2032	-0.1655	1.66	1.53	1.60	0.68	0.1615	0.1318	0.1551	-0.1914	1.45	1.35	1.43	0.64
	Meas. bc	0.2322	0.1834	0.1948	-0.1511	1.71	1.53	1.57	0.71	0.1733	0.1317	0.1467	-0.1770	1.49	1.35	1.40	0.67
	Target	0.2089	0.1839	0.1711	-0.1487	1.62	1.53	1.48	0.71	0.1501	0.1323	0.1230	-0.1746	1.41	1.36	1.33	0.67
pha06	Meas.	0.0297	0.0239	0.0748	-0.2735	1.07	1.06	1.19	0.53	0.0165	0.0006	0.0424	-0.2606	1.04	1.00	1.10	0.55
	Meas. bc	0.0415	0.0237	0.0664	-0.2591	1.10	1.06	1.17	0.55	0.0283	0.0004	0.0340	-0.2462	1.07	1.00	1.08	0.57
	Target	0.0426	0.0419	0.0432	-0.2581	1.10	1.10	1.10	0.55	0.0294	0.0186	0.0109	-0.2453	1.07	1.04	1.03	0.57
pha07	Meas.	0.1116	0.1004	0.1163	-0.2709	1.29	1.26	1.31	0.54	0.0640	0.0549	0.0772	-0.2743	1.16	1.13	1.19	0.53
	Meas. bc	0.1234	0.1002	0.1079	-0.2565	1.33	1.26	1.28	0.55	0.0758	0.0547	0.0688	-0.2599	1.19	1.13	1.17	0.55
	Target	0.1270	0.1170	0.0978	-0.2450	1.34	1.31	1.25	0.57	0.0794	0.0715	0.0587	-0.2484	1.20	1.18	1.14	0.56
pha08	Meas.	0.0506	0.0202	0.0320	-0.3083	1.12	1.05	1.08	0.49	0.0132	-0.0106	-0.0011	-0.3208	1.03	0.98	1.00	0.48
	Meas. bc	0.0624	0.0201	0.0278	-0.2933	1.15	1.05	1.07	0.51	0.0250	-0.0107	-0.0053	-0.3058	1.06	0.98	0.99	0.49
	Target	0.0555	0.0313	0.0124	-0.2814	1.14	1.07	1.03	0.52	0.0181	0.0006	-0.0206	-0.2939	1.04	1.00	0.95	0.51
pha09	Meas.	0.3415	0.2985	0.3460	-0.0727	2.20	1.99	2.22	0.85	0.3935	0.3434	0.3941	-0.0445	2.47	2.20	2.48	0.90
	Meas. bc	0.3585	0.3097	0.3376	-0.0583	2.28	2.04	2.18	0.87	0.4106	0.3546	0.3857	-0.0301	2.57	2.26	2.43	0.93
	Target	0.3213	0.2952	0.3138	-0.0574	2.10	1.97	2.06	0.88	0.3734	0.3401	0.3619	-0.0291	2.36	2.19	2.30	0.94
pha10	Meas.	-0.0486	-0.0622	-0.0530	-0.2864	0.89	0.87	0.89	0.52	-0.0557	-0.0708	-0.0674	-0.2859	0.88	0.85	0.86	0.52
	Meas. bc	-0.0368	-0.0624	-0.0571	-0.2714	0.92	0.87	0.88	0.54	-0.0439	-0.0710	-0.0716	-0.2709	0.90	0.85	0.85	0.54
	Target	-0.0504	-0.0629	-0.0765	-0.2756	0.89	0.87	0.84	0.53	-0.0575	-0.0715	-0.0910	-0.2752	0.88	0.85	0.81	0.53
pha11	Meas.	-0.0144	-0.0332	-0.0012	-0.2937	0.97	0.93	1.00	0.51	-0.0193	-0.0400	-0.0169	-0.2890	0.96	0.91	0.96	0.51
	Meas. bc	0.0026	-0.0220	-0.0053	-0.2787	1.01	0.95	0.99	0.53	-0.0023	-0.0288	-0.0210	-0.2740	0.99	0.94	0.95	0.53
	Target	-0.0249	-0.0421	-0.0441	-0.2669	0.94	0.91	0.90	0.54	-0.0299	-0.0488	-0.0598	-0.2622	0.93	0.89	0.87	0.55
pha12	Meas.	0.1006	0.0756	0.0825	-0.1749	1.26	1.19	1.21	0.67	0.0400	0.0199	0.0313	-0.2053	1.10	1.05	1.07	0.62
	Meas. bc	0.1124	0.0754	0.0740	-0.1605	1.30	1.19	1.19	0.69	0.0518	0.0197	0.0228	-0.1909	1.13	1.05	1.05	0.64
	Target	0.0980	0.0715	0.0557	-0.1629	1.25	1.18	1.14	0.69	0.0375	0.0158	0.0045	-0.1933	1.09	1.04	1.01	0.64
pha13	Meas.	0.3667	0.3129	0.3480	-0.0126	2.33	2.06	2.23	0.97	0.3498	0.3193	0.3523	-0.0282	2.24	2.09	2.25	0.94
	Meas. bc	0.3836	0.3239	0.3438	0.0024	2.42	2.11	2.21	1.01	0.3668	0.3303	0.3481	-0.0132	2.33	2.14	2.23	0.97
	Target	0.3582	0.3149	0.3128	-0.0099	2.28	2.06	2.06	0.98	0.3414	0.3214	0.3171	-0.0255	2.19	2.10	2.08	0.94
pha14	Meas.	0.3589	0.3217	0.3676	-0.0652	2.28	2.10	2.33	0.86	0.2404	0.2144	0.2619	-0.1208	1.74	1.64	1.83	0.76
	Meas. bc	0.3707	0.3216	0.3592	-0.0508	2.35	2.10	2.29	0.89	0.2522	0.2143	0.2534	-0.1064	1.79	1.64	1.79	0.78
	Target	0.3561	0.3215	0.3275	-0.0508	2.27	2.10	2.13	0.89	0.2377	0.2142	0.2217	-0.1064	1.73	1.64	1.67	0.78
pha15	Meas.	0.0039	-0.0027	-0.0252	-0.2821	1.01	0.99	0.94	0.52	-0.0287	-0.0382	-0.0564	-0.2873	0.94	0.92	0.88	0.52
	Meas. bc	0.0209	0.0084	-0.0293	-0.2671	1.05	1.02	0.93	0.54	-0.0117	-0.0271	-0.0605	-0.2723	0.97	0.94	0.87	0.53
	Target	-0.0146	-0.0149	-0.0496	-0.2643	0.97	0.97	0.89	0.54	-0.0473	-0.0504	-0.0808	-0.2695	0.90	0.89	0.83	0.54
pha16	Meas.	-0.0200	-0.0389	-0.0539	-0.2917	0.96	0.91	0.88	0.51	-0.0429	-0.0717	-0.0842	-0.2916	0.91	0.85	0.82	0.51
	Meas. bc	-0.0030	-0.0278	-0.0581	-0.2767	0.99	0.94	0.87	0.53	-0.0259	-0.0606	-0.0884	-0.2766	0.94	0.87	0.82	0.53
	Target	-0.0240	-0.0342	-0.0912	-0.2715	0.95	0.92	0.81	0.54	-0.0470	-0.0670	-0.1215	-0.2714	0.90	0.86	0.76	0.54
pha17	Meas.	0.0237	-0.0083	-0.0203	-0.3132	1.06	0.98	0.95	0.49	-0.2297	-0.2478	-0.2682	-0.4993	0.59	0.57	0.54	0.32
	Meas. bc	0.0407	0.0029	-0.0288	-0.2988	1.10	1.01	0.94	0.50	-0.2127	-0.2367	-0.2766	-0.4849	0.61	0.58	0.53	0.33
	Target	-0.0847	0.0207	-0.0888	-0.2538	0.82	1.05	0.82	0.56	-0.3381	-0.2188	-0.3366	-0.4399	0.46	0.60	0.46	0.38

Table 8 provides the results from the normalization process using the information in Table 5 and the screened data of Table A.3 (i.e., after screening the PCT results for solution-weight problems). A valid PCT [5] for a glass requires that at least 2 of the 3 triplicates pass the solution-weight criterion. The glasses whose PCT results do not satisfy that criterion are shaded in Table 8. All of the other entries of Table 8 are deemed valid PCTs. Exhibit A.12 in Appendix A provides correlations and scatter plots for the screened results (including both quenched and centerline cooled PCTs) offering an opportunity to investigate the consistency in the leaching across the elements for the glasses of this study. Correlations and scatter plots of just the valid PCTs are also provided as part of this exhibit. The consistency is typically demonstrated by a high degree of linear correlation among the values. PCT values normalized using targeted, measured, and bias-corrected compositions are investigated. A high degree of correlation is seen for these data for most pairs of elements. The smallest correlation (94%) is between Na and Si for the screened PCTs normalized using the targeted compositions.

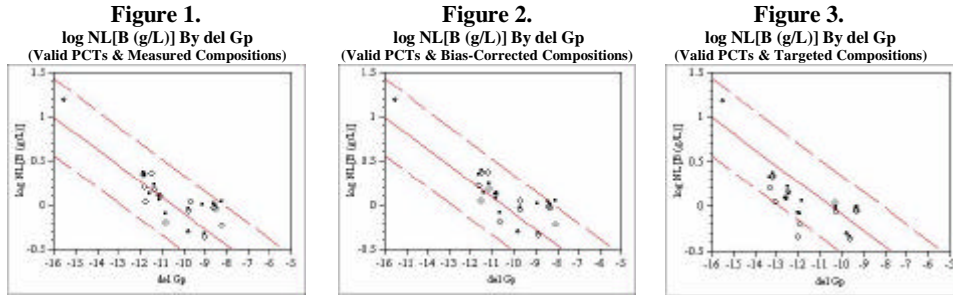
Table 8: Normalized PCTs after Screening for Solution-Weight Problems³

Glass ID	Composition	Quenched								Centerline Cooled							
		log NL [B(g/L)]	log NL [Li(g/L)]	log NL [Na(g/L)]	log NL [Si(g/L)]	NL B(g/L)	NL Li(g/L)	NL Na(g/L)	NL Si(g/L)	log NL [B(g/L)]	log NL [Li(g/L)]	log NL [Na(g/L)]	log NL [Si(g/L)]	NL B(g/L)	NL Si(g/L)	NL Na(g/L)	NL Li(g/L)
ARM	see [10]	-0.2997	-0.2428	-0.3010	-0.5639	0.50	0.57	0.50	0.27								
EA	see [10]	1.1970	0.9341	1.0951	0.5602	15.74	8.59	12.45	3.63								
pha01	Meas.	0.0953	0.0630	0.0840	-0.2463	1.25	1.16	1.21	0.57	0.1110	0.0879	0.1105	-0.2311	1.29	1.22	1.29	0.59
	Meas. bc	0.1123	0.0741	0.0756	-0.2319	1.30	1.19	1.19	0.59	0.1281	0.0990	0.1020	-0.2167	1.34	1.26	1.26	0.61
	Target	0.0753	0.0542	0.0535	-0.2323	1.19	1.13	1.13	0.59	0.0910	0.0790	0.0800	-0.2171	1.23	1.20	1.20	0.61
pha02	Meas.	-0.3407	-0.2899	-0.3282	-0.5529	0.46	0.51	0.47	0.28	-0.3556	-0.3027	-0.3400	-0.5555	0.44	0.50	0.46	0.28
	Meas. bc	-0.3289	-0.2901	-0.3324	-0.5379	0.47	0.51	0.47	0.29	-0.3438	-0.3029	-0.3442	-0.5405	0.45	0.50	0.45	0.29
	Target	-0.3467	-0.2896	-0.3367	-0.5406	0.45	0.51	0.46	0.29	-0.3615	-0.3024	-0.3485	-0.5433	0.43	0.50	0.45	0.29
pha03	Meas.	-0.1017	-0.1062	-0.1023	-0.4604	0.79	0.78	0.79	0.35	-0.1976	-0.1853	-0.1932	-0.4825	0.63	0.65	0.64	0.33
	Meas. bc	-0.0899	-0.1064	-0.1108	-0.4460	0.81	0.78	0.77	0.36	-0.1858	-0.1854	-0.2016	-0.4681	0.65	0.65	0.63	0.34
	Target	-0.0981	-0.1001	-0.1262	-0.4438	0.80	0.79	0.75	0.36	-0.1940	-0.1792	-0.2170	-0.4658	0.64	0.66	0.61	0.34
pha04	Meas.	-0.0410	-0.0368	-0.0497	-0.3723	0.91	0.92	0.89	0.42	-0.0890	-0.0699	-0.0831	-0.3836	0.81	0.85	0.83	0.41
	Meas. bc	-0.0240	-0.0257	-0.0538	-0.3573	0.95	0.94	0.88	0.44	-0.0720	-0.0589	-0.0872	-0.3686	0.85	0.87	0.82	0.43
	Target	-0.0448	-0.0425	-0.0667	-0.3554	0.90	0.91	0.86	0.44	-0.0927	-0.0756	-0.1001	-0.3667	0.81	0.84	0.79	0.43
pha05	Meas.	0.2149	0.1784	0.1914	-0.1710	1.64	1.51	1.55	0.67	0.1698	0.1332	0.1591	-0.1860	1.48	1.36	1.44	0.65
	Meas. bc	0.2266	0.1783	0.1830	-0.1566	1.69	1.51	1.52	0.70	0.1816	0.1331	0.1506	-0.1716	1.52	1.36	1.41	0.67
	Target	0.2034	0.1788	0.1594	-0.1542	1.60	1.51	1.44	0.70	0.1584	0.1336	0.1270	-0.1692	1.44	1.36	1.34	0.68
pha06	Meas.	0.0240	0.0178	0.0629	-0.2820	1.06	1.04	1.16	0.52	0.0336	0.0153	0.0598	-0.2411	1.08	1.04	1.15	0.57
	Meas. bc	0.0358	0.0176	0.0545	-0.2676	1.09	1.04	1.13	0.54	0.0454	0.0151	0.0514	-0.2266	1.11	1.04	1.13	0.59
	Target	0.0369	0.0358	0.0313	-0.2666	1.09	1.09	1.07	0.54	0.0465	0.0333	0.0283	-0.2257	1.11	1.08	1.07	0.59
pha07	Meas.	0.1116	0.1004	0.1163	-0.2709	1.29	1.26	1.31	0.54	0.0361	0.0282	0.0465	-0.3013	1.09	1.07	1.11	0.50
	Meas. bc	0.1234	0.1002	0.1079	-0.2565	1.33	1.26	1.28	0.55	0.0479	0.0280	0.0380	-0.2869	1.12	1.07	1.09	0.52
	Target	0.1270	0.1170	0.0978	-0.2450	1.34	1.31	1.25	0.57	0.0515	0.0448	0.0280	-0.2755	1.13	1.11	1.07	0.53
pha08	Meas.	0.0506	0.0202	0.0320	-0.3083	1.12	1.05	1.08	0.49								
	Meas. bc	0.0624	0.0201	0.0278	-0.2933	1.15	1.05	1.07	0.51								
	Target	0.0555	0.0313	0.0124	-0.2814	1.14	1.07	1.03	0.52								
pha09	Meas.									0.3586	0.3087	0.3579	-0.0663	2.28	2.04	2.28	0.86
	Meas. bc									0.3757	0.3199	0.3495	-0.0519	2.38	2.09	2.24	0.89
	Target									0.3384	0.3054	0.3257	-0.0510	2.18	2.02	2.12	0.89
pha10	Meas.	-0.0486	-0.0622	-0.0530	-0.2864	0.89	0.87	0.89	0.52	-0.0570	-0.0792	-0.0827	-0.2900	0.88	0.83	0.83	0.51
	Meas. bc	-0.0368	-0.0624	-0.0571	-0.2714	0.92	0.87	0.88	0.54	-0.0452	-0.0794	-0.0869	-0.2750	0.90	0.83	0.82	0.53
	Target	-0.0504	-0.0629	-0.0765	-0.2756	0.89	0.87	0.84	0.53	-0.0588	-0.0799	-0.1063	-0.2792	0.87	0.83	0.78	0.53
pha11	Meas.	-0.0079	-0.0320	-0.0056	-0.2922	0.98	0.93	0.99	0.51	-0.0420	-0.0441	-0.0184	-0.3004	0.91	0.90	0.96	0.50
	Meas. bc	0.0091	-0.0208	-0.0097	-0.2772	1.02	0.95	0.98	0.53	-0.0251	-0.0329	-0.0226	-0.2854	0.94	0.93	0.95	0.52
	Target	-0.0185	-0.0408	-0.0485	-0.2654	0.96	0.91	0.89	0.54	-0.0526	-0.0529	-0.0614	-0.2736	0.89	0.89	0.87	0.53
pha12	Meas.	0.1199	0.0747	0.0767	-0.1723	1.32	1.19	1.19	0.67	0.0400	0.0199	0.0313	-0.2053	1.10	1.05	1.07	0.62
	Meas. bc	0.1317	0.0745	0.0682	-0.1579	1.35	1.19	1.17	0.70	0.0518	0.0197	0.0228	-0.1909	1.13	1.05	1.05	0.64
	Target	0.1173	0.0706	0.0499	-0.1602	1.31	1.18	1.12	0.69	0.0375	0.0158	0.0045	-0.1933	1.09	1.04	1.01	0.64
pha13	Meas.	0.3667	0.3129	0.3480	-0.0126	2.33	2.06	2.23	0.97	0.3498	0.3193	0.3523	-0.0282	2.24	2.09	2.25	0.94
	Meas. bc	0.3836	0.3239	0.3438	0.0024	2.42	2.11	2.21	1.01	0.3668	0.3303	0.3481	-0.0132	2.33	2.14	2.23	0.97
	Target	0.3582	0.3149	0.3128	-0.0099	2.28	2.06	2.06	0.98	0.3414	0.3214	0.3171	-0.0255	2.19	2.10	2.08	0.94
pha14	Meas.	0.3223	0.2873	0.3332	-0.0909	2.10	1.94	2.15	0.81	0.2038	0.1815	0.2296	-0.1382	1.60	1.52	1.70	0.73
	Meas. bc	0.3341	0.2871	0.3248	-0.0765	2.16	1.94	2.11	0.84	0.2156	0.1814	0.2211	-0.1238	1.64	1.52	1.66	0.75
	Target	0.3195	0.2870	0.2931	-0.0766	2.09	1.94	1.96	0.84	0.2010	0.1813	0.1894	-0.1239	1.59	1.52	1.55	0.75
pha15	Meas.	0.0054	-0.0005	-0.0204	-0.2779	1.01	1.00	0.95	0.53	-0.0287	-0.0382	-0.0564	-0.2873	0.94	0.92	0.88	0.52
	Meas. bc	0.0224	0.0106	-0.0246	-0.2629	1.05	1.02	0.95	0.55	-0.0117	-0.0271	-0.0605	-0.2723	0.97	0.94	0.87	0.53
	Target	-0.0132	-0.0127	-0.0449	-0.2601	0.97	0.97	0.90	0.55	-0.0473	-0.0504	-0.0808	-0.2695	0.90	0.89	0.83	0.54
pha16	Meas.	-0.0200	-0.0389	-0.0539	-0.2917	0.96	0.91	0.88	0.51	-0.0429	-0.0717	-0.0842	-0.2916	0.91	0.85	0.82	0.51
	Meas. bc	-0.0030	-0.0278	-0.0581	-0.2767	0.99	0.94	0.87	0.53	-0.0259	-0.0606	-0.0884	-0.2766	0.94	0.87	0.82	0.53
	Target	-0.0240	-0.0342	-0.0912	-0.2715	0.95	0.92	0.81	0.54	-0.0470	-0.0670	-0.1215	-0.2714	0.90	0.86	0.76	0.54
pha17	Meas.	0.0237	-0.0123	-0.0275	-0.3141	1.06	0.97	0.94	0.49	-0.2297	-0.2478	-0.2682	-0.4993	0.59	0.57	0.54	0.32
	Meas. bc	0.0407	-0.0012	-0.0360	-0.2997	1.10	1.00	0.92	0.50	-0.2127	-0.2367	-0.2766	-0.4849	0.61	0.58	0.53	0.33
	Target	-0.0847	0.0167	-0.0960	-0.2547	0.82	1.04	0.80	0.56	-0.3381	-0.2188	-0.3366	-0.4399	0.46	0.60	0.46	0.36

As seen in Tables 7 and 8, the durabilities for the PHA glasses are much better than that of EA. (This is indicated for each glass by its normalized leachate being much smaller than that of EA.) Figures 1-3 provide an opportunity for a closer look at just the valid PCTs using measured, bias-corrected, and targeted compositions. Each of these figures is a plot of the DWPF model that relates the logarithm of the normalized PCT (in this case for B) to a linear function of a free energy of hydration term (ΔG_p , kcal/100g glass) derived from the glass (measured, bias-corrected, or targeted) compositions [10]. Prediction limits (at 95% confidence) for individual PCT results are also plotted around this linear fit. The PCT results for EA (shown as a diamond at $\Delta G_p \sim -15.5$), ARM (shown as a diamond at

³ Shaded rows in this table indicate glasses whose PCTs would not be considered valid due to less than 2 good results out of the 3 replicates conducted for that glass. Missing values indicate that none of the three PCTs for the indicated glass were considered valid.

ΔG_p slightly above -10), and the PHA glasses (quenched shown as small squares and centerline cooled shown as open circles) are presented on these plots. Note that the PHA results reveal acceptable PCTs and that the PCTs are well predicted by the current DWPF durability model for boron. (Most all of the PCT releases fall within the prediction interval for this model.) Exhibit A.13 in Appendix A repeats the plots of Figures 1-3, provides the same plots for Li, Na, and Si for the valid PCTs, and provides similar plots for B, Li, Na, and Si for the unscreened and screened PCTs. The behavior seen in these plots for B, Li, Na, and Si is similar to that demonstrated by the B results in Figures 1-3: acceptable and predictable (for almost all of the results) durabilities.



A look at the reproducibility of the PCT results between this and the FY99 study is provided in Table 9. This table provides the normalized PCT results (in grams/liter) from [3] for the FY99 glasses of interest and from Tables 7 and 8 for the quenched versions of those glasses repeated in this study. Recall that only quenched glasses were studied in the FY99 work. Good reproducibility between the two studies is demonstrated by the results presented in Table 9.

Table 9: Normalized PCTs for Glasses Common to the FY01 and FY99 Studies

Study & Glass ID	Composition Represented By	NL B(g/L)	NL Li(g/L)	NL Na(g/L)	NL Si(g/L)	Study & Glass ID	Composition	NL B(g/L)	NL Li(g/L)	NL Na(g/L)	NL Si(g/L)
FY01	Measured	1.25	1.16	1.21	0.57	FY01	Measured	0.46	0.51	0.47	0.28
pha01	Measured bias-cor.	1.30	1.19	1.19	0.59	pha02	Measured bias-cor.	0.47	0.51	0.47	0.29
	Targeted	1.19	1.13	1.13	0.59		Targeted	0.45	0.51	0.46	0.29
FY99	Measured	1.50	1.42	1.43	0.63	FY99	Measured	0.47	0.54	0.48	0.28
pha12	Measured bias-cor.	1.50	1.47	1.47	0.63	pha26	Measured bias-cor.	0.47	0.56	0.50	0.28
	Targeted	1.51	1.52	1.43	0.66		Targeted	0.46	0.55	0.48	0.29

Quenched versus Centerline-Cooled PCTs

A prime objective of this study was the investigation of potential cooling effects on the durability of the PHA glasses. Figures 1-3 and Exhibit A.13 provide a first look at differences in PCT response due to the cooling regime for the glasses. From those plots, there does not appear to be a lack of PCT predictability or acceptability due to cooling regime. A more rigorous comparison of the PCTs from quenched versus centerline cooled glasses is provided in this section.

Exhibit A.14 in Appendix A provides paired comparisons of the average common logarithm of the leachate concentrations in parts per million that were conducted using JMP® Version 4.0⁴. The data that are averaged are provided in Table A.3 of Appendix A. The exhibit investigates the unscreened, screened, and valid PCTs in turn. For boron (regardless of the PCT group: unscreened, screened, or

⁴ JMP® Version 4.0 is a commercial software product of SAS Institute, Inc. in Cary, NC. This product was used to support the statistical analyses presented in this report. See JMP® Statistical Discovery Software: Statistics and Graphics Guide (2000) published by SAS Institute for details.

valid), there is a statistically significant difference (at the 5% significance level) between the quenched and centerline cooled results with the boron PCT releases from the quenched glasses being slightly higher than those from the centerline cooled glasses. In log space, the difference (on average) is less than 0.06, which is of no real, practical concern.

The lithium and sodium results mirror those for boron, with the average difference being less than 0.06 in log space for each of these elements. The results for silicon releases for the two heat treatments are not statistically different at the 5% level for either PCT group (unscreened, screened, or valid), and the average log silicon release differences are all less than 0.03 in log space.

Another look at quenched versus centerline cooled PCTs is provided in Figures 4-7 for the valid PCTs. The plots provide the same type of comparisons as provided in Exhibit A.14, only framed in a different manner. These plots provide quenched versus centerline cooled scatter plots with the x and y axes drawn to the same scale. If the PCT response is not affected by cooling regime, the quenched and centerline cooled pair for a given glass should fall along the diagonal line for each of these plots. The 3 other lines (all three show if there is no overlap at the diagonal) on each plot represent the average difference due to cooling regime along with upper and lower confidence (at 95%) limits for this average.

Figure 4.

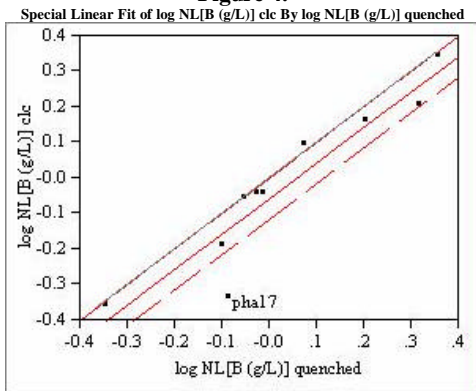


Figure 6.

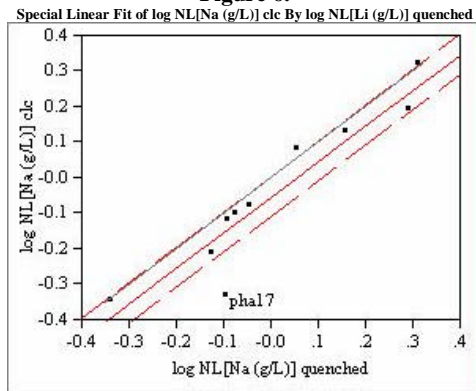


Figure 5.

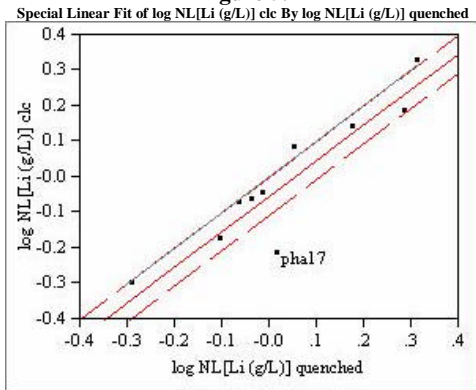
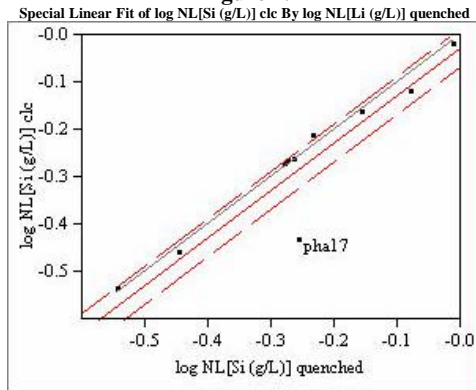


Figure 7.



The results for the pha17 glass have been labeled in these figures. This glass reveals the strongest impact of cooling on durability. It shows the clc version being more durable than the quenched version. More will be said regarding pha17 in the discussions that follow.

Overall, these results indicate no impact (of real importance) between the two cooling regimes (quenched and centerline cooled) for the PHA glasses of this study. What little effects that are seen appear to indicate that the quenched versions of the glasses are slightly less durable (on average) than the centerline cooled versions.

Phase Separation

The formation of separate amorphous phases in glass is referred to as amorphous phase separation or inhomogeneity. Crystal formation may indicate a “separation of phases,” but reflects crystalline particles within the glass matrix. Amorphous phase separation is to be avoided since the models currently used to predict durability do not apply for glasses predicted to be phase separated. The limit for the homogeneity constraint in the PCCS is nominally (for the Property Acceptance Region, PAR) a value of ~211 [4]. For the measurement acceptance region (MAR), the value will be even higher. In order for a glass to pass this constraint, the calculated value from chemical composition of the glass must be greater than the MAR value. The homogeneity values calculated using the targeted and measured, bias-corrected chemical compositions are all below the PAR value. Thus, all of these PHA glasses are predicted to be phase separated for these compositional views. The homogeneity values for the PHA glasses are given for all three compositional views in Table 10.

Table 10: Homogeneity Property Predictions

Homogeneity Property Prediction Based on - Acceptability Requires a Value > 211 - (Shaded Values are Acceptable)			
Glass ID	Target Composition	Measured Composition	Bias-Corrected Composition
pha01	205.2	204.8	199.8
pha02	208.8	208.7	203.5
pha03	194.8	194.2	190.0
pha04	196.5	197.4	191.3
pha05	193.2	192.6	188.2
pha06	210.8	213.0	208.3
pha07	202.8	209.9	205.2
pha08	210.3	214.7	208.6
pha09	210.8	210.2	205.2
pha10	204.6	205.1	199.4
pha11	210.9	210.3	204.1
pha12	194.0	195.1	190.7
pha13	193.2	192.1	186.1
pha14	203.8	203.9	199.2
pha15	202.0	203.5	197.4
pha16	210.6	212.8	206.4
pha17	210.3	214.9	210.0

The homogeneity constraint was developed for a glass compositional region that included PHA. Therefore, the predictability of phase separation by this model should be valid. A significant search for amorphous phase separation in these glasses was beyond the scope of work for this task, except when routine SEM analysis was performed.

XRD and SEM Analysis of the Glasses

None of the quenched glasses exhibited any visually apparent crystalline phases. However, several of the centerline-cooled glasses did have crystalline phases as observed visually. These glasses were

submitted to ADS for X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses. This section presents the results of these analyses.

The only glass that exhibited a significant crystalline presence in the glass matrix was pha17 (clc). The crystalline species identified by XRD in this glass was Trevorite (NiFe_2O_4). The clc pha17, a glass with 30 wt% sludge loading, also had another phase evident at the Trevorite crystal boundary. This phase consisted mainly of Cu. Therefore, it appears that a Cu phase precipitated out once the Trevorite crystals had formed for this glass. It is not clear if these results have any bearing on the pha17 durabilities shown in Figures 4-7, which indicate that the clc version of pha17 was more durable than the quenched version of this glass.

Crystals on the surface of several clc glasses were analyzed by SEM. Two of the glasses, pha04 and pha05, were at the low end of sludge loading (22 wt% oxide). The glass pha04 contained a blend type of sludge while pha05 contained a Purex type of sludge. These clc glasses had Spinel crystals that contained small amounts of Ti and Mn. In addition, both of these glasses contained phases that were high in K and Ti. These high K and Ti phases formed at the boundary of the smaller Spinel crystals. It appears that the Spinel crystals formed first and acted as a nucleating site for the appearance of the secondary phase. The secondary phase was much larger in size (20 to 50 microns in diameter) than the Spinel crystals (several microns in diameter). For the clc pha06, a glass with 28 wt% Purex loading, crystals of Trevorite were evident.

CONCLUSIONS

The results showed that there was no practical difference between the PCT responses for glasses subjected to the two cooling profiles. In fact, although small, the centerline cooling glasses were generally more durable than the rapidly quenched glasses. These results reveal that no deleterious phase separation (amorphous or crystalline) occurred under either cooling regime.

X-ray diffraction and scanning electron microscopy were used to evaluate crystalline phases observed in the centerline cooled glasses. In one glass, pha17, a phase rich in copper was detected that had formed at the interface of Trevorite crystals. Trevorite crystals were also detected in several other glasses. A secondary phase precipitated out at the boundary of Trevorite crystals in glasses that had low sludge loadings (~22 wt% oxides in the glass) and high PHA (~13 wt% oxides in the glass). This phase was rich in potassium and titanium.

All of the glasses readily satisfied the requirement that the PCT responses be at least two standard deviations below the PCT response of the EA glass. Furthermore, the PCT responses were almost entirely within the prediction intervals of the DWPF durability model.

The results showed that there was no practical difference between the PCT responses for glasses subjected to the two cooling profiles. In fact, although to a small extent, the centerline-cooled (clc) glasses were generally more durable than the rapidly quenched glasses in this study. These results reveal that no deleterious phase separation (amorphous or crystalline) occurred under either cooling regime.

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APPENDIX A.

Supplemental Tables and Exhibits

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Table A.1: SRTC-ML Measurements for Samples Prepared Using Lithium Metaborate Method
(Values are in elemental weight percents, wt% 's.)

Block	Sub-Block	Sequence (LM)	SRTC-ML ID (LM)	Glass ID (LM)	Al(v)	Ca(v)	Cr(v)	Cu(v)	Fe(v)	K(v)	Mg(v)	Mn(v)	Na(v)	Ni(v)	Si(v)	Ti(v)	Zr(v)
1	1	1	stdlm111	Batch 1	2.57	0.897	0.072	0.311	8.98	3.04	0.858	1.31	6.57	0.521	24.0	0.390	0.056
1	1	2	t07lm11	pha17	1.75	0.771	<0.010	0.920	9.34	2.65	0.840	2.09	5.27	1.02	22.7	1.35	0.141
1	1	3	t13lm21	pha07	1.63	0.798	<0.010	0.755	7.52	4.43	0.883	1.36	6.06	<0.010	23.4	1.34	0.110
1	1	4	t05lm21	pha03	2.79	0.303	<0.010	0.744	4.02	4.76	0.861	1.71	5.80	0.275	25.0	1.28	0.075
1	1	5	t07lm21	pha17	1.75	0.770	<0.010	0.919	9.55	2.64	0.844	2.09	5.18	1.03	22.9	1.35	0.142
1	1	6	t17lm11	pha06	1.96	0.897	<0.010	0.729	8.41	4.72	0.816	1.53	6.13	0.769	21.3	1.30	0.124
1	1	7	ustdlm111	Ustd	2.06	0.912	0.163	0.006	8.89	2.37	0.715	2.05	7.58	0.743	21.3	0.561	<0.010
1	1	8	t14lm11	pha01	1.54	0.741	<0.010	0.816	8.04	4.40	0.829	1.54	5.88	0.789	22.0	1.3	0.107
1	1	9	t06lm11	pha14	1.46	0.754	<0.010	0.917	8.31	4.54	0.816	1.85	6.16	0.938	21.4	1.29	0.124
1	1	10	t01lm21	pha05	1.43	0.625	<0.010	1.01	5.94	4.05	0.852	1.47	5.91	0.709	23.6	1.24	0.092
1	1	11	t14lm21	pha01	1.54	0.748	<0.010	0.828	8.11	4.37	0.825	1.57	5.84	0.762	22.1	1.29	0.131
1	1	12	stdlm112	Batch 1	2.52	0.886	0.070	0.306	9.28	3.03	0.848	1.28	6.50	0.512	24.4	0.386	0.055
1	1	13	t06lm21	pha14	1.43	0.749	<0.010	0.905	8.32	4.66	0.805	1.84	6.25	0.920	21.4	1.27	0.119
1	1	14	t04lm11	pha09	1.58	0.864	<0.010	0.714	8.84	4.63	0.788	1.49	6.15	0.758	21.3	1.26	0.118
1	1	15	t05lm11	pha03	2.77	0.301	<0.010	0.726	4.07	4.85	0.838	1.64	5.80	0.272	25.0	1.27	0.076
1	1	16	t16lm11	pha12	1.25	0.606	<0.010	0.502	6.45	2.85	0.909	1.43	5.84	0.717	25.5	1.28	0.091
1	1	17	ustdlm112	Ustd	2.06	0.923	0.160	0.008	9.4	2.38	0.698	2.02	7.57	0.728	22.2	0.554	<0.010
1	1	18	t01lm11	pha05	1.43	0.632	<0.010	1.00	6.02	4.07	0.839	1.43	5.92	0.696	23.8	1.23	0.097
1	1	19	t16lm21	pha12	1.23	0.597	<0.010	0.507	6.39	2.80	0.905	1.41	5.77	0.716	25.7	1.27	0.090
1	1	20	t13lm11	pha07	1.62	0.800	<0.010	0.754	7.54	4.45	0.857	1.30	6.05	<0.010	23.4	1.32	0.106
1	1	21	t17lm21	pha06	1.98	0.918	<0.010	0.711	8.39	5.05	0.770	1.44	6.25	0.722	21.5	1.25	0.120
1	1	22	t04lm21	pha09	1.58	0.875	<0.010	0.724	8.79	4.71	0.769	1.47	6.22	0.742	21.4	1.24	0.115
1	1	23	stdlm113	Batch 1	2.55	0.907	0.068	0.308	9.08	3.07	0.817	1.24	6.62	0.497	24.2	0.38	0.054
1	2	1	stdlm121	Batch 1	2.57	0.906	0.065	0.311	9.13	3.06	0.842	1.27	6.60	0.513	24.1	0.383	0.050
1	2	2	t14lm22	pha01	1.56	0.768	<0.010	0.842	8.08	4.44	0.818	1.55	5.91	0.757	22.0	1.29	0.127
1	2	3	t14lm12	pha01	1.55	0.759	<0.010	0.822	8.06	4.41	0.817	1.54	5.89	0.784	21.8	1.29	0.102
1	2	4	t16lm12	pha12	1.26	0.611	<0.010	0.508	6.37	2.86	0.917	1.43	5.83	0.725	25.3	1.29	0.087
1	2	5	t04lm22	pha09	1.59	0.885	<0.010	0.731	8.91	4.72	0.789	1.50	6.16	0.757	21.5	1.26	0.111
1	2	6	t01lm12	pha05	1.44	0.643	<0.010	1.02	5.95	4.07	0.843	1.43	5.92	0.700	23.4	1.24	0.095
1	2	7	ustdlm121	Ustd	2.06	0.930	0.155	0.005	9.34	2.41	0.701	2.01	7.60	0.729	22.1	0.554	<0.010
1	2	8	t05lm22	pha03	2.81	0.306	<0.010	0.747	4.08	4.76	0.840	1.64	5.74	0.267	25.1	1.27	0.068
1	2	9	t13lm12	pha07	1.65	0.809	<0.010	0.762	7.57	4.46	0.864	1.32	6.07	<0.010	23.2	1.33	0.102
1	2	10	t07lm12	pha17	1.75	0.777	<0.010	0.922	9.6	2.66	0.820	2.05	5.28	1.00	23.0	1.33	0.134
1	2	11	t07lm22	pha17	1.75	0.775	<0.010	0.922	9.67	2.65	0.822	2.05	5.15	1.01	22.9	1.34	0.136
1	2	12	stdlm122	Batch 1	2.56	0.907	0.064	0.310	9.3	3.04	0.838	1.26	6.53	0.509	24.6	0.38	0.051
1	2	13	t17lm12	pha06	1.96	0.900	<0.010	0.728	8.43	4.70	0.795	1.47	6.03	0.752	21.3	1.27	0.117
1	2	14	t16lm22	pha12	1.24	0.600	<0.010	0.509	6.46	2.78	0.905	1.41	5.75	0.716	25.6	1.27	0.085
1	2	15	t06lm22	pha14	1.43	0.759	<0.010	0.908	8.3	4.63	0.789	1.80	6.13	0.912	21.2	1.26	0.112
1	2	16	t01lm22	pha05	1.43	0.634	<0.010	1.01	6	4.07	0.830	1.41	5.86	0.690	23.5	1.22	0.085
1	2	17	ustdlm122	Ustd	2.06	0.933	0.154	0.007	9.37	2.40	0.692	1.99	7.57	0.724	22.2	0.547	<0.010
1	2	18	t06lm12	pha14	1.44	0.752	<0.010	0.911	8.37	4.50	0.801	1.83	6.11	0.923	21.3	1.28	0.117
1	2	19	t17lm22	pha06	1.98	0.920	<0.010	0.713	8.46	4.99	0.780	1.46	6.22	0.728	21.4	1.25	0.115
1	2	20	t04lm12	pha09	1.57	0.866	<0.010	0.715	8.85	4.59	0.783	1.48	6.06	0.753	21.3	1.25	0.113
1	2	21	t05lm12	pha03	2.77	0.299	<0.010	0.724	4.09	4.81	0.829	1.62	5.75	0.270	24.9	1.26	0.070
1	2	22	t13lm22	pha07	1.63	0.806	<0.010	0.753	7.6	4.43	0.853	1.32	6.08	<0.010	23.4	1.31	0.101
1	2	23	stdlm123	Batch 1	2.52	0.892	0.063	0.306	9.21	2.99	0.834	1.27	6.48	0.508	24.3	0.377	0.046
2	1	1	stdlm211	Batch 1	2.57	0.921	0.069	0.313	9.19	3.06	0.841	1.28	6.62	0.514	24.2	0.391	0.058
2	1	2	t08lm11	pha13	1.32	0.615	<0.010	0.899	6.43	4.59	0.847	1.45	6.03	0.724	23.3	1.29	0.090

Table A1: SRTC-ML Measurements for Samples Prepared Using Lithium Metaborate Method – (continued)
(Values are in elemental weight percents, wt% 's.)

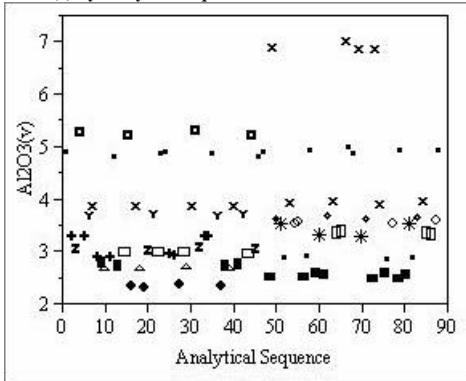
Block	Sub-Block	Sequence (LM)	SRTC-ML ID (LM)	Glass ID (LM)	Al(v)	Ca(v)	Cr(v)	Cu(v)	Fe(v)	K(v)	Mg(v)	Mn(v)	Na(v)	Ni(v)	Si(v)	Ti(v)	Zr(v)
2	1	3	t11m21	pha02	3.65	0.400	<0.010	0.598	5.05	3.52	0.835	1.64	5.84	0.273	24.8	0.624	0.084
2	1	4	t03m21	pha04	1.93	0.642	<0.010	0.752	5.76	4.81	0.829	1.66	5.06	0.603	24.2	1.27	0.094
2	1	5	t02m21	pha11	1.88	0.958	<0.010	0.451	8.5	2.95	0.852	1.62	6.09	0.966	22.8	1.30	0.144
2	1	6	t12m21	pha16	1.50	0.781	<0.010	0.447	8.6	2.62	0.904	1.44	5.35	0.705	24.5	1.29	0.130
2	1	7	ustdlm211	Ustd	2.09	0.946	0.161	0.008	9.33	2.43	0.707	2.05	7.67	0.739	22.1	0.566	<0.010
2	1	8	t10m21	pha08	1.88	0.836	<0.010	1.01	8.89	4.42	0.796	2.02	5.82	0.650	21.1	1.31	0.136
2	1	9	t10m11	pha08	1.89	0.836	<0.010	1.02	8.93	4.47	0.803	2.03	5.88	0.651	21.2	1.32	0.138
2	1	10	t08m21	pha13	1.32	0.618	<0.010	0.896	6.37	4.65	0.840	1.44	5.94	0.716	22.3	1.28	0.089
2	1	11	t12m11	pha16	1.52	0.783	<0.010	0.449	8.73	2.62	0.905	1.43	5.35	0.714	24.4	1.29	0.131
2	1	12	stdlm212	Batch 1	2.58	0.928	0.068	0.314	9.33	3.09	0.845	1.29	6.62	0.522	24.2	0.394	0.056
2	1	13	t15m11	pha15	1.36	0.735	<0.010	0.498	7.19	2.58	0.930	1.21	5.41	0.596	25.2	1.28	0.097
2	1	14	t02m11	pha11	1.77	0.920	<0.010	0.429	8.02	2.82	0.814	1.54	5.82	0.926	23.6	1.24	0.136
2	1	15	t15m21	pha15	1.34	0.734	<0.010	0.493	7.11	2.55	0.916	1.20	5.30	0.597	26.1	1.26	0.086
2	1	16	t03m11	pha04	1.96	0.652	<0.010	0.759	5.76	4.85	0.852	1.68	5.09	0.614	24.3	1.29	0.092
2	1	17	ustdlm212	Ustd	2.10	0.948	0.162	0.007	9.31	2.44	0.716	2.06	7.69	0.749	22.3	0.572	<0.010
2	1	18	t09m11	pha10	1.77	0.766	0.011	0.531	7.73	2.88	0.885	1.89	6.14	0.923	22.9	1.30	0.140
2	1	19	t09m21	pha10	1.78	0.765	<0.010	0.532	7.78	2.86	0.890	1.84	6.21	0.929	23.7	1.31	0.122
2	1	20	t11m11	pha02	3.72	0.409	<0.010	0.617	5.19	3.54	0.851	1.74	5.91	0.277	25.4	0.642	0.086
2	1	21	stdlm213	Batch 1	2.62	0.938	0.069	0.317	9.27	3.15	0.849	1.30	6.75	0.520	24.5	0.395	0.058
2	2	1	stdlm221	Batch 1	2.56	0.913	0.067	0.312	9.13	3.03	0.861	1.30	6.52	0.517	24.1	0.389	0.053
2	2	2	t11m12	pha02	3.64	0.396	<0.010	0.605	5.09	3.43	0.858	1.73	5.75	0.275	25.0	0.631	0.080
2	2	3	t02m12	pha11	1.74	0.904	<0.010	0.426	8.04	2.77	0.826	1.55	5.74	0.920	24.6	1.23	0.130
2	2	4	t03m12	pha04	1.93	0.639	<0.010	0.750	5.75	4.75	0.857	1.66	4.99	0.611	24.5	1.28	0.086
2	2	5	t08m12	pha13	1.31	0.605	<0.010	0.895	6.48	4.52	0.878	1.48	5.89	0.737	23.3	1.31	0.085
2	2	6	t11m22	pha02	3.63	0.398	<0.010	0.599	5.01	3.47	0.858	1.71	5.78	0.276	24.7	0.629	0.079
2	2	7	ustdlm221	Ustd	2.07	0.943	0.163	0.009	9.31	2.40	0.731	2.09	7.56	0.755	22.1	0.570	<0.010
2	2	8	t15m12	pha15	1.36	0.735	<0.010	0.496	7.51	2.55	0.941	1.22	5.38	0.596	25.2	1.29	0.091
2	2	9	t12m22	pha16	1.49	0.775	<0.010	0.446	8.59	2.58	0.939	1.48	5.32	0.719	24.5	1.30	0.126
2	2	10	t10m22	pha08	1.88	0.826	<0.010	1.00	9.01	4.36	0.819	2.03	5.77	0.657	21.2	1.33	0.133
2	2	11	t08m22	pha13	1.31	0.609	<0.010	0.896	6.46	4.58	0.875	1.48	5.87	0.731	22.4	1.30	0.085
2	2	12	stdlm222	Batch 1	2.58	0.917	0.068	0.314	9.24	3.04	0.878	1.32	6.58	0.527	24.3	0.394	0.054
2	2	13	t15m22	pha15	1.34	0.730	<0.010	0.493	7.15	2.53	0.947	1.23	5.28	0.600	26.7	1.27	0.082
2	2	14	t02m22	pha11	1.88	0.961	<0.010	0.455	8.63	2.93	0.879	1.64	6.00	0.982	23.0	1.31	0.141
2	2	15	t12m12	pha16	1.51	0.774	<0.010	0.447	8.93	2.56	0.945	1.49	5.26	0.727	24.6	1.31	0.128
2	2	16	t03m22	pha04	1.94	0.635	<0.010	0.758	5.83	4.77	0.871	1.69	5.04	0.623	24.4	1.29	0.091
2	2	17	ustdlm222	Ustd	2.10	0.944	0.165	0.009	9.35	2.40	0.743	2.11	7.65	0.764	22.1	0.578	<0.010
2	2	18	t09m12	pha10	1.76	0.756	<0.010	0.531	7.81	2.87	0.916	1.88	6.09	0.932	23.0	1.31	0.136
2	2	19	t09m22	pha10	1.75	0.755	<0.010	0.530	7.85	2.81	0.920	1.89	6.16	0.943	23.8	1.33	0.118
2	2	20	t10m12	pha08	1.90	0.832	<0.010	1.02	9.14	4.41	0.841	2.12	5.90	0.675	21.4	1.36	0.136
2	2	21	stdlm223	Batch 1	2.58	0.921	0.070	0.317	9.38	3.05	0.891	1.35	6.60	0.538	24.5	0.399	0.051

Table A2: SRTC-ML Measurements for Samples Prepared Using the Peroxide Fusion Method
(Values are in elemental weight percents, wt% 's.)

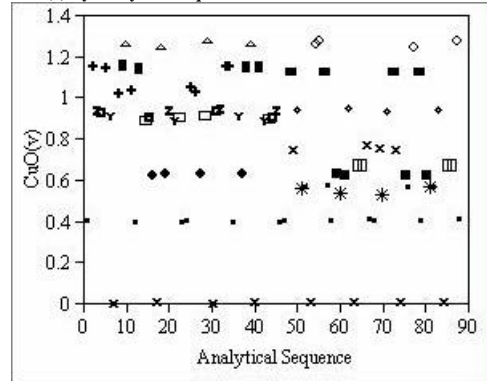
Block	Sub-Block	Sequence	SRTC-ML ID (pf)	Glass ID	B(v)	Li(v)	U(v)	Block	Sub-Block	Sequence	SRTC-ML ID (pf)	Glass ID	B(v)	Li(v)	U(v)
1	1	1	stdpf111	Batch 1	2.47	1.97	<0.100	1	2	22	t11pf12	pha02	2.49	1.96	0.705
1	1	2	t05pf11	pha03	2.97	2.10	0.659	1	2	23	stdpf123	Batch 1	2.52	2.13	<0.100
1	1	3	t11pf21	pha02	2.83	2.14	0.734	2	1	1	stdpf211	Batch 1	2.57	2.13	<0.100
1	1	4	t17pf21	pha06	2.98	2.04	2.19	2	1	2	t08pf21	pha13	2.79	2.00	1.83
1	1	5	t13pf11	pha07	3.30	2.07	<0.100	2	1	3	t15pf11	pha15	2.52	2.21	1.55
1	1	6	t16pf21	pha12	2.44	2.22	1.91	2	1	4	t03pf11	pha04	2.85	2.02	1.21
1	1	7	ustdpf111	Ustd	2.95	1.44	2.02	2	1	5	t07pf21	pha17	2.33	1.93	1.63
1	1	8	t17pf11	pha06	2.78	1.87	1.98	2	1	6	t02pf21	pha11	2.19	1.99	2.61
1	1	9	t01pf21	pha05	3.11	2.02	1.87	2	1	7	ustdpf211	Ustd	2.96	1.47	2.07
1	1	10	t05pf21	pha03	2.96	2.09	0.638	2	1	8	t04pf11	pha09	2.64	1.84	1.93
1	1	11	t09pf21	pha10	2.32	2.08	2.50	2	1	9	t14pf11	pha01	2.84	1.89	2.03
1	1	12	stdpf112	Batch 1	2.51	2.10	<0.100	2	1	10	t02pf11	pha11	2.16	1.94	2.57
1	1	13	t06pf21	pha14	2.75	1.86	2.46	2	1	11	t15pf21	pha15	2.50	2.24	1.57
1	1	14	t16pf11	pha12	2.47	2.27	1.99	2	1	12	stdpf212	Batch 1	2.49	2.15	<0.100
1	1	15	t13pf21	pha07	3.19	2.04	<0.100	2	1	13	t07pf11	pha17	2.32	1.91	1.61
1	1	16	t11pf11	pha02	2.61	2.01	0.683	2	1	14	t12pf21	pha16	2.45	2.13	1.51
1	1	17	ustd112	Ustd	2.94	1.42	1.99	2	1	15	t04pf21	pha09	2.73	1.96	1.97
1	1	18	t10pf11	pha08	2.98	1.80	1.70	2	1	16	t08pf11	pha13	2.78	2.04	1.90
1	1	19	t10pf21	pha08	3.03	1.83	1.74	2	1	17	ustdpf212	Ustd	2.92	1.45	2.04
1	1	20	t06pf11	pha14	2.70	1.84	2.44	2	1	18	t03pf21	pha04	2.87	2.04	1.21
1	1	21	t01pf11	pha05	3.08	2.03	1.92	2	1	19	t12pf11	pha16	2.48	2.18	1.59
1	1	22	t09pf11	pha10	2.26	2.02	2.44	2	1	20	t14pf21	pha01	2.84	1.92	2.03
1	1	23	stdpf113	Batch 1	2.48	2.07	<0.100	2	1	21	stdpf213	Batch 1	2.49	2.13	<0.100
1	2	1	stdpf121	Batch 1	2.42	1.98	<0.100	2	2	1	stdpf221	Batch 1	2.62	2.13	<0.100
1	2	2	t17pf12	pha06	2.61	1.80	1.95	2	2	2	t02pf22	pha11	2.07	1.80	2.41
1	2	3	t06pf12	pha14	2.62	1.78	2.37	2	2	3	t02pf12	pha11	2.10	1.87	2.48
1	2	4	t10pf22	pha08	2.92	1.76	1.68	2	2	4	t07pf22	pha17	2.20	1.80	1.52
1	2	5	t05pf12	pha03	2.73	1.94	0.639	2	2	5	t12pf12	pha16	2.31	2.00	1.45
1	2	6	t09pf12	pha10	2.14	1.95	2.36	2	2	6	t08pf12	pha13	2.83	2.08	1.71
1	2	7	ustdpf121	Ustd	2.82	1.39	1.97	2	2	7	ustdpf221	Ustd	2.79	1.36	1.92
1	2	8	t09pf22	pha10	2.17	1.95	2.38	2	2	8	t15pf22	pha15	2.26	1.97	1.37
1	2	9	t17pf22	pha06	2.67	1.84	1.97	2	2	9	t08pf22	pha13	2.69	1.93	1.77
1	2	10	t11pf22	pha02	2.51	1.94	0.687	2	2	10	t12pf22	pha16	2.41	2.05	1.49
1	2	11	t05pf22	pha03	2.74	1.99	0.638	2	2	11	t04pf22	pha09	2.35	1.65	1.69
1	2	12	stdpf122	Batch 1	2.49	2.09	<0.100	2	2	12	stdpf222	Batch 1	2.50	2.11	<0.100
1	2	13	t10pf12	pha08	2.87	1.78	1.69	2	2	13	t03pf12	pha04	2.69	1.89	1.11
1	2	14	t16pf12	pha12	2.27	2.09	1.86	2	2	14	t14pf22	pha01	2.62	1.76	1.85
1	2	15	t01pf22	pha05	2.98	1.99	1.87	2	2	15	t07pf12	pha17	2.24	1.83	1.53
1	2	16	t06pf22	pha14	2.58	1.76	2.34	2	2	16	t14pf12	pha01	2.67	1.78	1.86
1	2	17	ustdpf122	Ustd	2.85	1.40	1.97	2	2	17	ustdpf222	Ustd	2.77	1.36	1.91
1	2	18	t01pf12	pha05	2.96	1.98	1.88	2	2	18	t15pf12	pha15	2.43	2.12	1.43
1	2	19	t13pf22	pha07	3.06	1.94	<0.100	2	2	19	t04pf12	pha09	2.51	1.74	1.81
1	2	20	t16pf22	pha12	2.28	2.12	1.87	2	2	20	t03pf22	pha04	2.80	1.96	1.17
1	2	21	t13pf12	pha07	3.15	2.01	<0.100	2	2	21	stdpf223	Batch 1	2.40	2.01	<0.100

**Exhibit A.1: Measurements of Samples Prepared
Using Lithium Metaborate (LM) Method in Analytical Sequence by Oxide**
(x – Ustd; small square – Batch 1; PHA Study glasses – Other symbols)

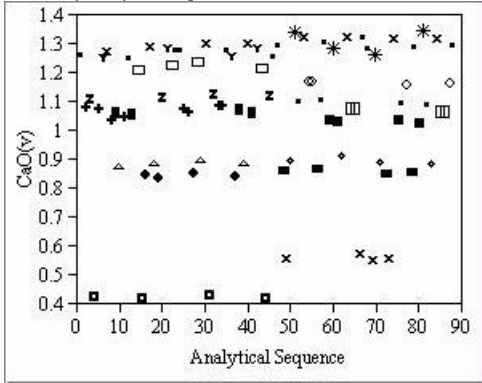
Al₂O₃(v) By Analytical Sequence



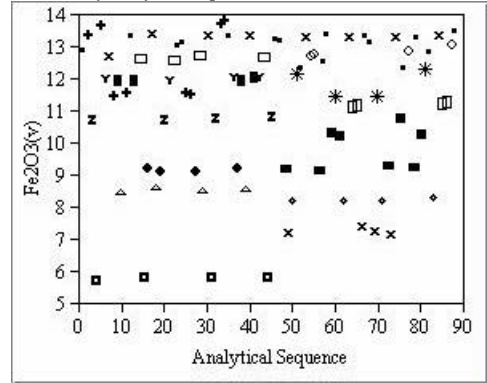
CuO(v) By Analytical Sequence



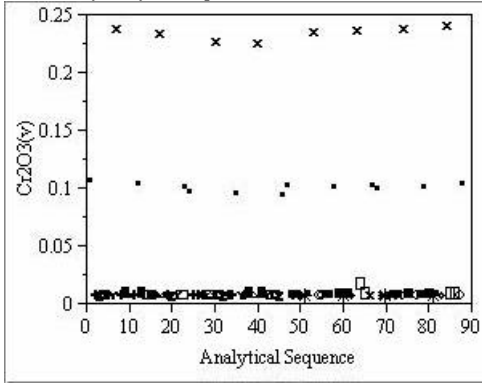
CaO(v) By Analytical Sequence



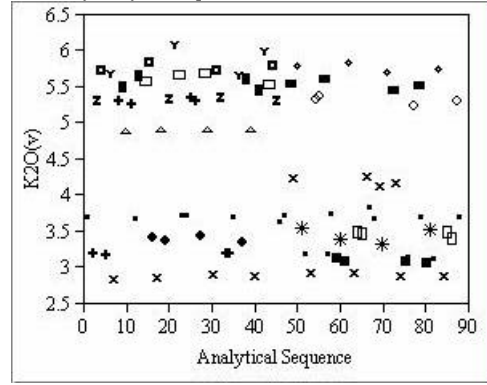
Fe₂O₃(v) By Analytical Sequence



Cr₂O₃(v) By Analytical Sequence

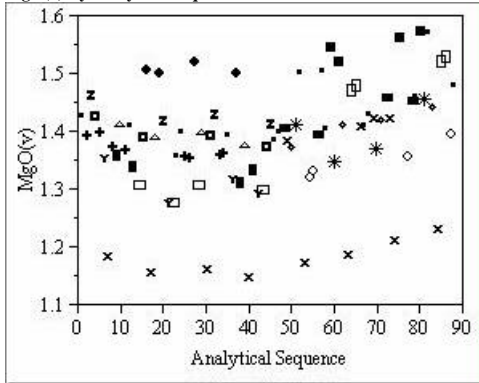


K₂O(v) By Analytical Sequence

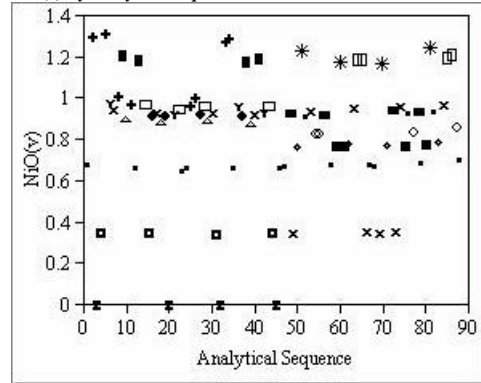


**Exhibit A.1: Measurements of Samples Prepared
Using Lithium Metaborate (LM) Method in Analytical Sequence by Oxide (continued)**
(x – Ustd; small square – Batch 1; PHA Study glasses – Other symbols)

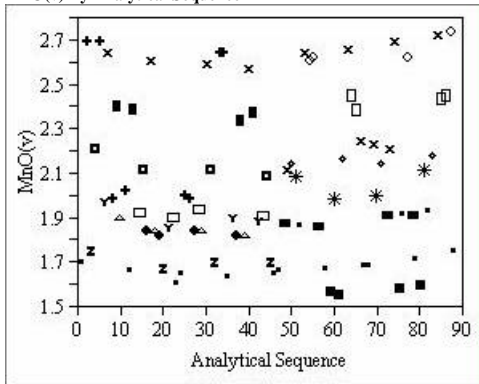
MgO(v) By Analytical Sequence



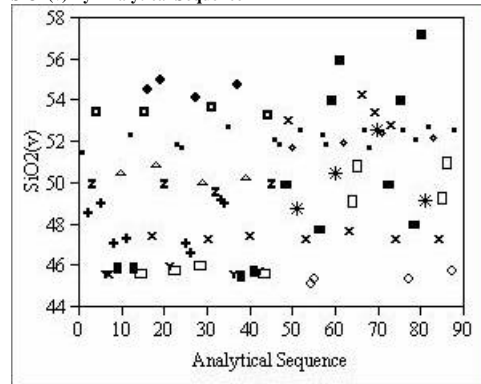
NiO(v) By Analytical Sequence



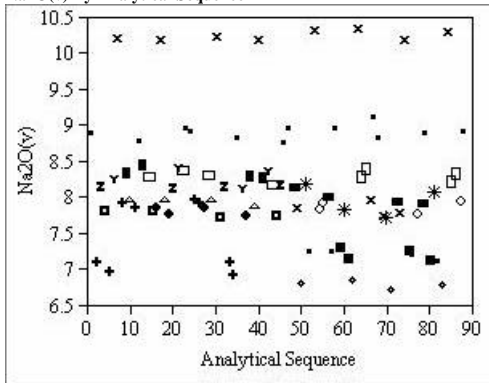
MnO(v) By Analytical Sequence



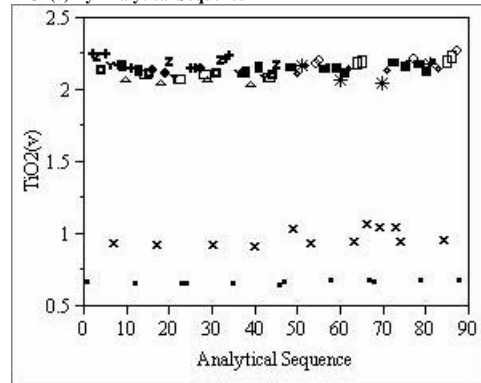
SiO2(v) By Analytical Sequence



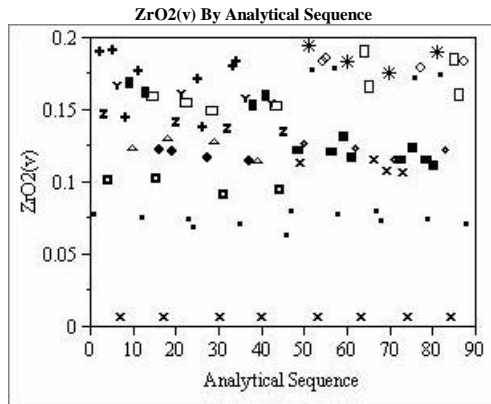
Na2O(v) By Analytical Sequence



TiO2(v) By Analytical Sequence



**Exhibit A.1: Measurements of Samples Prepared
Using Lithium Metaborate (LM) Method in Analytical Sequence by Oxide (continued)**
(x - Ustd; small square - Batch 1; PHA Study glasses - Other symbols)



**Exhibit A.2: Measurements of Samples Prepared
Using Peroxide Fusion (pf) Method in Analytical Sequence by Oxide (continued)**
(x – Ustd; small square – Batch 1; PHA Study glasses – Other symbols)

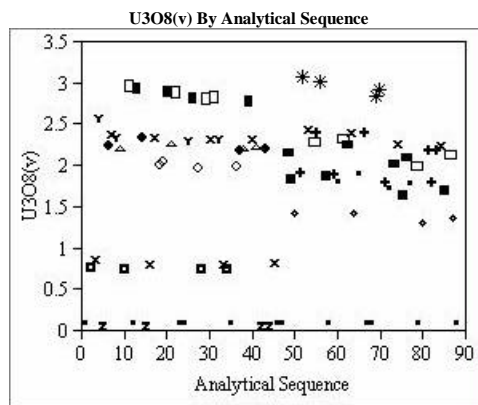
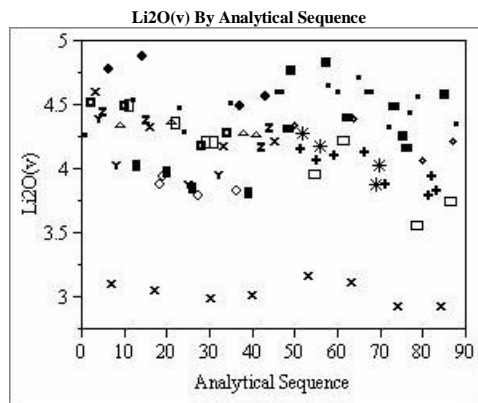
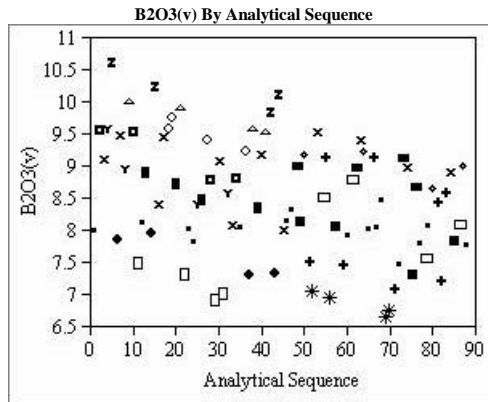
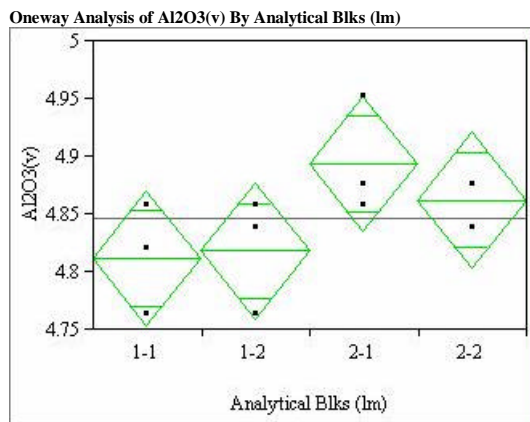


Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block
(small square – Batch 1 standard; x – Ustd;)



Oneway Anova
Summary of Fit

Rsquare	0.465021
Adj Rsquare	0.264403
Root Mean Square Error	0.043976
Mean of Response	4.846567
Observations (or Sum Wgts)	12

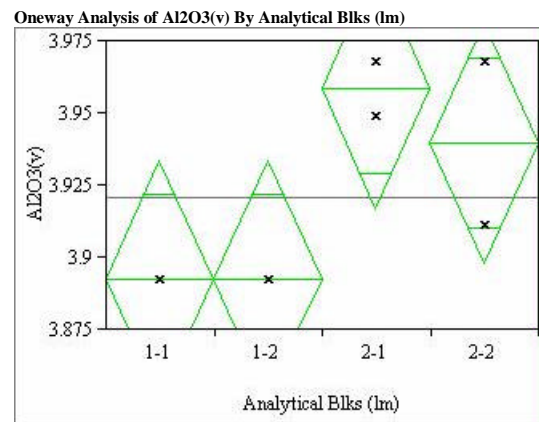
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.01344779	0.004483	2.3179	0.1520
Error	8	0.01547091	0.001934		
C. Total	11	0.02891870			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	4.81193	0.02539	4.7534	4.8705
1-2	3	4.81822	0.02539	4.7597	4.8768
2-1	3	4.89380	0.02539	4.8353	4.9524
2-2	3	4.86231	0.02539	4.8038	4.9209

Std Error uses a pooled estimate of error variance



Oneway Anova
Summary of Fit

Rsquare	0.791667
Adj Rsquare	0.635417
Root Mean Square Error	0.021125
Mean of Response	3.920713
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00678340	0.002261	5.0667	0.0755
Error	4	0.00178511	0.000446		
C. Total	7	0.00856850			

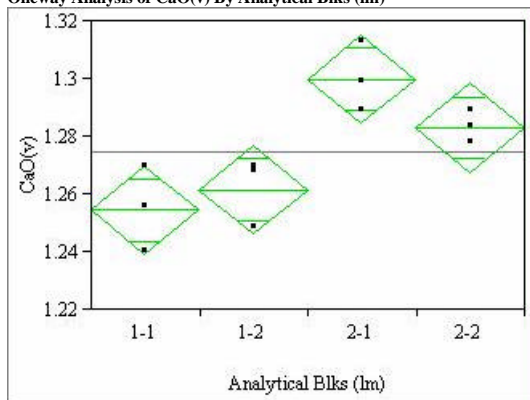
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	3.89237	0.01494	3.8509	3.9338
1-2	2	3.89237	0.01494	3.8509	3.9338
2-1	2	3.95850	0.01494	3.9170	4.0000
2-2	2	3.93961	0.01494	3.8981	3.9811

Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block
(continued)
(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of CaO(v) By Analytical Blks (lm)



Oneway Anova

Summary of Fit

Rsquare	0.784
Adj Rsquare	0.703
Root Mean Square Error	0.011489
Mean of Response	1.274788
Observations (or Sum Wgts)	12

Analysis of Variance

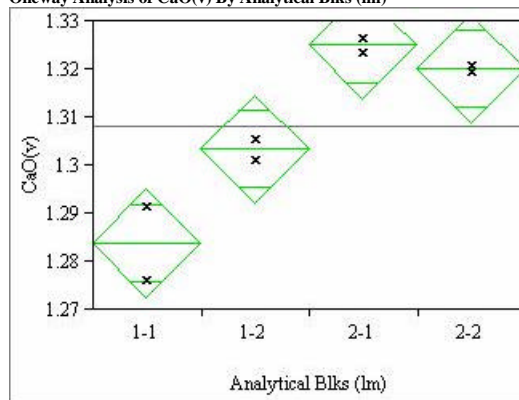
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00383248	0.001277	9.6790	0.0049
Error	8	0.00105589	0.000132		
C. Total	11	0.00488837			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	1.25462	0.00663	1.2393	1.2699
1-2	3	1.26161	0.00663	1.2463	1.2769
2-1	3	1.29986	0.00663	1.2846	1.3152
2-2	3	1.28307	0.00663	1.2678	1.2984

Std Error uses a pooled estimate of error variance

Oneway Analysis of CaO(v) By Analytical Blks (lm)



Oneway Anova

Summary of Fit

Rsquare	0.940627
Adj Rsquare	0.896097
Root Mean Square Error	0.005748
Mean of Response	1.308077
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00209358	0.000698	21.1235	0.0065
Error	4	0.00013215	0.000033		
C. Total	7	0.00222573			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	1.28377	0.00406	1.2725	1.2951
1-2	2	1.30335	0.00406	1.2921	1.3146
2-1	2	1.32504	0.00406	1.3138	1.3363
2-2	2	1.32015	0.00406	1.3089	1.3314

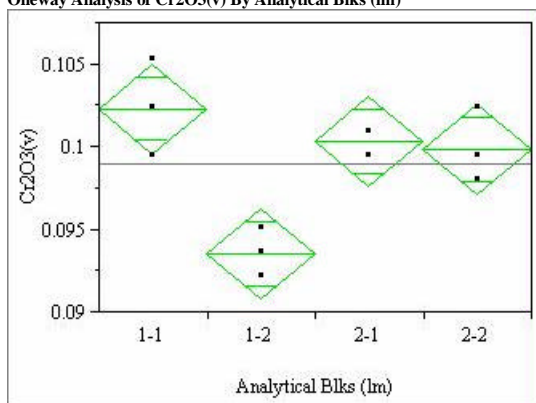
Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block

(continued)

(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of Cr2O3(v) By Analytical Blks (lm)



Oneway Anova
Summary of Fit

Rsquare	0.798907
Adj Rsquare	0.723497
Root Mean Square Error	0.002023
Mean of Response	0.099023
Observations (or Sum Wgts)	12

Analysis of Variance

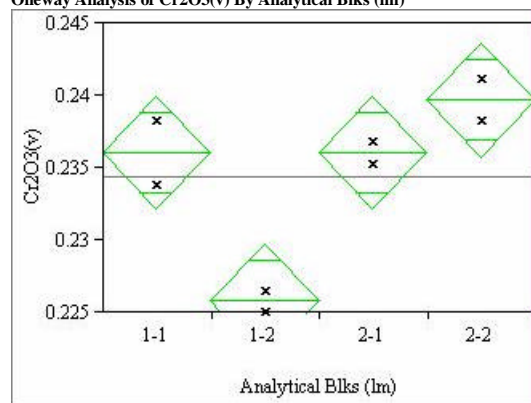
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00013013	0.000043	10.5942	0.0037
Error	8	0.00003276	0.000004		
C. Total	11	0.00016289			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	0.102312	0.00117	0.09962	0.10501
1-2	3	0.093542	0.00117	0.09085	0.09624
2-1	3	0.100363	0.00117	0.09767	0.10306
2-2	3	0.099876	0.00117	0.09718	0.10257

Std Error uses a pooled estimate of error variance

Oneway Analysis of Cr2O3(v) By Analytical Blks (lm)



Oneway Anova
Summary of Fit

Rsquare	0.930475
Adj Rsquare	0.878331
Root Mean Square Error	0.002001
Mean of Response	0.234404
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00021443	0.000071	17.8444	0.0089
Error	4	0.00001602	0.000004		
C. Total	7	0.00023045			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	0.236048	0.00142	0.23212	0.23998
1-2	2	0.225817	0.00142	0.22189	0.22975
2-1	2	0.236048	0.00142	0.23212	0.23998
2-2	2	0.239702	0.00142	0.23577	0.24363

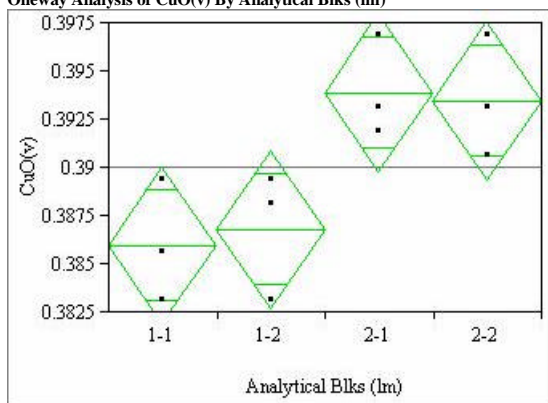
Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block

(continued)

(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of CuO(v) By Analytical Blks (lm)



Oneway Anova
Summary of Fit

Rsquare	0.681944
Adj Rsquare	0.562673
Root Mean Square Error	0.003066
Mean of Response	0.39004
Observations (or Sum Wgts)	12

Analysis of Variance

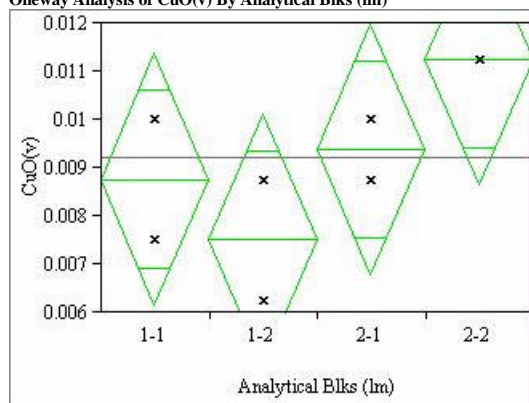
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00016127	0.000054	5.7176	0.0217
Error	8	0.00007522	0.000009		
C. Total	11	0.00023649			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	0.385972	0.00177	0.38189	0.39005
1-2	3	0.386806	0.00177	0.38272	0.39089
2-1	3	0.393900	0.00177	0.38982	0.39798
2-2	3	0.393482	0.00177	0.38940	0.39756

Std Error uses a pooled estimate of error variance

Oneway Analysis of CuO(v) By Analytical Blks (lm)



Oneway Anova
Summary of Fit

Rsquare	0.675676
Adj Rsquare	0.432432
Root Mean Square Error	0.001328
Mean of Response	0.009232
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00001469	0.0000049	2.7778	0.1744
Error	4	0.00000705	0.0000018		
C. Total	7	0.00002174			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	0.008763	0.00094	0.00616	0.01137
1-2	2	0.007511	0.00094	0.00490	0.01012
2-1	2	0.009389	0.00094	0.00678	0.01200
2-2	2	0.011266	0.00094	0.00866	0.01387

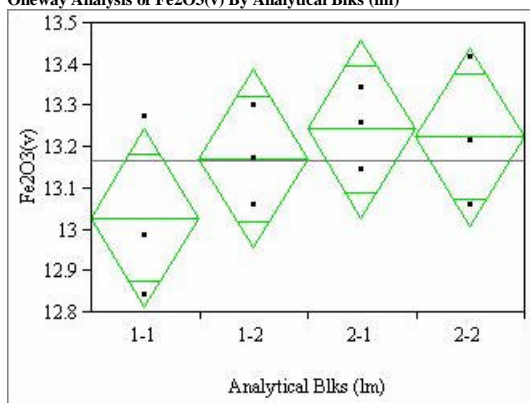
Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block

(continued)

(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of Fe2O3(v) By Analytical Blks (lm)



**Oneway Anova
Summary of Fit**

Rsquare	0.2879
Adj Rsquare	0.020862
Root Mean Square Error	0.161752
Mean of Response	13.16754
Observations (or Sum Wgts)	12

Analysis of Variance

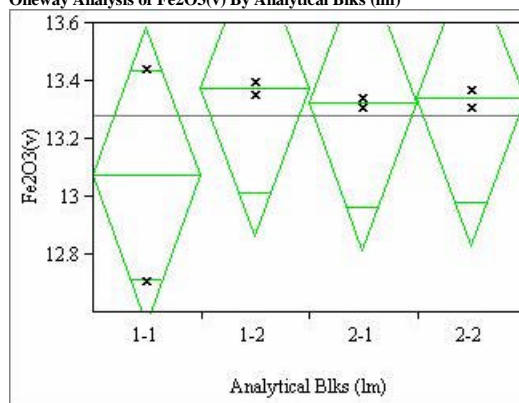
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.08462334	0.028208	1.0781	0.4116
Error	8	0.20930991	0.026164		
C. Total	11	0.29393325			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	13.0293	0.09339	12.814	13.245
1-2	3	13.1723	0.09339	12.957	13.388
2-1	3	13.2438	0.09339	13.028	13.459
2-2	3	13.2247	0.09339	13.009	13.440

Std Error uses a pooled estimate of error variance

Oneway Analysis of Fe2O3(v) By Analytical Blks (lm)



**Oneway Anova
Summary of Fit**

Rsquare	0.296603
Adj Rsquare	-0.23094
Root Mean Square Error	0.259226
Mean of Response	13.27834
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.11334213	0.037781	0.5622	0.6680
Error	4	0.26879153	0.067198		
C. Total	7	0.38213367			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	13.0746	0.18330	12.566	13.584
1-2	2	13.3748	0.18330	12.866	13.884
2-1	2	13.3248	0.18330	12.816	13.834
2-2	2	13.3391	0.18330	12.830	13.848

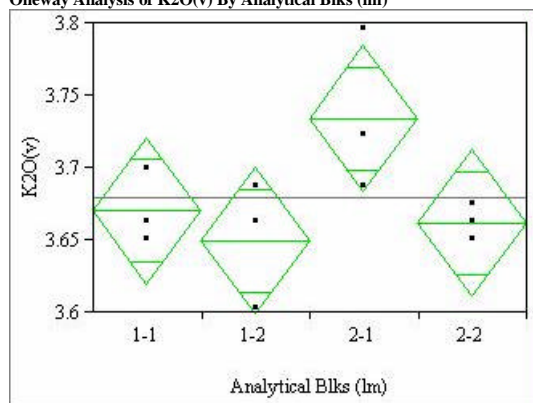
Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block

(continued)

(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of K2O(v) By Analytical Blks (lm)



**Oneway Anova
Summary of Fit**

Rsquare	0.528707
Adj Rsquare	0.351972
Root Mean Square Error	0.037774
Mean of Response	3.679049
Observations (or Sum Wgts)	12

Analysis of Variance

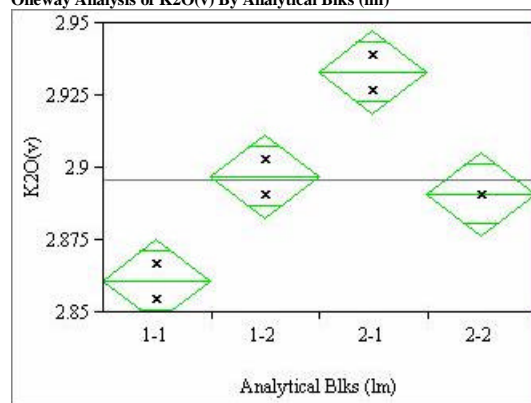
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.01280561	0.004269	2.9915	0.0957
Error	8	0.01141501	0.001427		
C. Total	11	0.02422063			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	3.67001	0.02181	3.6197	3.7203
1-2	3	3.64994	0.02181	3.5996	3.7002
2-1	3	3.73426	0.02181	3.6840	3.7846
2-2	3	3.66198	0.02181	3.6117	3.7123

Std Error uses a pooled estimate of error variance

Oneway Analysis of K2O(v) By Analytical Blks (lm)



**Oneway Anova
Summary of Fit**

Rsquare	0.960396
Adj Rsquare	0.930693
Root Mean Square Error	0.007377
Mean of Response	2.895557
Observations (or Sum Wgts)	8

Analysis of Variance

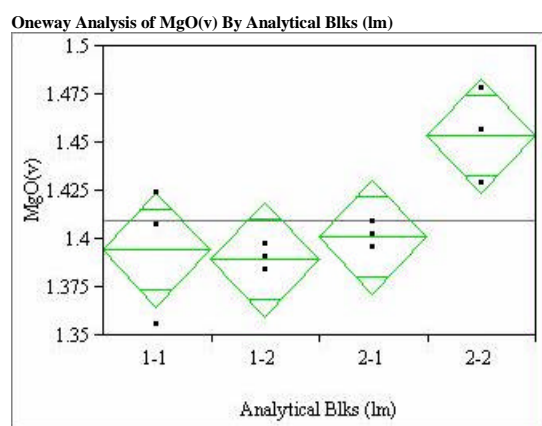
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00527823	0.001759	32.3333	0.0029
Error	4	0.00021766	0.000054		
C. Total	7	0.00549589			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	2.86092	0.00522	2.8464	2.8754
1-2	2	2.89706	0.00522	2.8826	2.9115
2-1	2	2.93320	0.00522	2.9187	2.9477
2-2	2	2.89104	0.00522	2.8766	2.9055

Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block
(continued)
(small square – Batch 1 standard; x – Ustd;)



Oneway Anova Summary of Fit

Rsquare	0.668341
Adj Rsquare	0.543969
Root Mean Square Error	0.022174
Mean of Response	1.409661
Observations (or Sum Wgts)	12

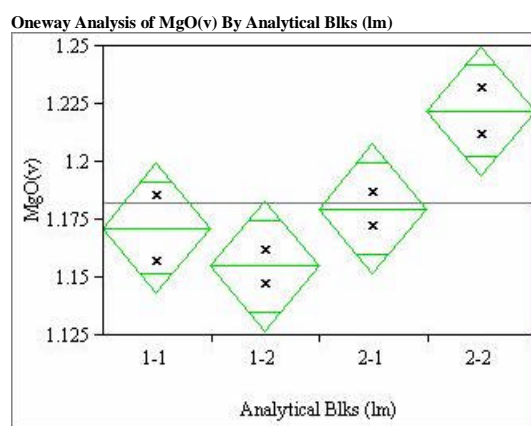
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00792622	0.002642	5.3737	0.0255
Error	8	0.00393333	0.000492		
C. Total	11	0.01185954			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	1.39446	0.01280	1.3649	1.4240
1-2	3	1.38949	0.01280	1.3600	1.4190
2-1	3	1.40109	0.01280	1.3716	1.4306
2-2	3	1.45360	0.01280	1.4241	1.4831

Std Error uses a pooled estimate of error variance



Oneway Anova Summary of Fit

Rsquare	0.857169
Adj Rsquare	0.750045
Root Mean Square Error	0.0143
Mean of Response	1.182018
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00490852	0.001636	8.0017	0.0364
Error	4	0.00081792	0.000204		
C. Total	7	0.00572644			

Means for Oneway Anova

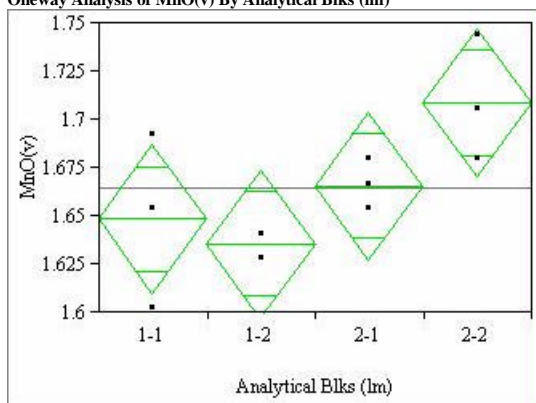
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	1.17145	0.01011	1.1434	1.1995
1-2	2	1.15487	0.01011	1.1268	1.1829
2-1	2	1.17974	0.01011	1.1517	1.2078
2-2	2	1.22202	0.01011	1.1939	1.2501

Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block

(continued)
(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of MnO(v) By Analytical Blks (lm)



Oneway Anova
Summary of Fit

Rsquare	0.578578
Adj Rsquare	0.420544
Root Mean Square Error	0.028872
Mean of Response	1.664572
Observations (or Sum Wgts)	12

Analysis of Variance

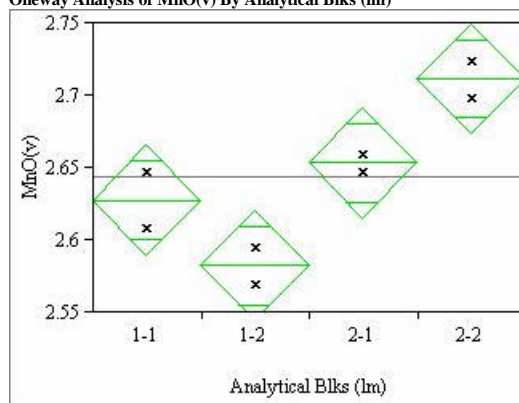
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00915569	0.003052	3.6611	0.0631
Error	8	0.00666879	0.000834		
C. Total	11	0.01582448			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	1.64843	0.01667	1.6100	1.6869
1-2	3	1.63552	0.01667	1.5971	1.6740
2-1	3	1.66565	0.01667	1.6272	1.7041
2-2	3	1.70869	0.01667	1.6702	1.7471

Std Error uses a pooled estimate of error variance

Oneway Analysis of MnO(v) By Analytical Blks (lm)



Oneway Anova
Summary of Fit

Rsquare	0.920705
Adj Rsquare	0.861233
Root Mean Square Error	0.019368
Mean of Response	2.643732
Observations (or Sum Wgts)	8

Analysis of Variance

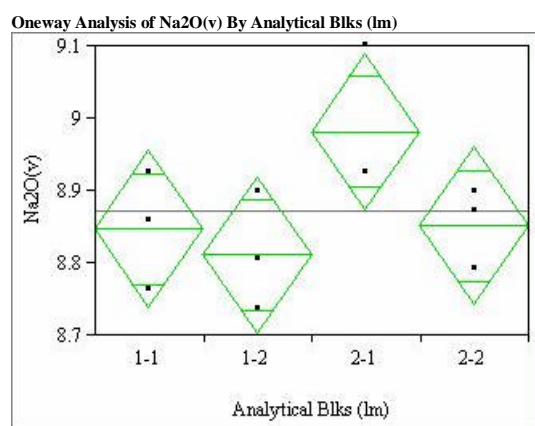
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.01742221	0.005807	15.4815	0.0115
Error	4	0.00150048	0.000375		
C. Total	7	0.01892269			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	2.62759	0.01370	2.5896	2.6656
1-2	2	2.58240	0.01370	2.5444	2.6204
2-1	2	2.65342	0.01370	2.6154	2.6914
2-2	2	2.71152	0.01370	2.6735	2.7495

Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block
(continued)
(small square – Batch 1 standard; x – Ustd;)



Oneway Anova

Summary of Fit

Rsquare	0.486775
Adj Rsquare	0.294315
Root Mean Square Error	0.081533
Mean of Response	8.87321
Observations (or Sum Wgts)	12

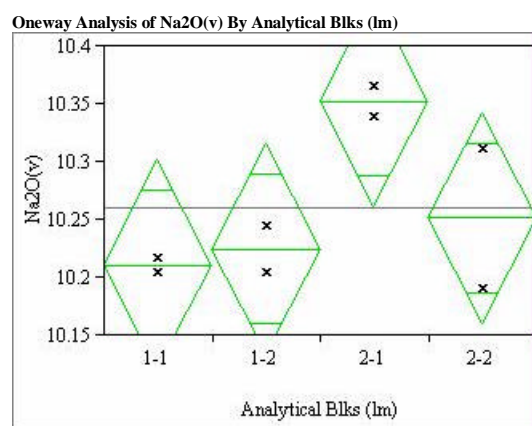
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.05043978	0.016813	2.5292	0.1308
Error	8	0.05318058	0.006648		
C. Total	11	0.10362036			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	8.84737	0.04707	8.7388	8.9559
1-2	3	8.81143	0.04707	8.7029	8.9200
2-1	3	8.98217	0.04707	8.8736	9.0907
2-2	3	8.85187	0.04707	8.7433	8.9604

Std Error uses a pooled estimate of error variance



Oneway Anova

Summary of Fit

Rsquare	0.74026
Adj Rsquare	0.545455
Root Mean Square Error	0.046452
Mean of Response	10.25997
Observations (or Sum Wgts)	8

Analysis of Variance

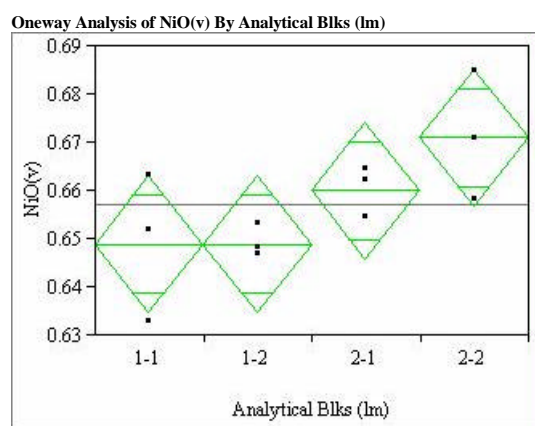
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.02459905	0.008200	3.8000	0.1149
Error	4	0.00863124	0.002158		
C. Total	7	0.03323029			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	10.2111	0.03285	10.120	10.302
1-2	2	10.2246	0.03285	10.133	10.316
2-1	2	10.3526	0.03285	10.261	10.444
2-2	2	10.2515	0.03285	10.160	10.343

Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block
(continued)
(small square – Batch 1 standard; x – Ustd;)



Oneway Anova

Summary of Fit

Rsquare	0.52381
Adj Rsquare	0.345238
Root Mean Square Error	0.010678
Mean of Response	0.657246
Observations (or Sum Wgts)	12

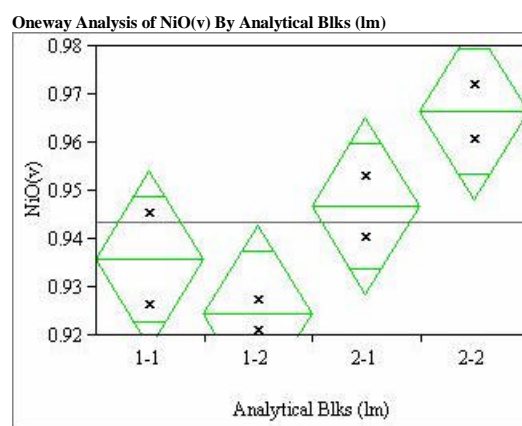
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00100340	0.000334	2.9333	0.0994
Error	8	0.00091218	0.000114		
C. Total	11	0.00191558			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	0.648975	0.00617	0.63476	0.66319
1-2	3	0.648975	0.00617	0.63476	0.66319
2-1	3	0.660003	0.00617	0.64579	0.67422
2-2	3	0.671032	0.00617	0.65682	0.68525

Std Error uses a pooled estimate of error variance



Oneway Anova

Summary of Fit

Rsquare	0.845837
Adj Rsquare	0.730216
Root Mean Square Error	0.00934
Mean of Response	0.9434
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00191457	0.000638	7.3155	0.0422
Error	4	0.00034895	0.000087		
C. Total	7	0.00226352			

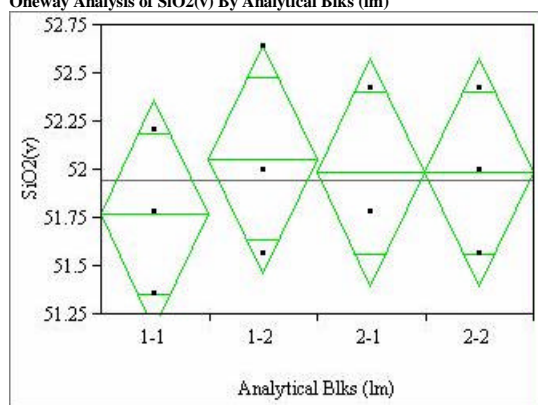
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	0.935924	0.00660	0.91759	0.95426
1-2	2	0.924471	0.00660	0.90613	0.94281
2-1	2	0.946740	0.00660	0.92840	0.96508
2-2	2	0.966464	0.00660	0.94813	0.98480

Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block
(continued)
(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of SiO2(v) By Analytical Blks (lm)



**Oneway Anova
Summary of Fit**

Rsquare	0.079646
Adj Rsquare	-0.26549
Root Mean Square Error	0.445331
Mean of Response	51.94933
Observations (or Sum Wgts)	12

Analysis of Variance

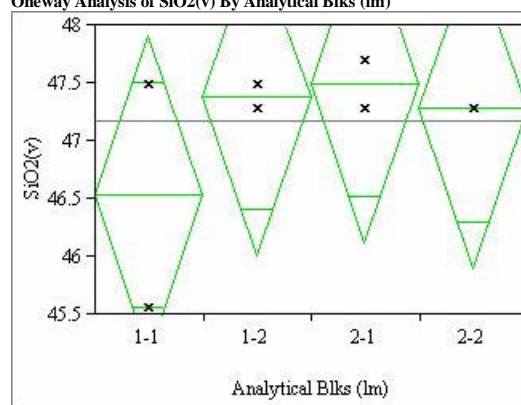
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.1372981	0.045766	0.2308	0.8725
Error	8	1.5865562	0.198320		
C. Total	11	1.7238544			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	51.7711	0.25711	51.178	52.364
1-2	3	52.0563	0.25711	51.463	52.649
2-1	3	51.9850	0.25711	51.392	52.578
2-2	3	51.9850	0.25711	51.392	52.578

Std Error uses a pooled estimate of error variance

Oneway Analysis of SiO2(v) By Analytical Blks (lm)



**Oneway Anova
Summary of Fit**

Rsquare	0.367647
Adj Rsquare	-0.10662
Root Mean Square Error	0.701416
Mean of Response	47.17157
Observations (or Sum Wgts)	8

Analysis of Variance

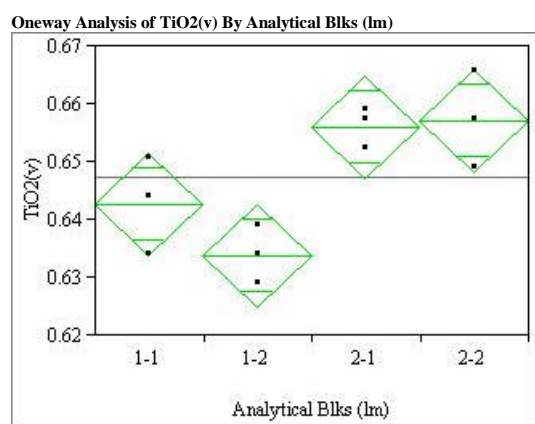
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	1.1441511	0.381384	0.7752	0.5656
Error	4	1.9679399	0.491985		
C. Total	7	3.1120911			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	46.5298	0.49598	45.153	47.907
1-2	2	47.3855	0.49598	46.008	48.763
2-1	2	47.4925	0.49598	46.115	48.870
2-2	2	47.2785	0.49598	45.901	48.656

Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block
(continued)
(small square – Batch 1 standard; x – Ustd;)



Oneway Anova

Summary of Fit

Rsquare	0.761399
Adj Rsquare	0.671924
Root Mean Square Error	0.006655
Mean of Response	0.647462
Observations (or Sum Wgts)	12

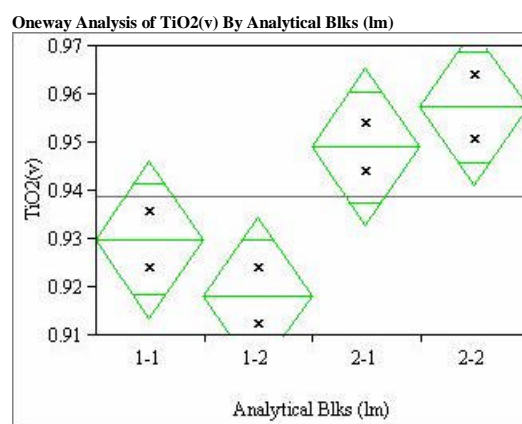
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00113051	0.000377	8.5096	0.0072
Error	8	0.00035427	0.000044		
C. Total	11	0.00148478			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	0.642736	0.00384	0.63388	0.65160
1-2	3	0.633840	0.00384	0.62498	0.64270
2-1	3	0.656080	0.00384	0.64722	0.66494
2-2	3	0.657192	0.00384	0.64833	0.66605

Std Error uses a pooled estimate of error variance



Oneway Anova

Summary of Fit

Rsquare	0.873966
Adj Rsquare	0.77944
Root Mean Square Error	0.008298
Mean of Response	0.938667
Observations (or Sum Wgts)	8

Analysis of Variance

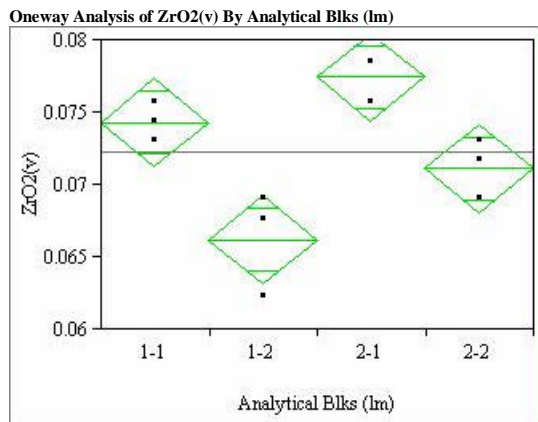
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00191000	0.000637	9.2458	0.0285
Error	4	0.00027544	0.000069		
C. Total	7	0.00218544			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	0.929910	0.00587	0.91362	0.94620
1-2	2	0.918234	0.00587	0.90194	0.93453
2-1	2	0.949092	0.00587	0.93280	0.96538
2-2	2	0.957432	0.00587	0.94114	0.97372

Std Error uses a pooled estimate of error variance

Exhibit A.3: Measurements of Standards Prepared Using the Lithium Metaborate (LM) Method by Oxide by Analytical Block
(continued)
(small square – Batch 1 standard; x – Ustd;)



Oneway Anova Summary of Fit

Rsquare	0.829684
Adj Rsquare	0.765815
Root Mean Square Error	0.002307
Mean of Response	0.072268
Observations (or Sum Wgts)	12

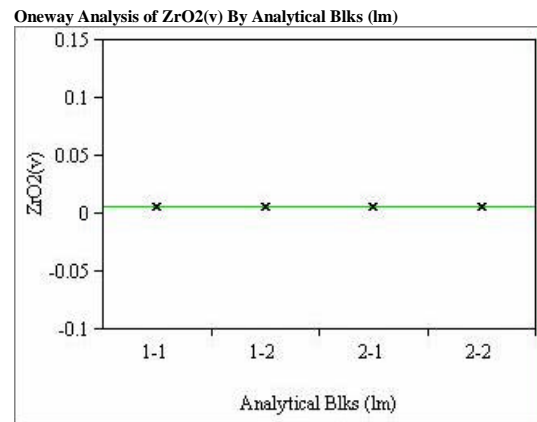
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0.00020740	0.000069	12.9905	0.0019
Error	8	0.00004258	0.000005		
C. Total	11	0.00024998			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	0.074294	0.00133	0.07122	0.07737
1-2	3	0.066189	0.00133	0.06312	0.06926
2-1	3	0.077446	0.00133	0.07437	0.08052
2-2	3	0.071142	0.00133	0.06807	0.07421

Std Error uses a pooled estimate of error variance



Oneway Anova Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.006754
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Blks (lm)	3	0	0		-1.0000
Error	4	0	0		
C. Total	7	0			

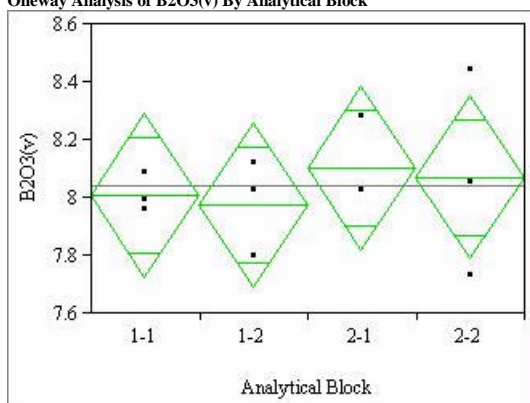
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	0.006754	0	0.00675	0.00675
1-2	2	0.006754	0	0.00675	0.00675
2-1	2	0.006754	0	0.00675	0.00675
2-2	2	0.006754	0	0.00675	0.00675

Std Error uses a pooled estimate of error variance

Exhibit A.4: Measurements of Standards Prepared Using the Peroxide Fusion (pf) Method by Oxide by Analytical Block
(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of B2O3(v) By Analytical Block



Oneway Anova
Summary of Fit

Rsquare	0.079646
Adj Rsquare	-0.26549
Root Mean Square Error	0.21196
Mean of Response	8.039017
Observations (or Sum Wgts)	12

Analysis of Variance

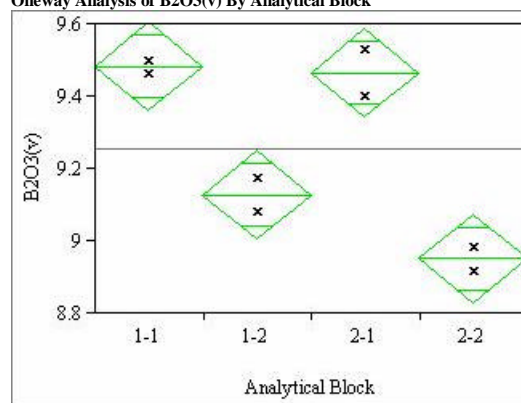
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Block	3	0.03110327	0.010368	0.2308	0.8725
Error	8	0.35941554	0.044927		
C. Total	11	0.39051881			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	8.00682	0.12238	7.7246	8.2890
1-2	3	7.97462	0.12238	7.6924	8.2568
2-1	3	8.10342	0.12238	7.8212	8.3856
2-2	3	8.07122	0.12238	7.7890	8.3534

Std Error uses a pooled estimate of error variance

Oneway Analysis of B2O3(v) By Analytical Block



Oneway Anova
Summary of Fit

Rsquare	0.963415
Adj Rsquare	0.935976
Root Mean Square Error	0.062353
Mean of Response	9.257212
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Block	3	0.40952636	0.136509	35.1111	0.0025
Error	4	0.01555163	0.003888		
C. Total	7	0.42507800			

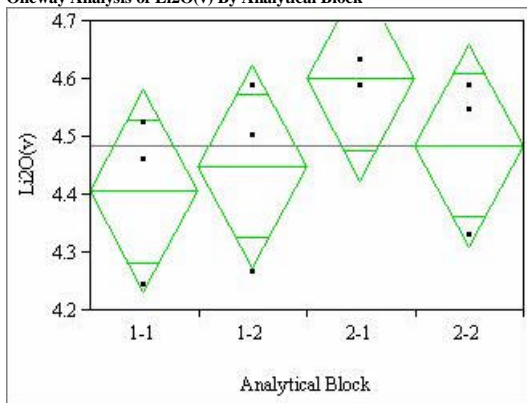
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	9.48261	0.04409	9.3602	9.6050
1-2	2	9.12842	0.04409	9.0060	9.2508
2-1	2	9.46651	0.04409	9.3441	9.5889
2-2	2	8.95132	0.04409	8.8289	9.0737

Std Error uses a pooled estimate of error variance

Exhibit A.4: Measurements of Standards Prepared Using the Peroxide Fusion (pf) Method by Oxide by Analytical Block
(small square – Batch 1 standard; x – Ustd;)

Oneway Analysis of Li2O(v) By Analytical Block



Oneway Anova
Summary of Fit

Rsquare	0.309707
Adj Rsquare	0.050847
Root Mean Square Error	0.131544
Mean of Response	4.485208
Observations (or Sum Wgts)	12

Analysis of Variance

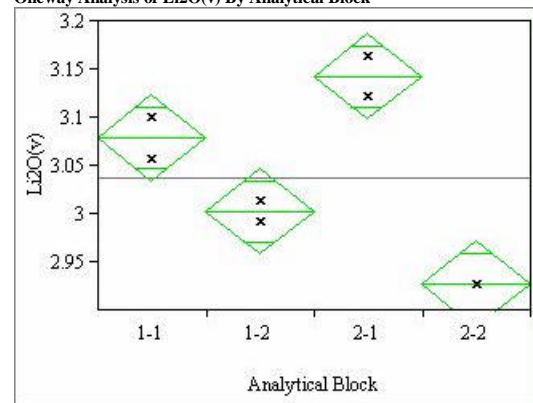
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Block	3	0.06210871	0.020703	1.1964	0.3712
Error	8	0.13843136	0.017304		
C. Total	11	0.20054007			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	4.40627	0.07595	4.2311	4.5814
1-2	3	4.44933	0.07595	4.2742	4.6245
2-1	3	4.60003	0.07595	4.4249	4.7752
2-2	3	4.48521	0.07595	4.3101	4.6603

Std Error uses a pooled estimate of error variance

Oneway Analysis of Li2O(v) By Analytical Block



Oneway Anova
Summary of Fit

Rsquare	0.961497
Adj Rsquare	0.93262
Root Mean Square Error	0.022835
Mean of Response	3.03828
Observations (or Sum Wgts)	8

Analysis of Variance

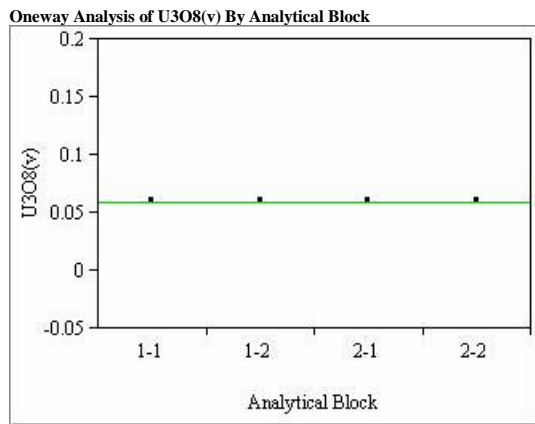
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Block	3	0.05208557	0.017362	33.2963	0.0027
Error	4	0.00208574	0.000521		
C. Total	7	0.05417131			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	3.07865	0.01615	3.0338	3.1235
1-2	2	3.00330	0.01615	2.9585	3.0481
2-1	2	3.14323	0.01615	3.0984	3.1881
2-2	2	2.92794	0.01615	2.8831	2.9728

Std Error uses a pooled estimate of error variance

Exhibit A.4: Measurements of Standards Prepared Using the Peroxide Fusion (pf) Method by Oxide by Analytical Block
(small square – Batch 1 standard; x – Ustd;)



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.05896
Observations (or Sum Wgts)	12

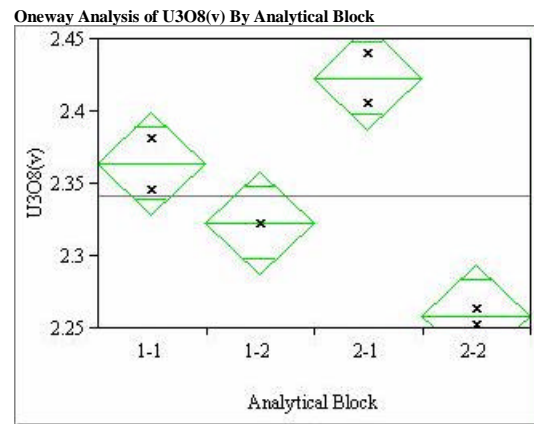
Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Block	3	0	0	.	-1.0000
Error	8	0	0		
C. Total	11	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	3	0.058960	0	0.05896	0.05896
1-2	3	0.058960	0	0.05896	0.05896
2-1	3	0.058960	0	0.05896	0.05896
2-2	3	0.058960	0	0.05896	0.05896

Std Error uses a pooled estimate of error variance



Oneway Anova
Summary of Fit

Rsquare	0.956397
Adj Rsquare	0.923695
Root Mean Square Error	0.018173
Mean of Response	2.342186
Observations (or Sum Wgts)	8

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Analytical Block	3	0.02897481	0.009658	29.2456	0.0035
Error	4	0.00132099	0.000330		
C. Total	7	0.03029579			

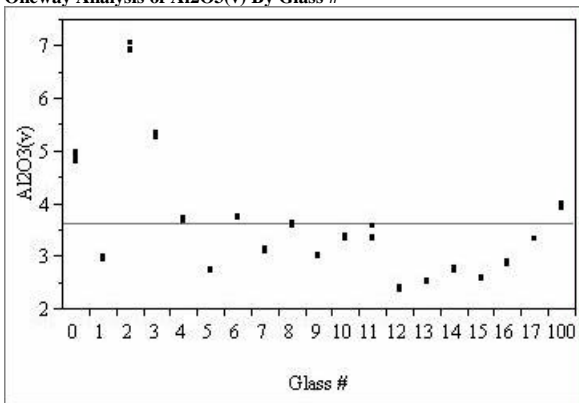
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1-1	2	2.36430	0.01285	2.3286	2.4000
1-2	2	2.32302	0.01285	2.2873	2.3587
2-1	2	2.42326	0.01285	2.3876	2.4589
2-2	2	2.25817	0.01285	2.2225	2.2938

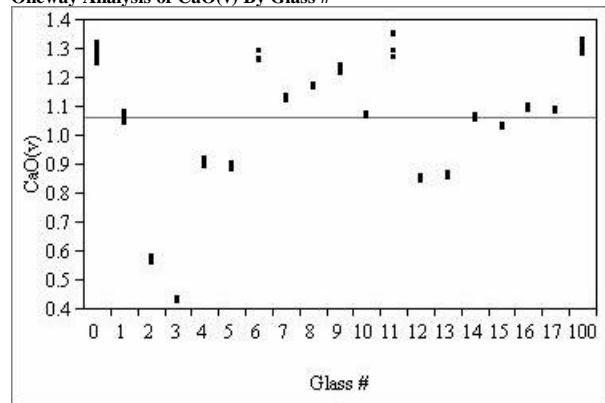
Std Error uses a pooled estimate

Exhibit A.5: Lithium Metaborate (LM) Measurements and Bias-Corrected Measurements by Glass Number by Oxide
(0 – Batch 1, 100 – Ustd, and 1-17 PHA glasses)

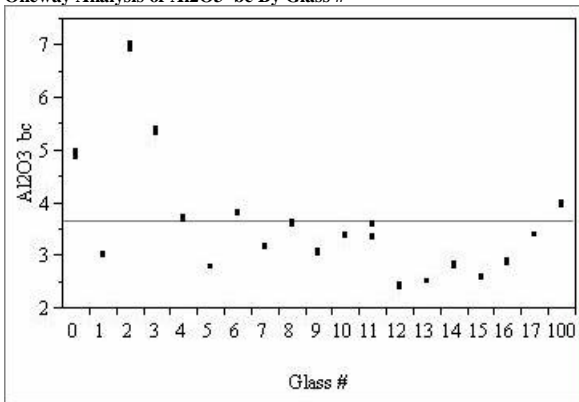
Oneway Analysis of Al₂O₃(v) By Glass #



Oneway Analysis of CaO(v) By Glass #



Oneway Analysis of Al₂O₃ bc By Glass #



Oneway Analysis of Ca₂O₃ bc By Glass #

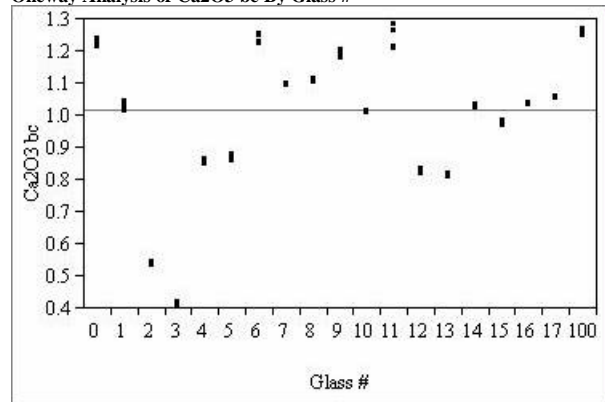
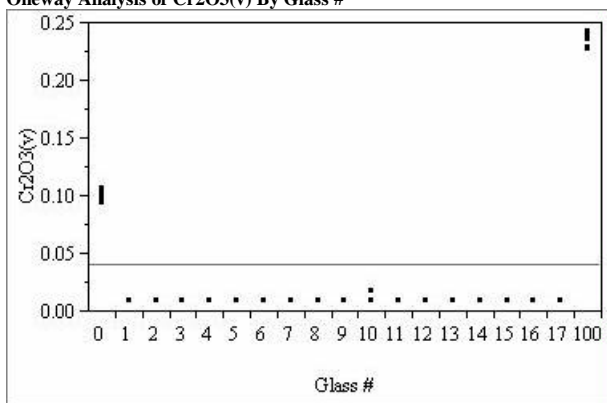
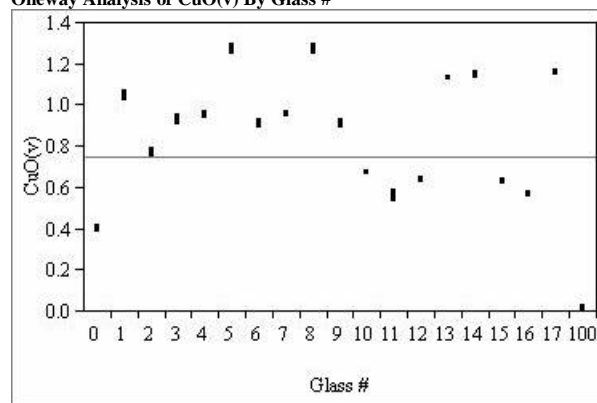


Exhibit A.5: Lithium Metaborate (LM) Measurements and Bias-Corrected Measurements by Glass Number by Oxide *(continued)*
(0 – Batch 1, 100 – Ustd, and 1-17 PHA glasses)

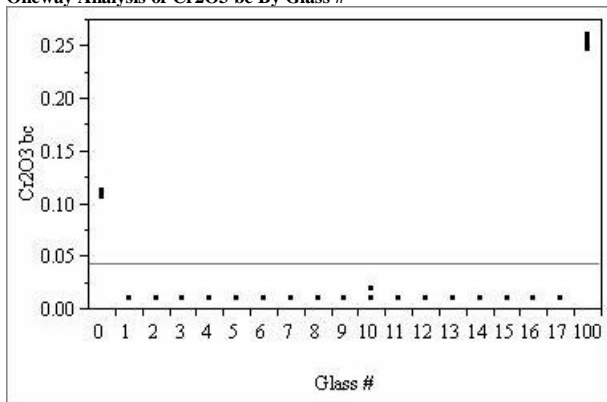
Oneway Analysis of Cr2O3(v) By Glass #



Oneway Analysis of CuO(v) By Glass #



Oneway Analysis of Cr2O3 bc By Glass #



Oneway Analysis of CuO bc By Glass #

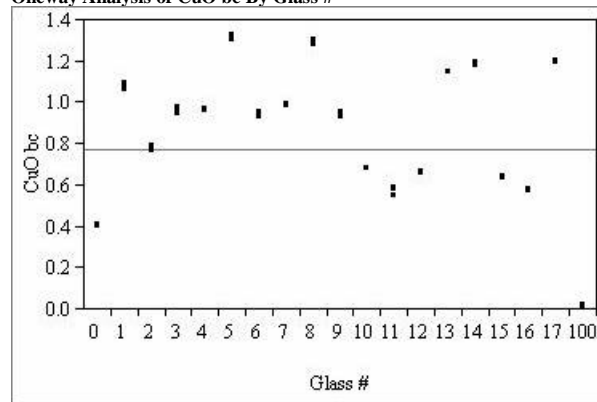
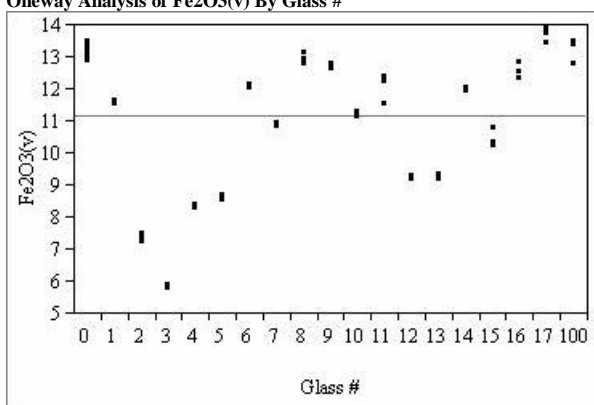
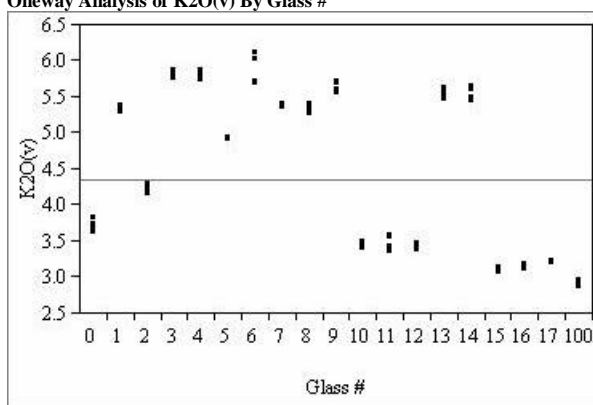


Exhibit A.5: Lithium Metaborate (LM) Measurements and Bias-Corrected Measurements by Glass Number by Oxide *(continued)*
(0 – Batch 1, 100 – Ustd, and 1-17 PHA glasses)

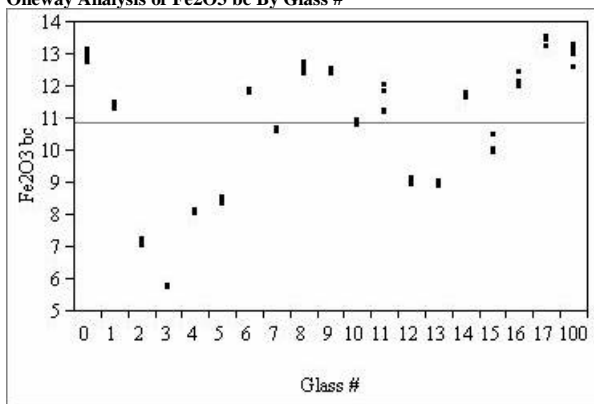
Oneway Analysis of Fe2O3(v) By Glass #



Oneway Analysis of K2O(v) By Glass #



Oneway Analysis of Fe2O3 bc By Glass #



Oneway Analysis of K2O bc By Glass #

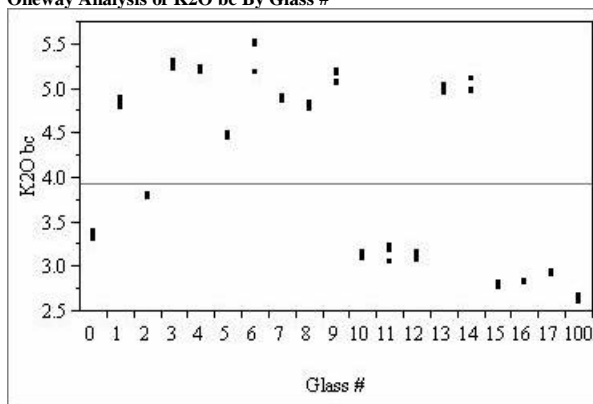
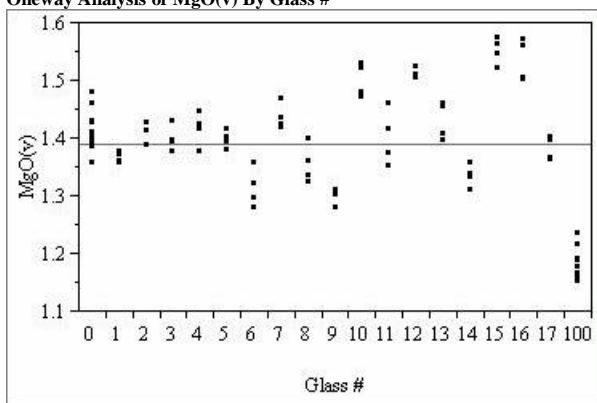
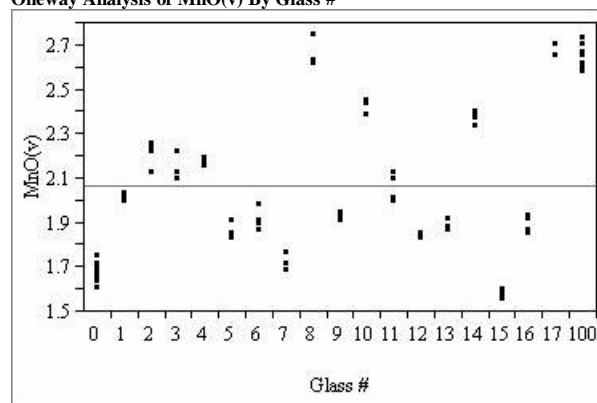


Exhibit A.5: Lithium Metaborate (LM) Measurements and Bias-Corrected Measurements by Glass Number by Oxide (*continued*)
(0 – Batch 1, 100 – Ustd, and 1-17 PHA glasses)

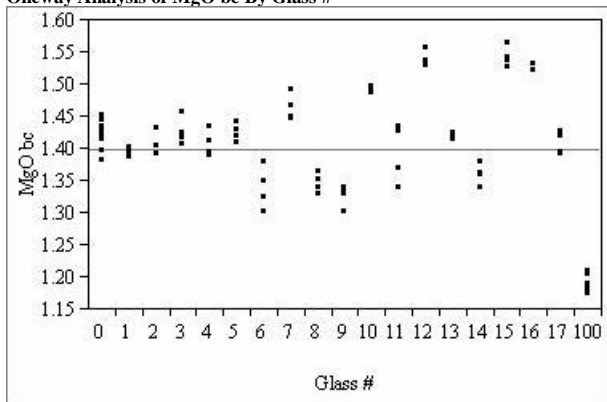
Oneway Analysis of MgO(v) By Glass #



Oneway Analysis of MnO(v) By Glass #



Oneway Analysis of MgO bc By Glass #



Oneway Analysis of MnO bc By Glass #

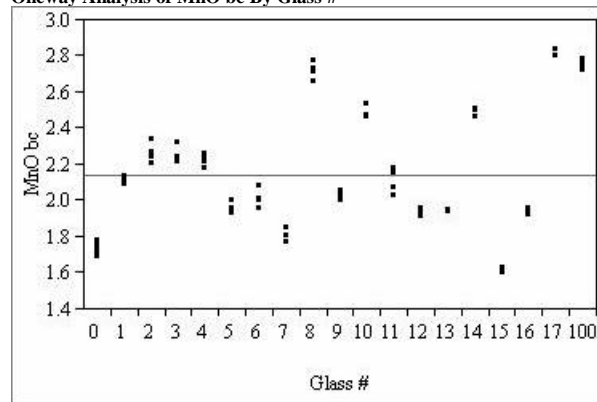
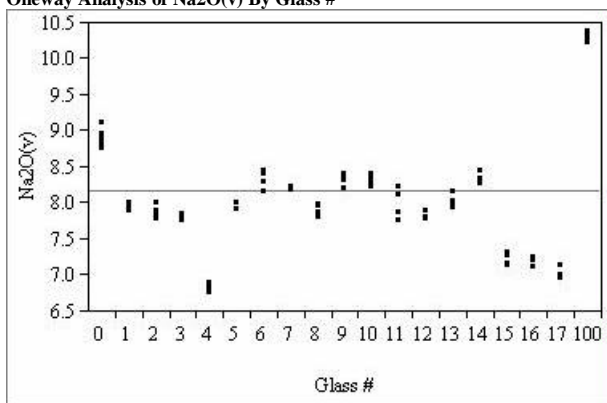
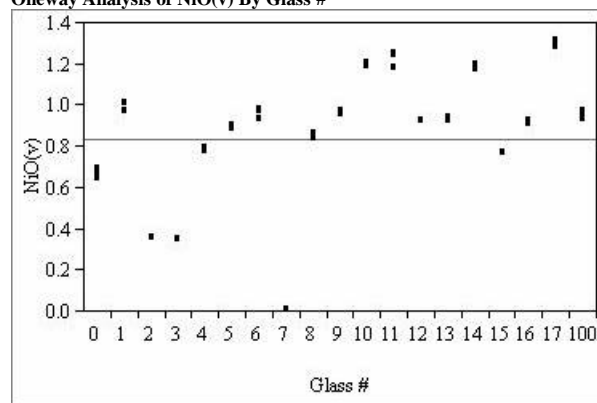


Exhibit A.5: Lithium Metaborate (LM) Measurements and Bias-Corrected Measurements by Glass Number by Oxide *(continued)*
(0 – Batch 1, 100 – Ustd, and 1-17 PHA glasses)

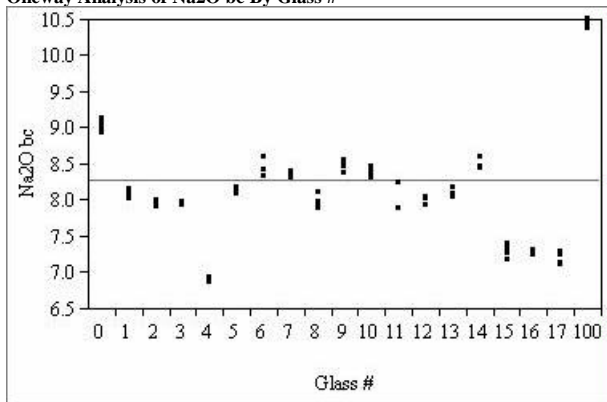
Oneway Analysis of Na2O(v) By Glass #



Oneway Analysis of NiO(v) By Glass #



Oneway Analysis of Na2O bc By Glass #



Oneway Analysis of NiO bc By Glass #

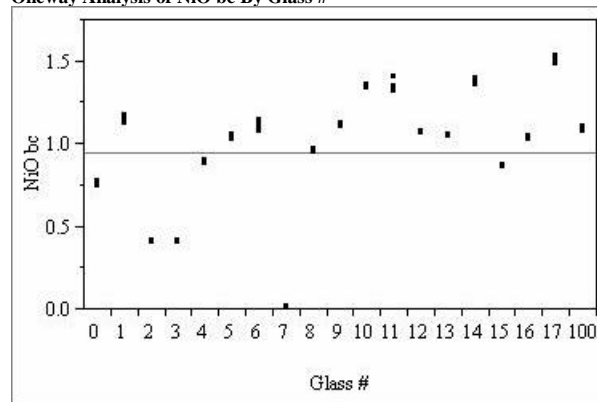
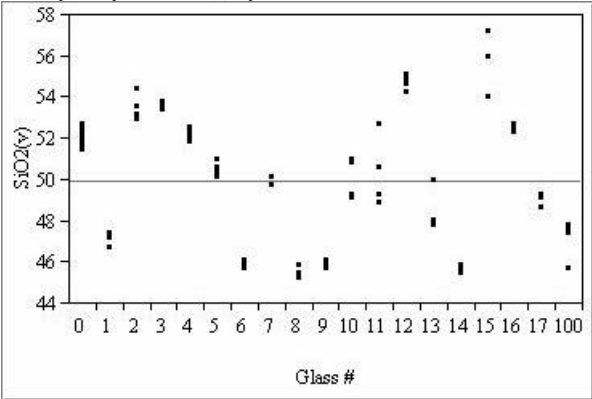
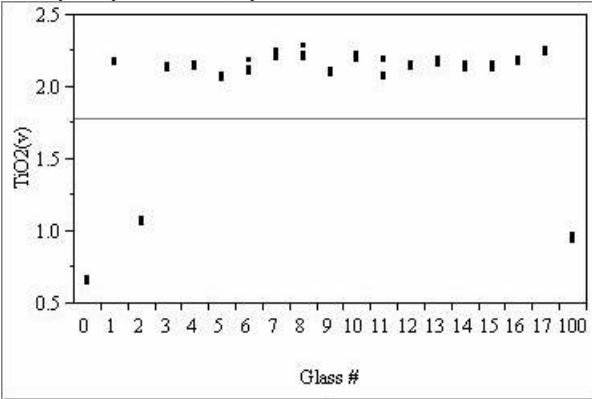


Exhibit A.5: Lithium Metaborate (LM) Measurements and Bias-Corrected Measurements by Glass Number by Oxide (continued)
(0 – Batch 1, 100 – Ustd, and 1-17 PHA glasses)

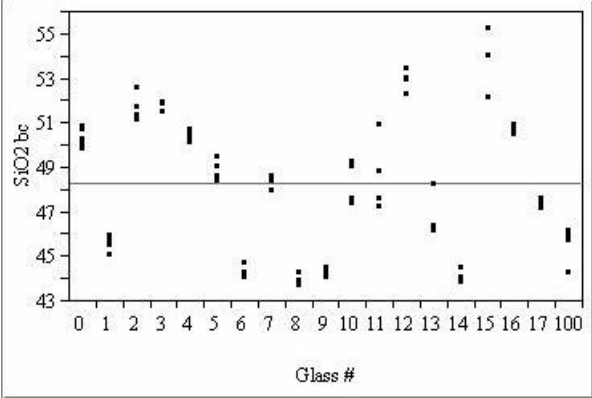
Oneway Analysis of SiO2(v) By Glass #



Oneway Analysis of TiO2(v) By Glass #



Oneway Analysis of SiO2 bc By Glass #



Oneway Analysis of TiO2 bc By Glass #

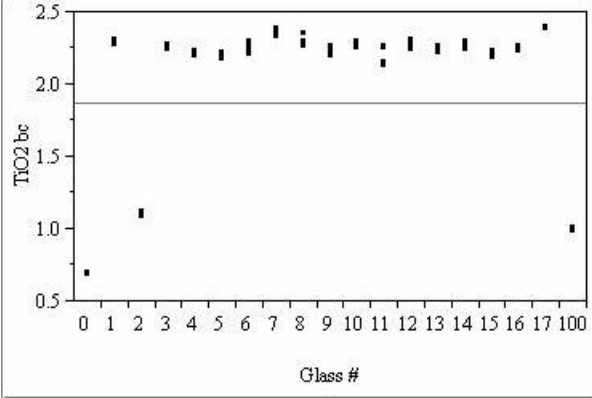


Exhibit A.5: Lithium Metaborate (LM) Measurements and Bias-Corrected Measurements by Glass Number by Oxide (*continued*)
(0 – Batch 1, 100 – Ustd, and 1-17 PHA glasses)

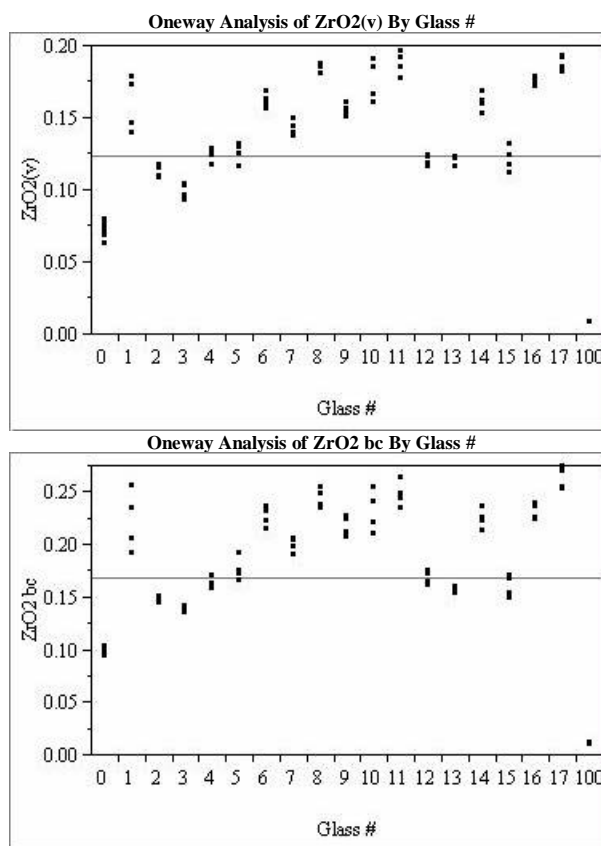
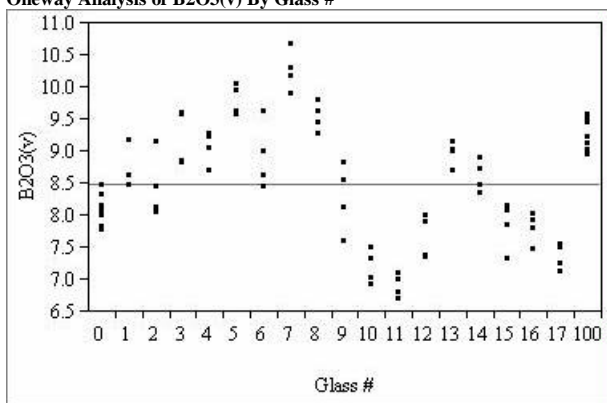
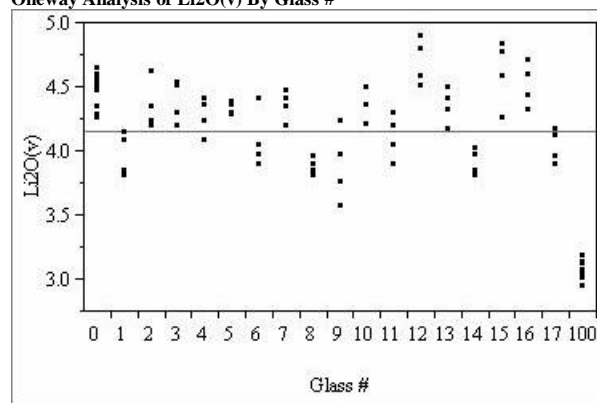


Exhibit A.6: Peroxide Fusion (pf) Measurements and Bias-Corrected Measurements by Glass Number by Oxide (continued)
(0 – Batch 1, 100 – Ustd, and 1-17 PHA glasses)

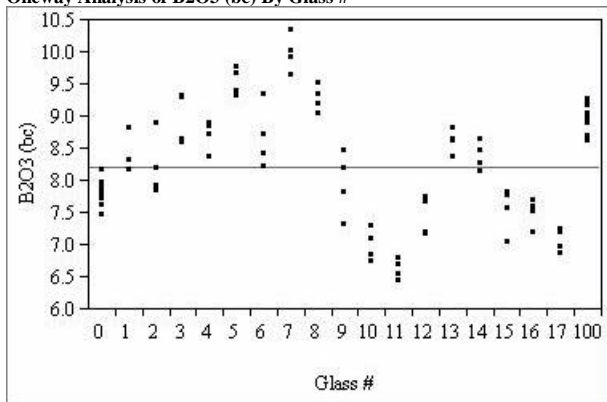
Oneway Analysis of B2O3(v) By Glass #



Oneway Analysis of Li2O(v) By Glass #



Oneway Analysis of B2O3 (bc) By Glass #



Oneway Analysis of Li2O (bc) By Glass #

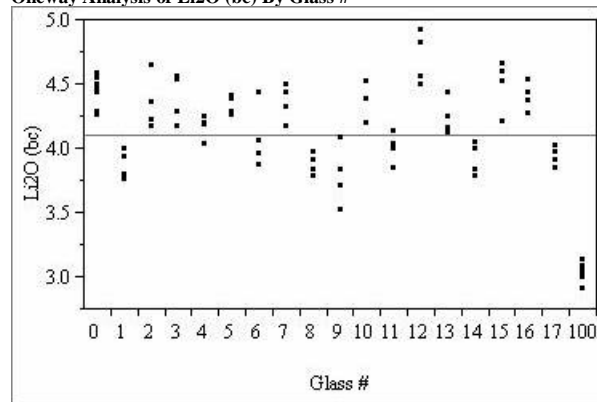


Exhibit A.6: Peroxide Fusion (pf) Measurements and Bias-Corrected (bc) Measurements by Glass Number by Oxide (continued)
(0 - Batch 1, 100 - Ustd, and 1-17 PHA glasses)

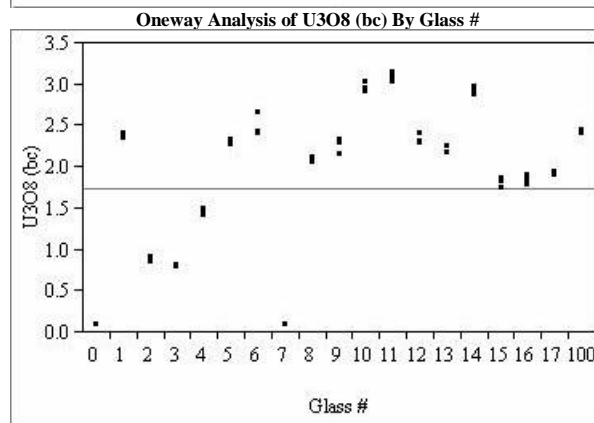
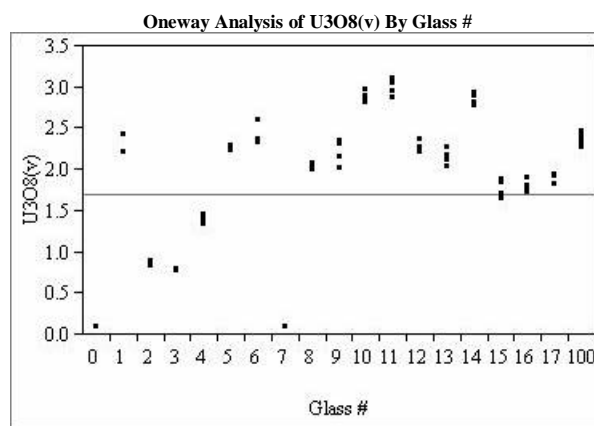
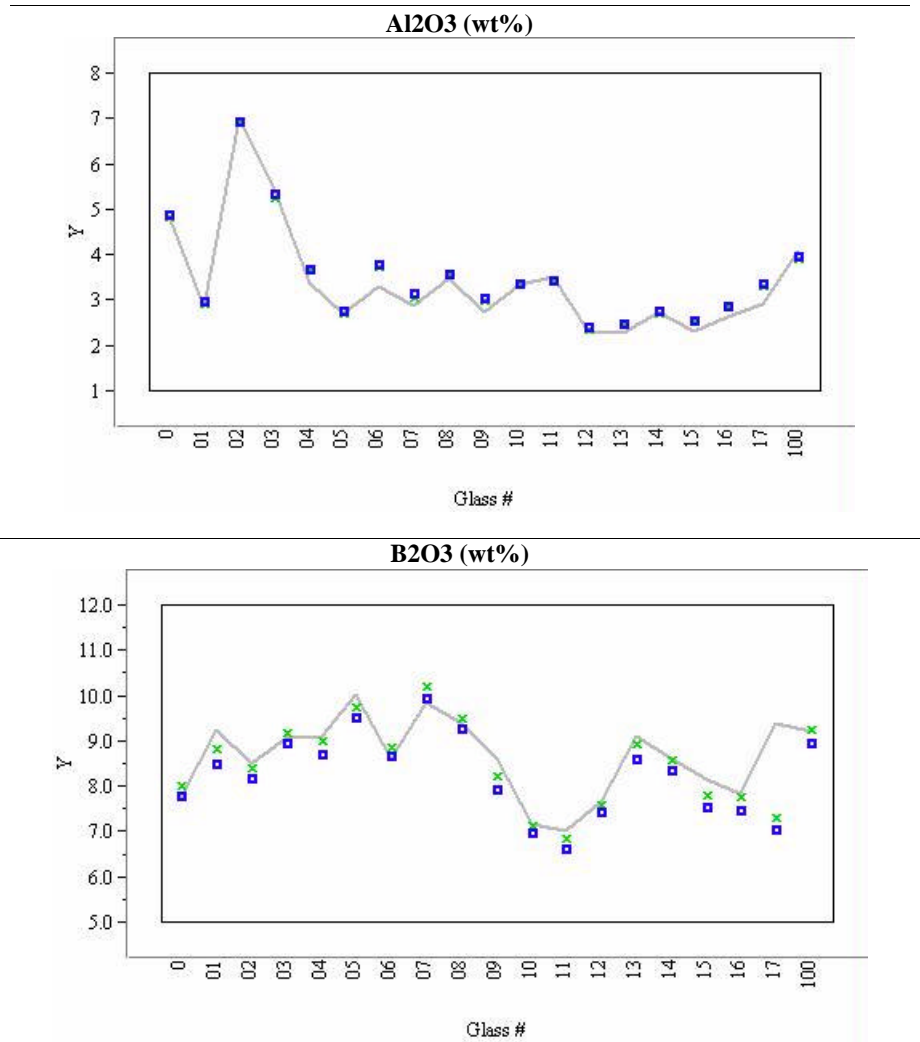


Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)



Y
— Target
* Measured
■ Measure Bias-Corrected (bc)

Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)

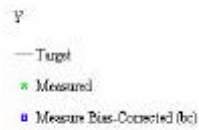
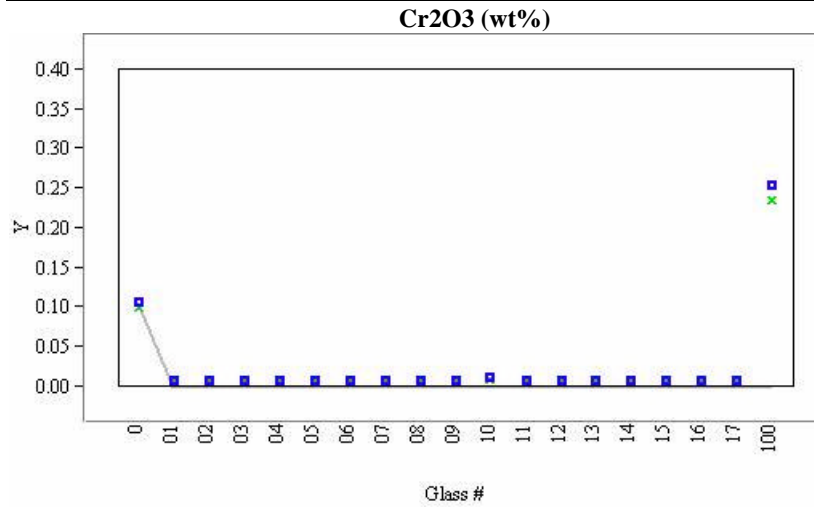
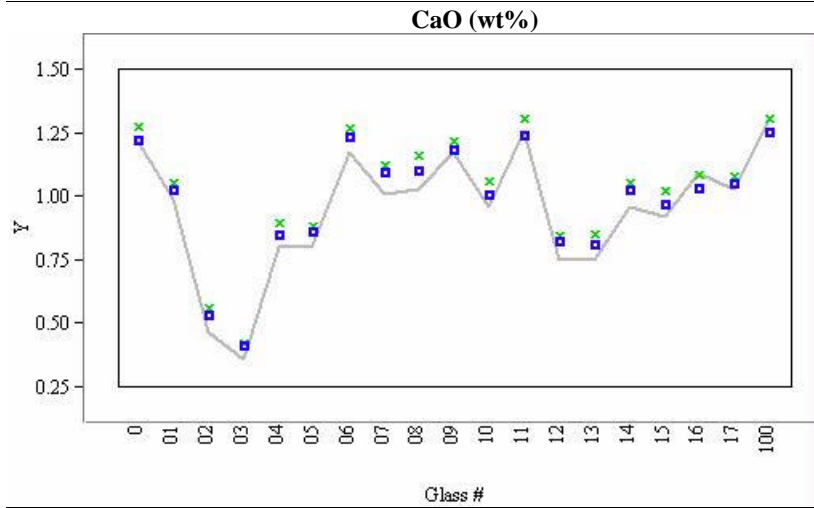
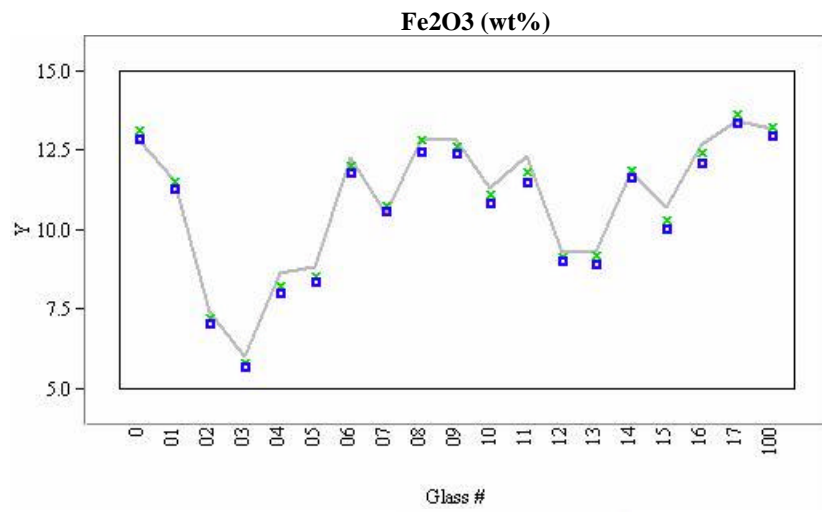
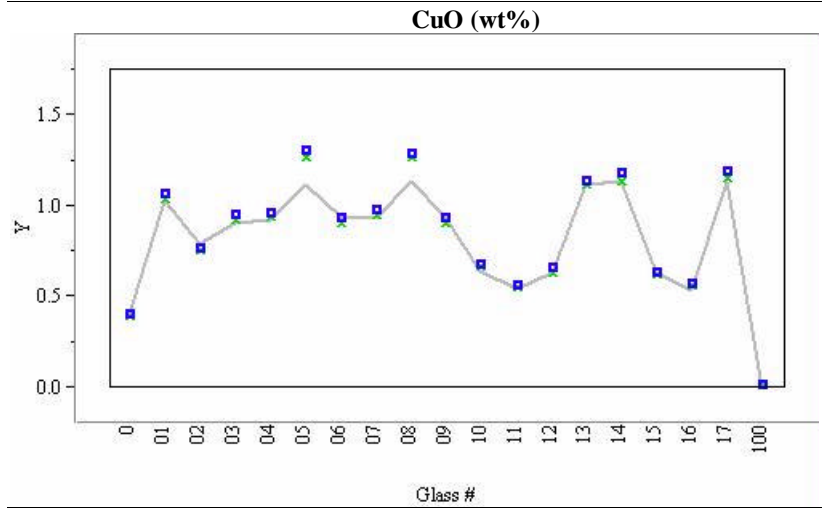


Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)



Y

- Target
- * Measured
- Measure Bias-Corrected (bc)

Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)

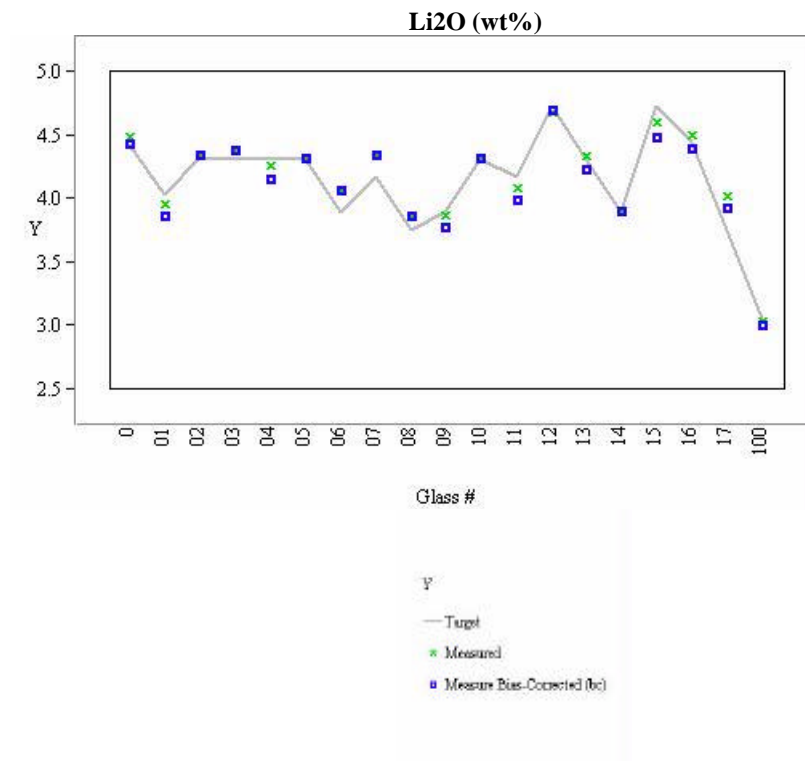
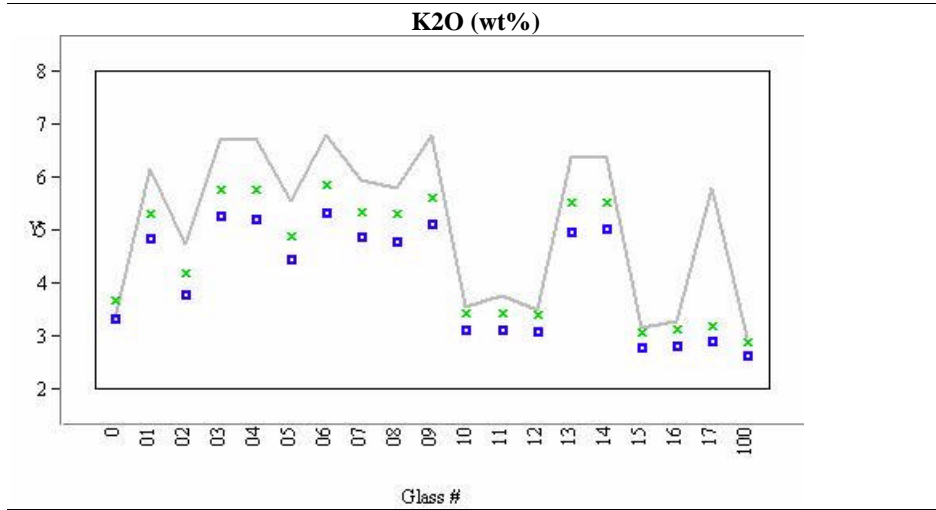


Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)

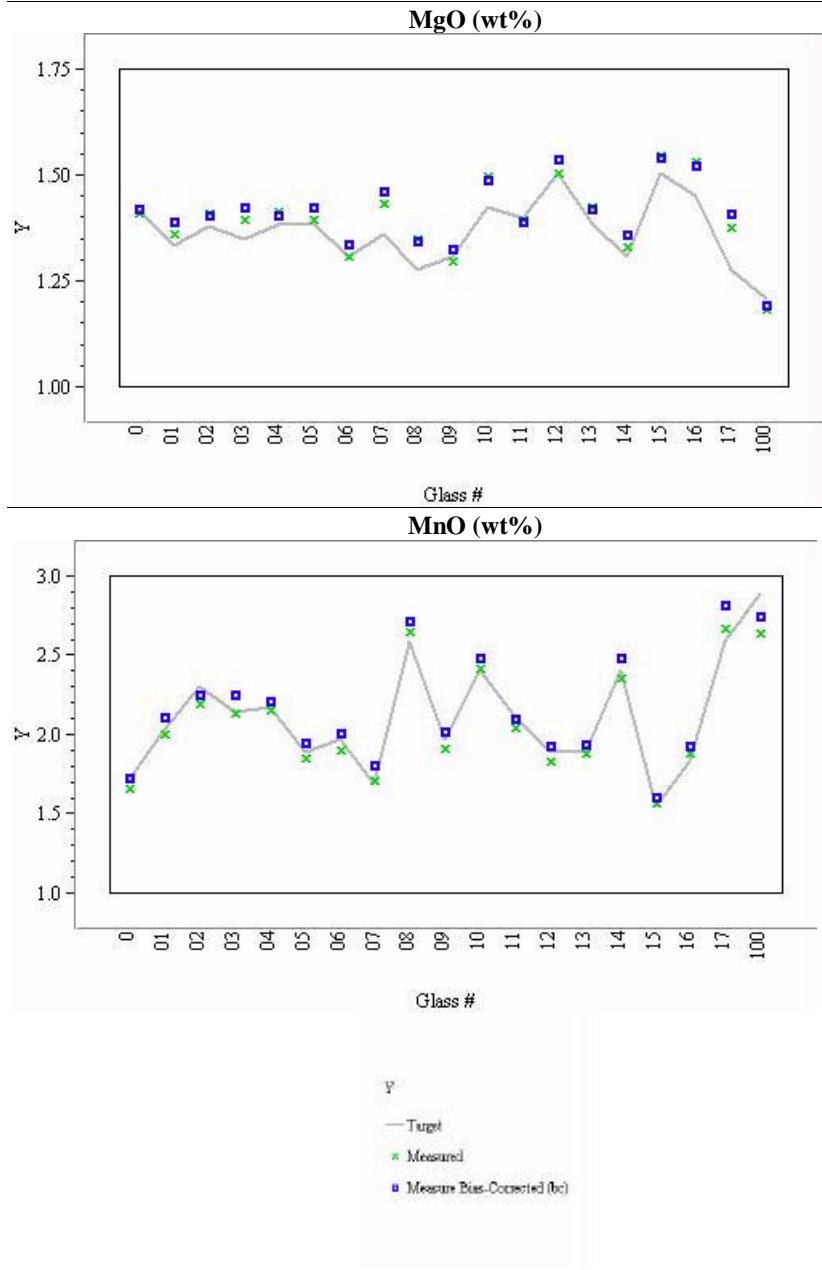
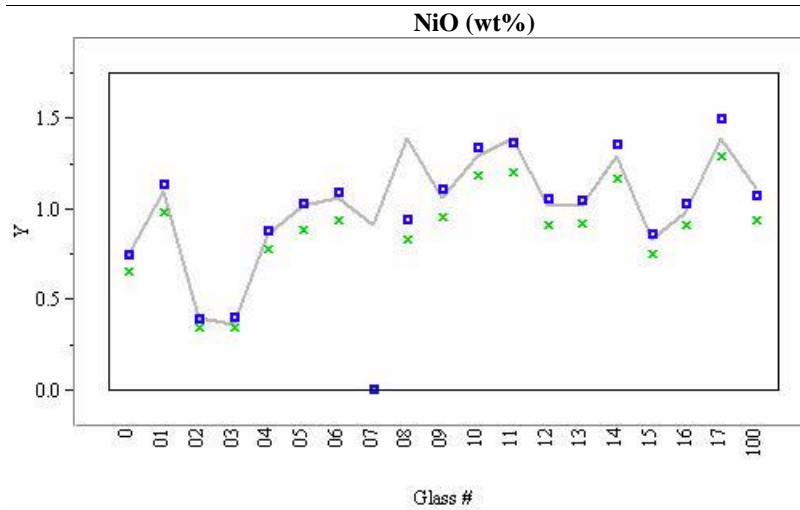
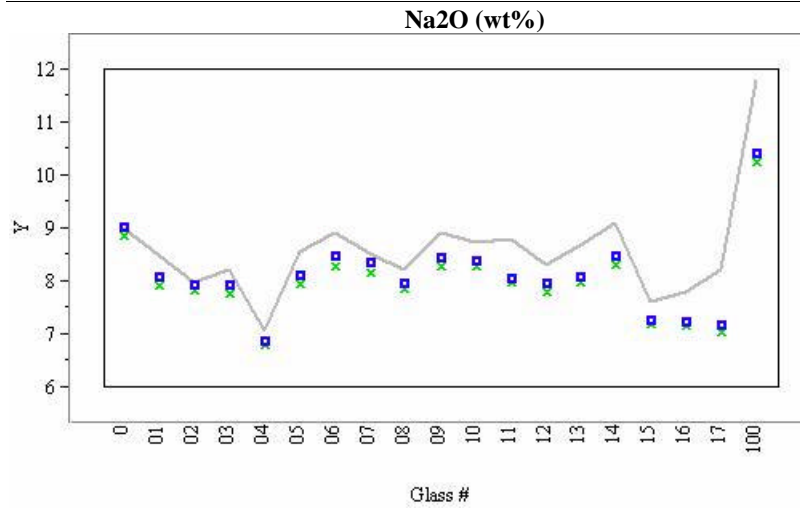


Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)



Y

- Target
- x Measured
- Measur Bias-Corrected (bc)

Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)

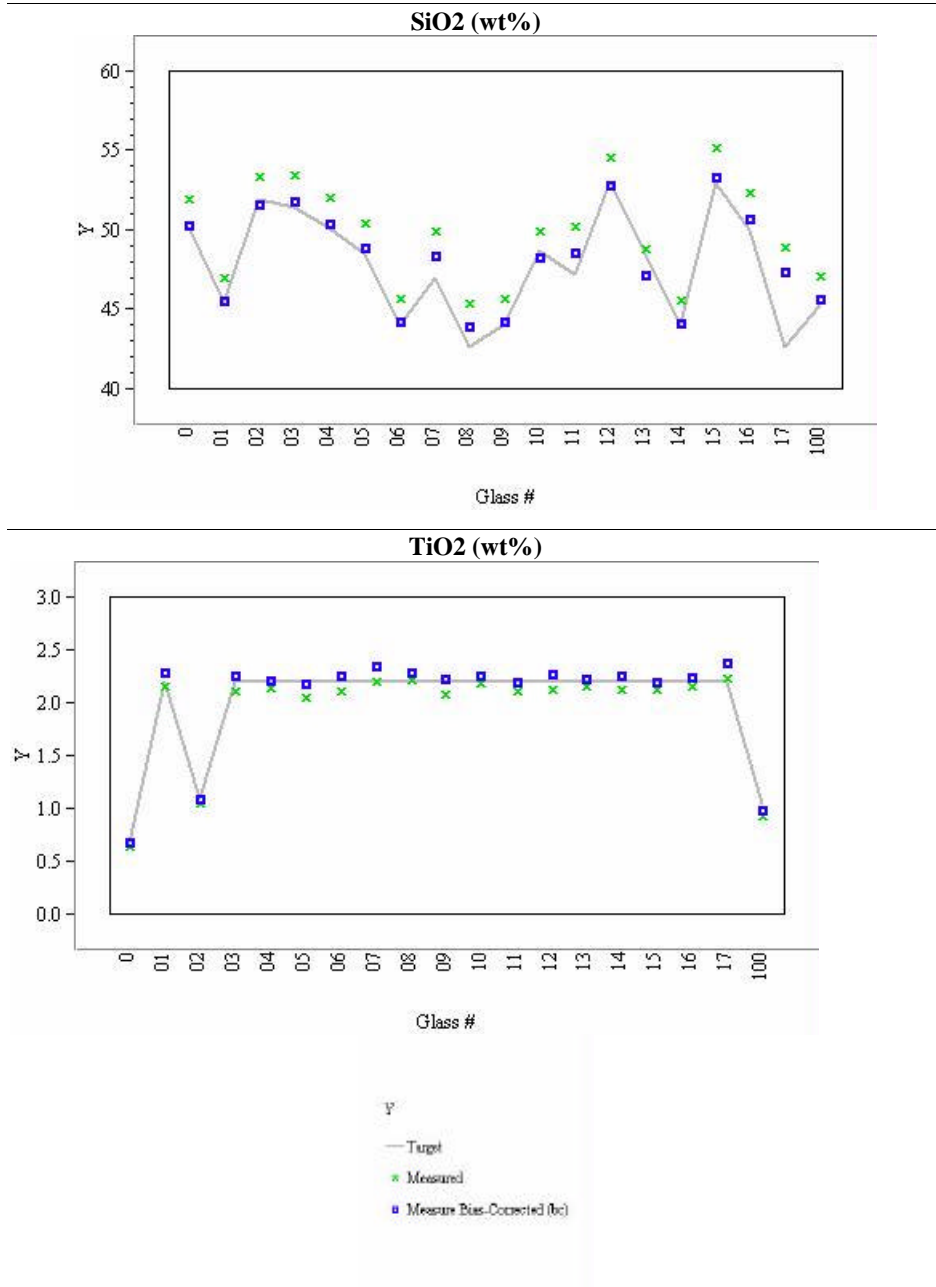
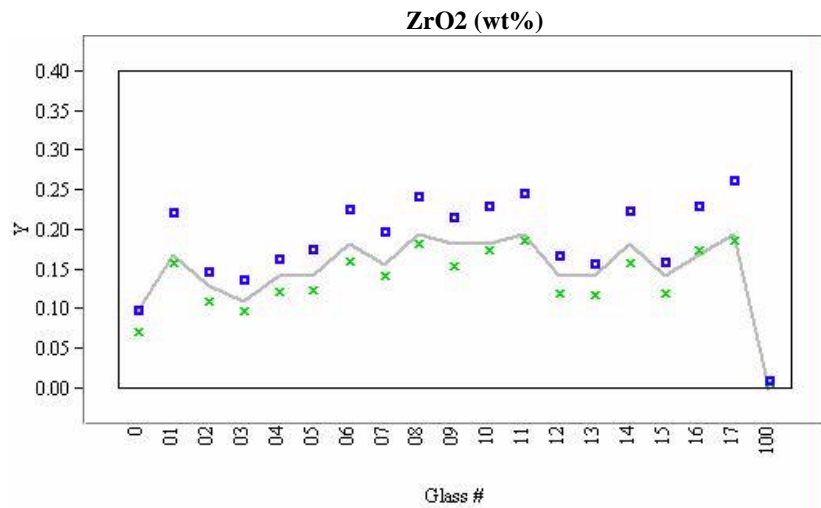
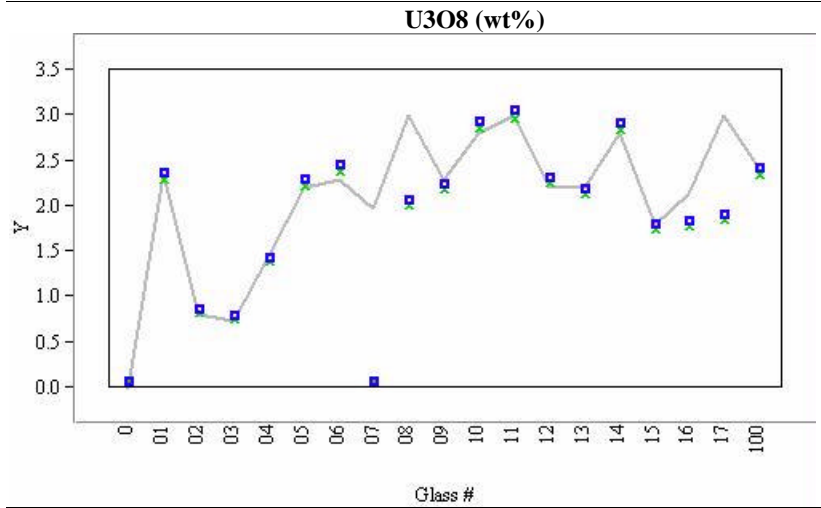


Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)



Y

- Target
- x Measured
- Measure Bias-Corrected (bc)

Exhibit A.7: Comparisons of Measurements versus Target Compositions by Glass #
(Concentrations in weight percents; 0 – Batch 1, 100 – Ustd, and 1-17 PHA Glasses)

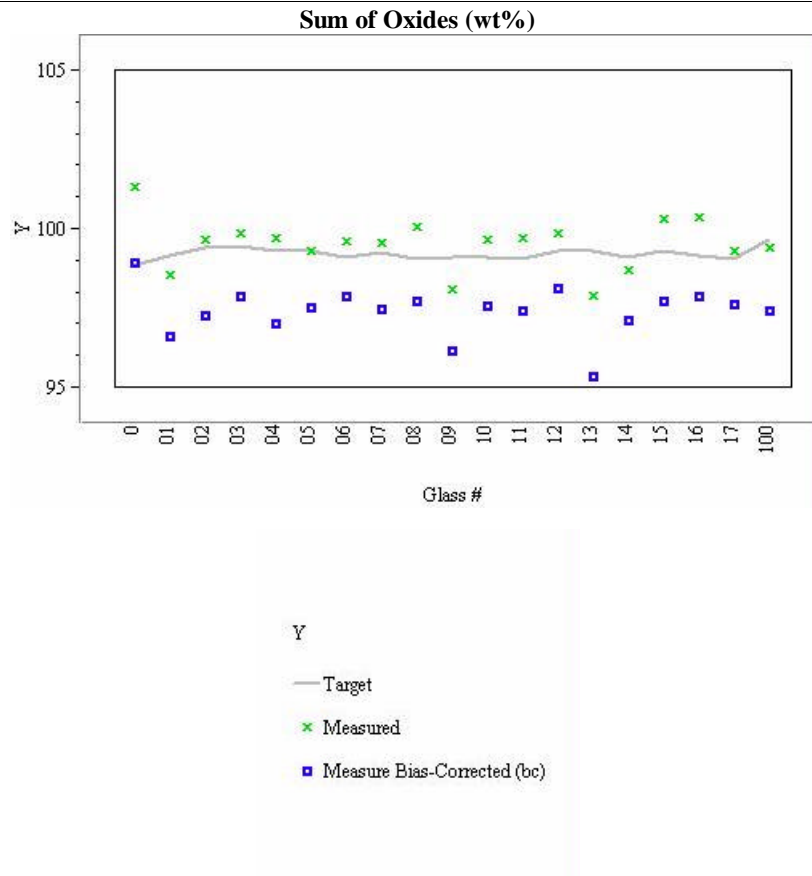
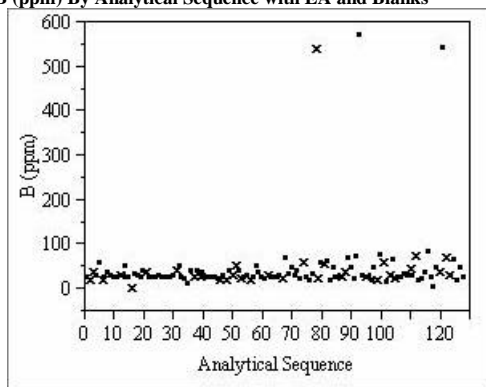
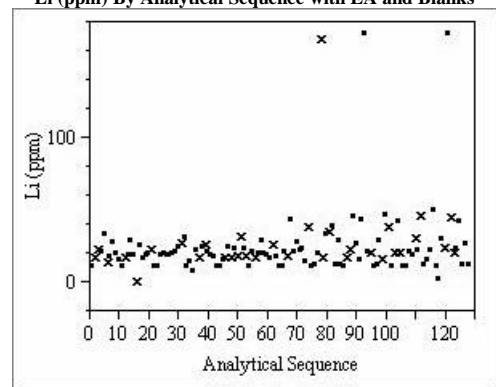


Exhibit A.8: Plots of the Leachate Concentrations in Analytical Sequence by Element

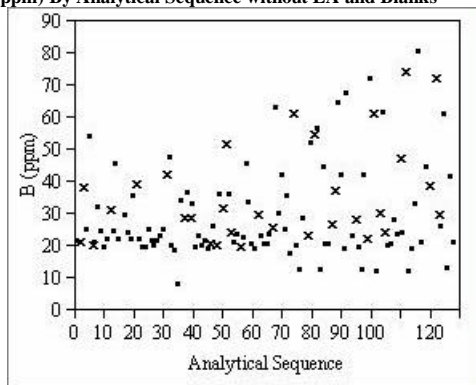
B (ppm) By Analytical Sequence with EA and Blanks



Li (ppm) By Analytical Sequence with EA and Blanks



B (ppm) By Analytical Sequence without EA and Blanks



Li (ppm) By Analytical Sequence without EA and Blanks

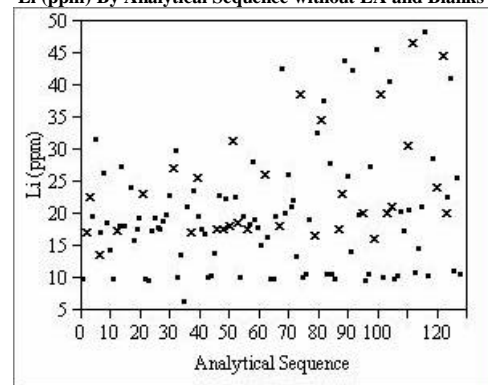


Exhibit A.8: Plots of the Leachate Concentrations in Analytical Sequence by Element

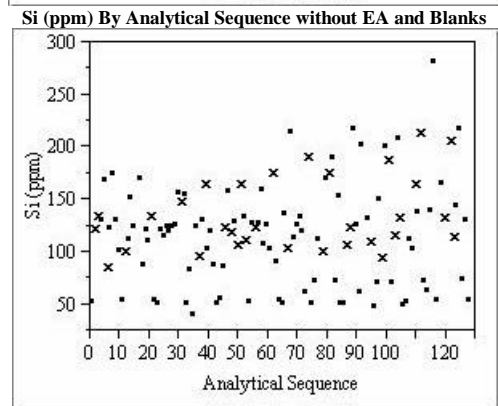
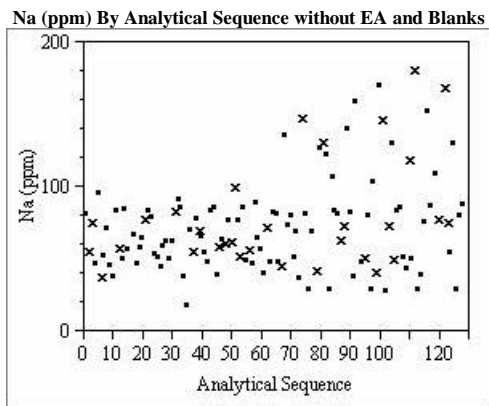
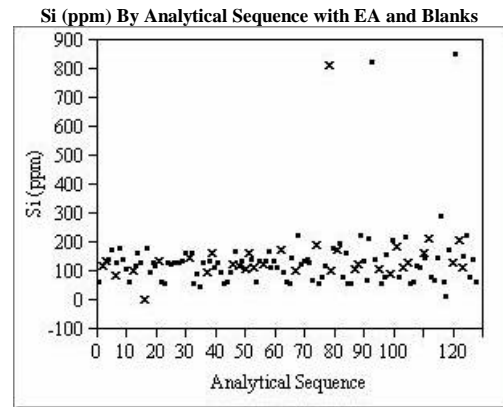
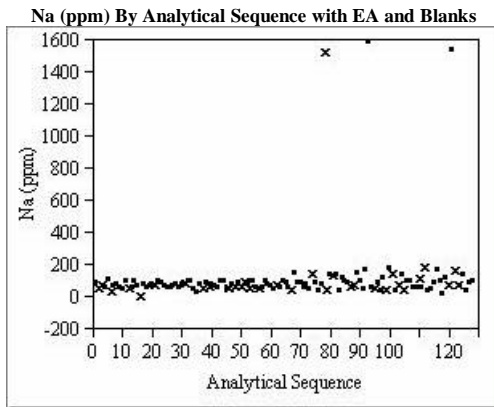


Exhibit A.9: Plots of the Leachate Concentrations by Glass ID by Element

B (ppm) By Sample ID

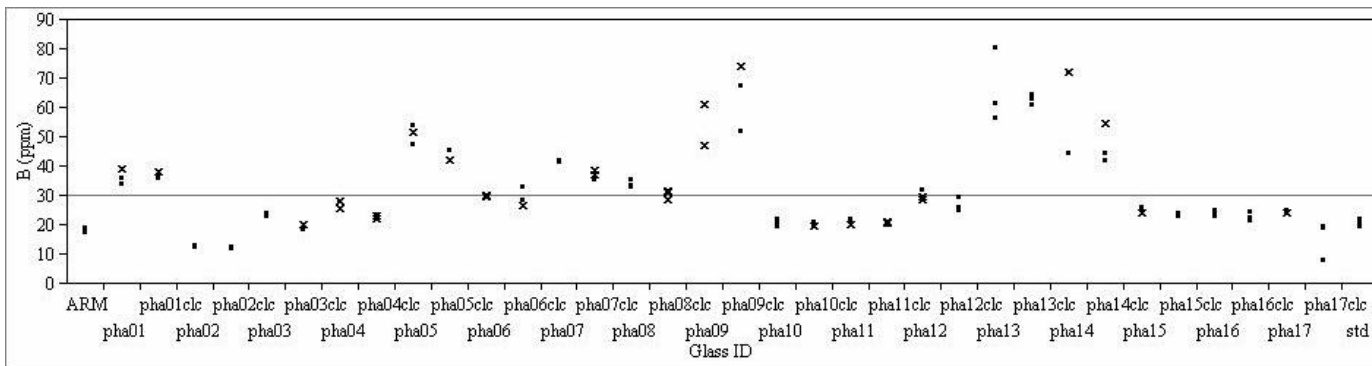
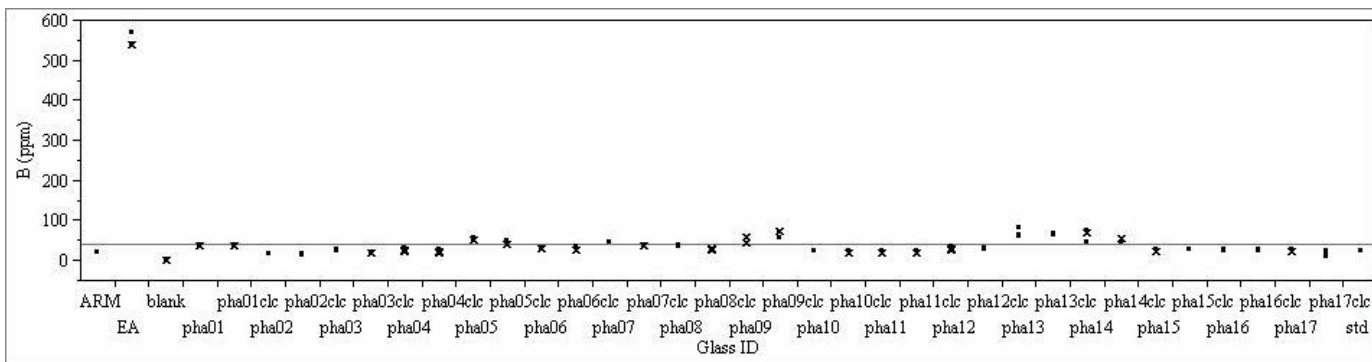


Exhibit A.9: Plots of the Leachate Concentrations by Glass ID by Element (continued)

Li (ppm) By Sample ID

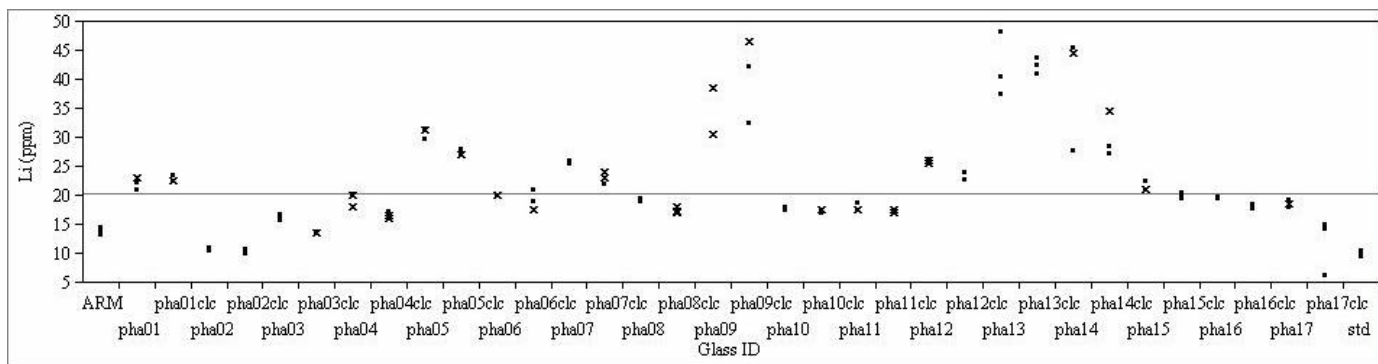
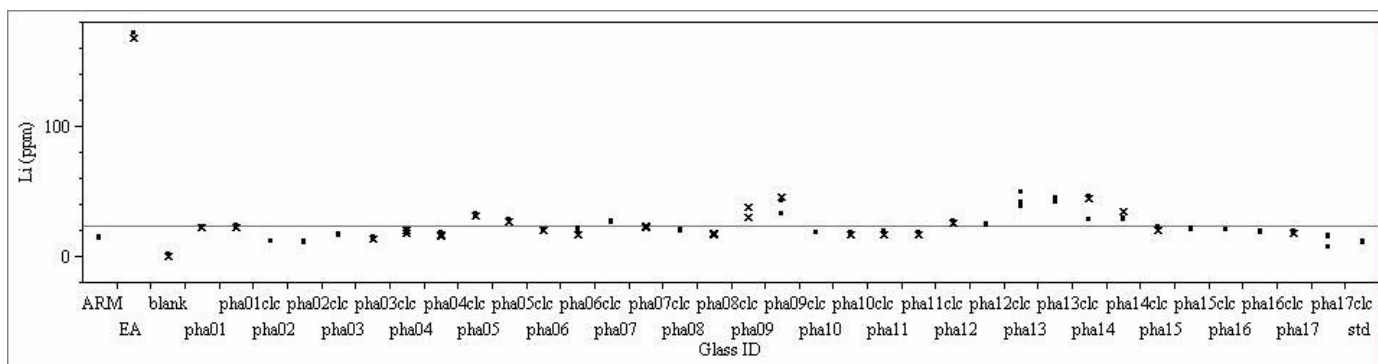


Exhibit A.9: Plots of the Leachate Concentrations by Glass ID by Element (continued)

Na (ppm) By Sample ID

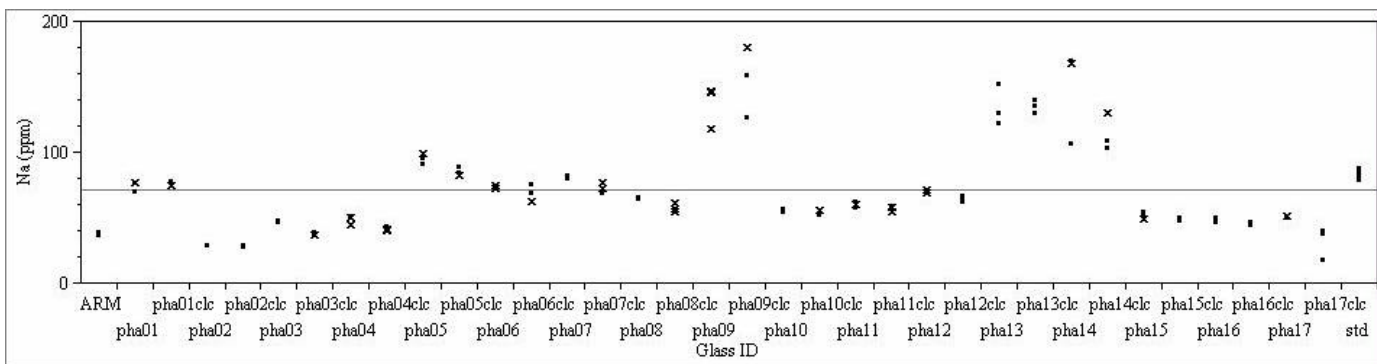
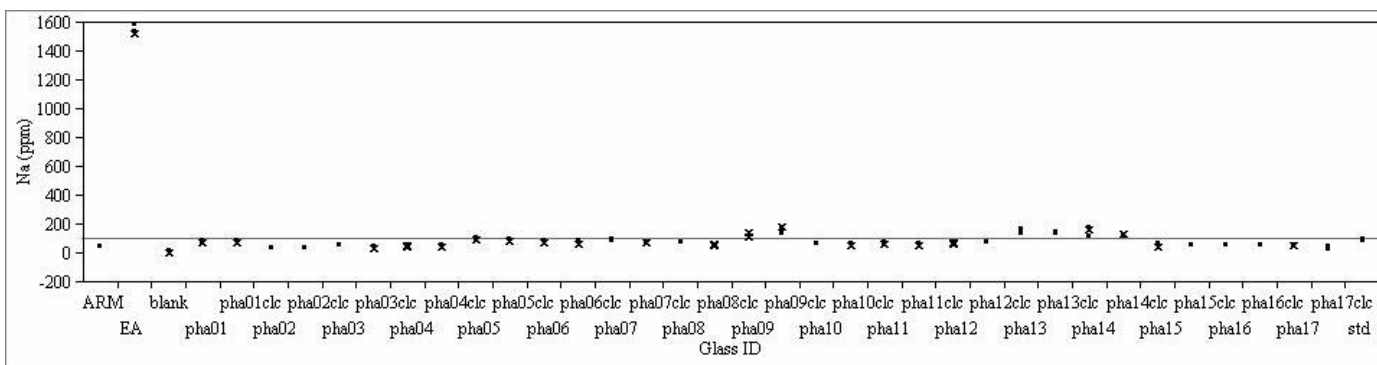


Exhibit A.9: Plots of the Leachate Concentrations by Glass ID by Element (continued)

Si (ppm) By Sample ID

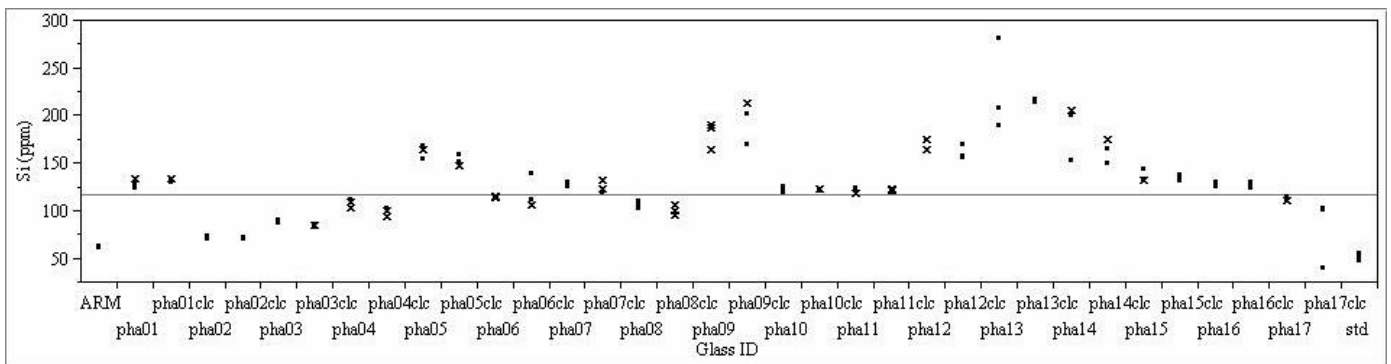
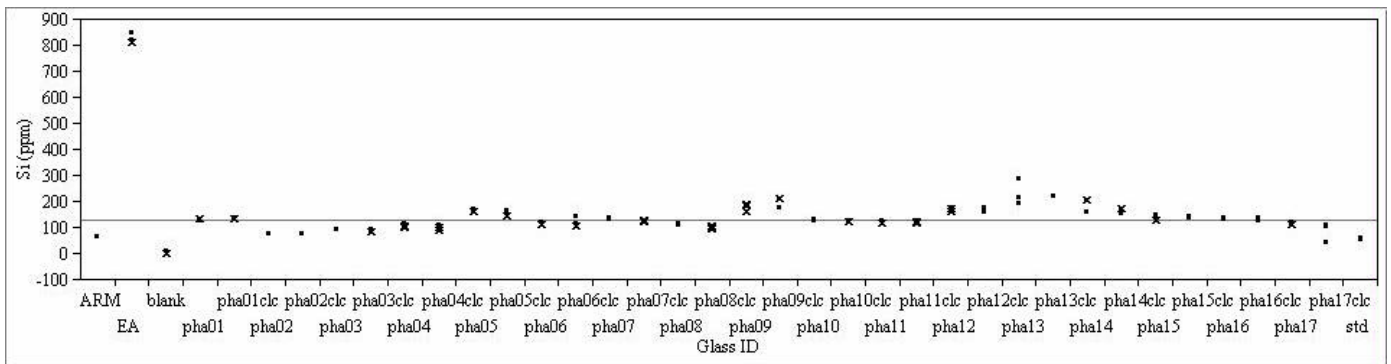
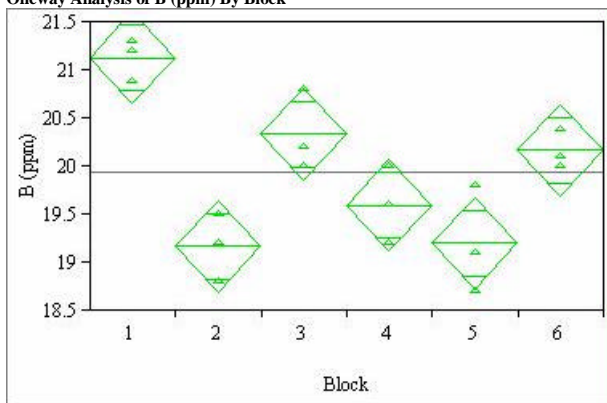


Exhibit A.10: Plots of the Multi-Element Solution Standard by Element

Oneway Analysis of B (ppm) By Block



Oneway Anova

Summary of Fit

Rsquare	0.835581
Adj Rsquare	0.767073
Root Mean Square Error	0.377124
Mean of Response	19.93333
Observations (or Sum Wgts)	18

Analysis of Variance

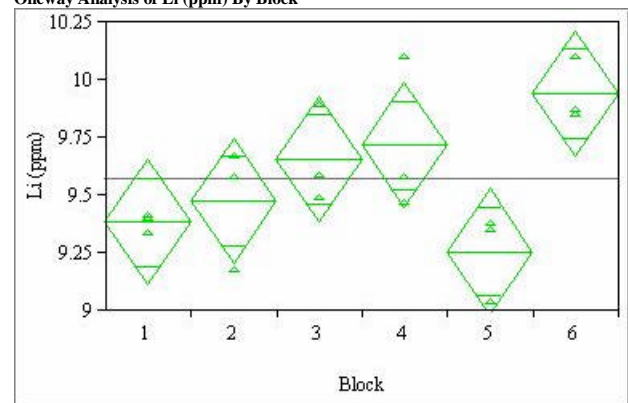
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	5	8.673333	1.73467	12.1969	0.0002
Error	12	1.706667	0.14222		
C. Total	17	10.380000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	21.1333	0.21773	20.659	21.608
2	3	19.1667	0.21773	18.692	19.641
3	3	20.3333	0.21773	19.859	20.808
4	3	19.6000	0.21773	19.126	20.074
5	3	19.2000	0.21773	18.726	19.674
6	3	20.1667	0.21773	19.692	20.641

Std Error uses a pooled estimate of error variance

Oneway Analysis of Li (ppm) By Block



Oneway Anova

Summary of Fit

Rsquare	0.621723
Adj Rsquare	0.464108
Root Mean Square Error	0.21632
Mean of Response	9.571667
Observations (or Sum Wgts)	18

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	5	0.9229167	0.184583	3.9446	0.0239
Error	12	0.5615333	0.046794		
C. Total	17	1.4844500			

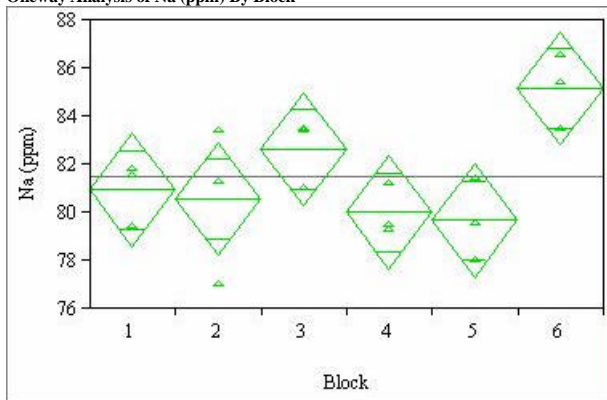
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	9.38333	0.12489	9.1112	9.655
2	3	9.47667	0.12489	9.2045	9.749
3	3	9.65667	0.12489	9.3845	9.929
4	3	9.71667	0.12489	9.4445	9.989
5	3	9.25667	0.12489	8.9845	9.529
6	3	9.94000	0.12489	9.6679	10.212

Std Error uses a pooled estimate of error variance

Exhibit A.10: Plots of the Multi-Element Solution Standard by Element (continued)

Oneway Analysis of Na (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0.607738
Adj Rsquare	0.444295
Root Mean Square Error	1.863986
Mean of Response	81.49444
Observations (or Sum Wgts)	18

Analysis of Variance

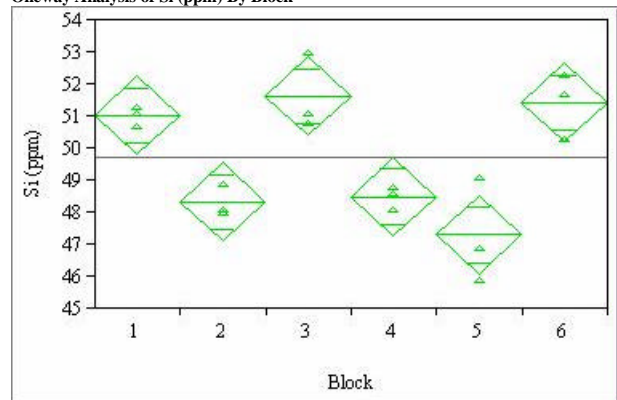
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	5	64.59611	12.9192	3.7184	0.0289
Error	12	41.69333	3.4744		
C. Total	17	106.28944			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	80.9333	1.0762	78.589	83.278
2	3	80.5667	1.0762	78.222	82.911
3	3	82.6333	1.0762	80.289	84.978
4	3	80.0000	1.0762	77.655	82.345
5	3	79.6667	1.0762	77.322	82.011
6	3	85.1667	1.0762	82.822	87.511

Std Error uses a pooled estimate of error variance

Oneway Analysis of Si (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0.824297
Adj Rsquare	0.751087
Root Mean Square Error	0.968102
Mean of Response	49.70556
Observations (or Sum Wgts)	18

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	5	52.762778	10.5526	11.2594	0.0003
Error	12	11.246667	0.9372		
C. Total	17	64.009444			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	51.0333	0.55893	49.816	52.251
2	3	48.3333	0.55893	47.116	49.551
3	3	51.6333	0.55893	50.416	52.851
4	3	48.5000	0.55893	47.282	49.718
5	3	47.3000	0.55893	46.082	48.518
6	3	51.4333	0.55893	50.216	52.651

Std Error uses a pooled estimate of error variance

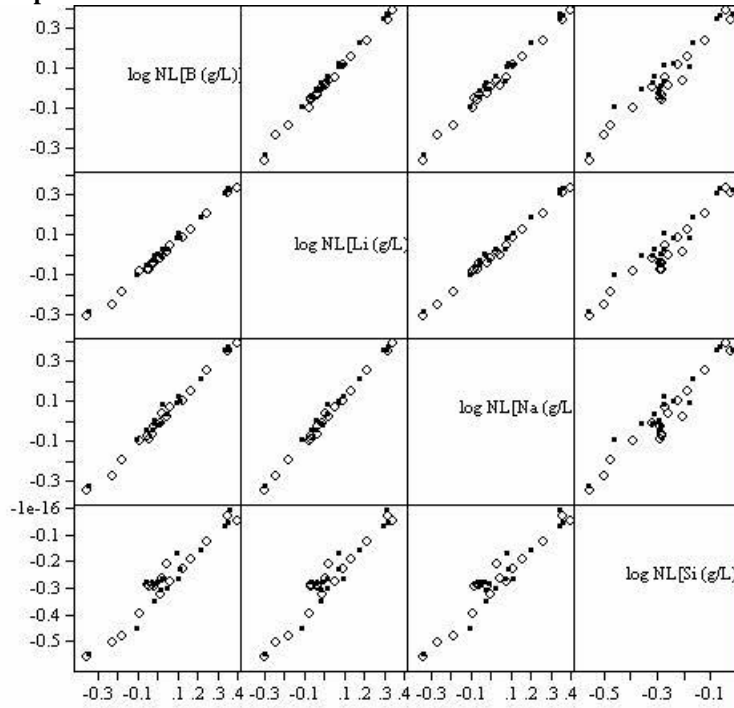
**Exhibit A.11: Correlations and Scatter Plots of the Normalized PCT's
 before Screening for Solution Weight Loss Problems**

Measured Compositions – Linear Correlations & Scatter Plots

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9968	0.9943	0.9669
log NL[Li (g/L)]	0.9968	1.0000	0.9967	0.9619
log NL[Na (g/L)]	0.9943	0.9967	1.0000	0.9600
log NL[Si (g/L)]	0.9669	0.9619	0.9600	1.0000

Scatterplot Matrix



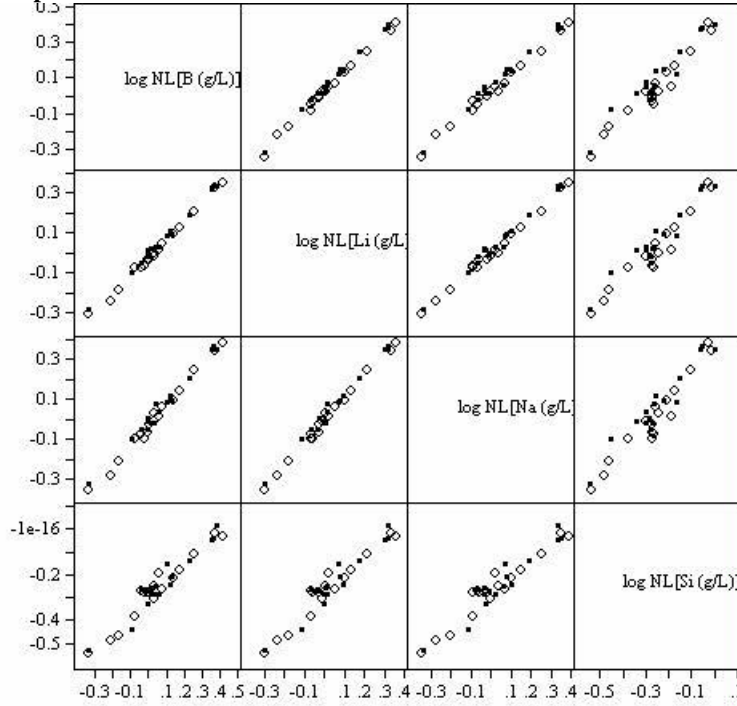
**Exhibit A.11: Correlations and Scatter Plots of the Normalized PCT's
before Screening for Solution Weight Loss Problems** *(continued)*

Measured Bias-Corrected Compositions – Linear Correlations & Scatter Plots

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9970	0.9937	0.9667
log NL[Li (g/L)]	0.9970	1.0000	0.9955	0.9623
log NL[Na (g/L)]	0.9937	0.9955	1.0000	0.9608
log NL[Si (g/L)]	0.9667	0.9623	0.9608	1.0000

Scatterplot Matrix



**Exhibit A.11: Correlations and Scatter Plots of the Normalized PCT's
 before Screening for Solution Weight Loss Problems (continued)**

Target Compositions – Linear Correlations & Scatter Plots

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9883	0.9952	0.9558
log NL[Li (g/L)]	0.9883	1.0000	0.9921	0.9577
log NL[Na (g/L)]	0.9952	0.9921	1.0000	0.9486
log NL[Si (g/L)]	0.9558	0.9577	0.9486	1.0000

Scatterplot Matrix

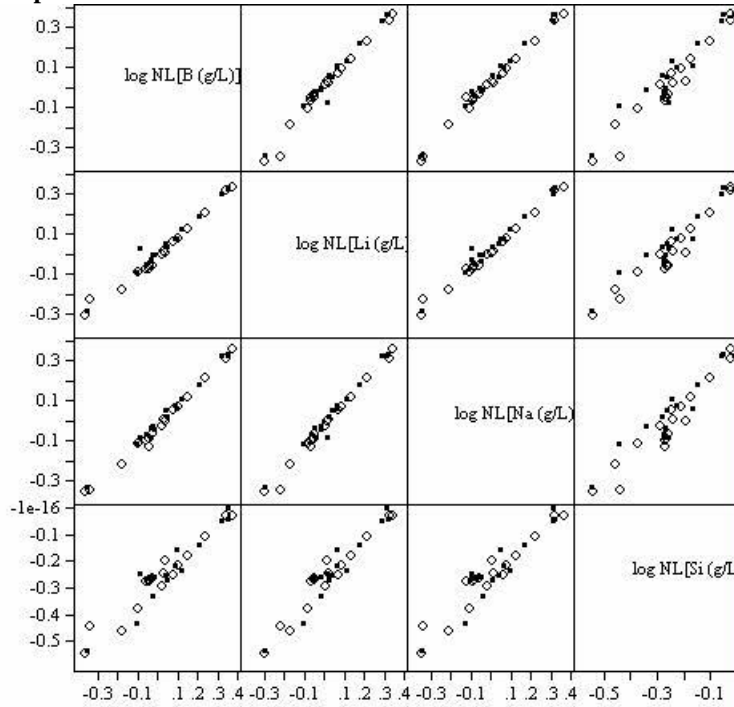


Exhibit A.12: Correlations and Scatter Plots of the Normalized PCTs after Screening for Solution Weight Loss Problems

Measured Compositions

Correlations of Screened PCTs

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9958	0.9923	0.9655
log NL[Li (g/L)]	0.9958	1.0000	0.9961	0.9575
log NL[Na (g/L)]	0.9923	0.9961	1.0000	0.9545
log NL[Si (g/L)]	0.9655	0.9575	0.9545	1.0000

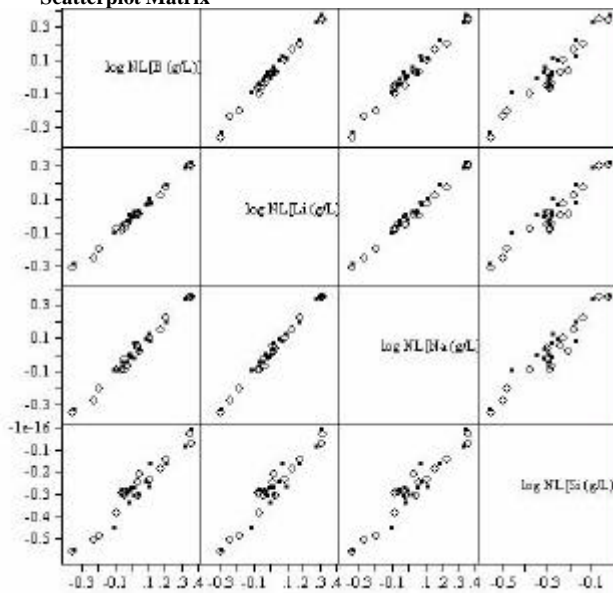
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Correlations of Valid PCTs

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9968	0.9947	0.9685
log NL[Li (g/L)]	0.9968	1.0000	0.9973	0.9645
log NL[Na (g/L)]	0.9947	0.9973	1.0000	0.9617
log NL[Si (g/L)]	0.9685	0.9645	0.9617	1.0000

8 rows not used due to missing values.

Scatterplot Matrix



Scatterplot Matrix

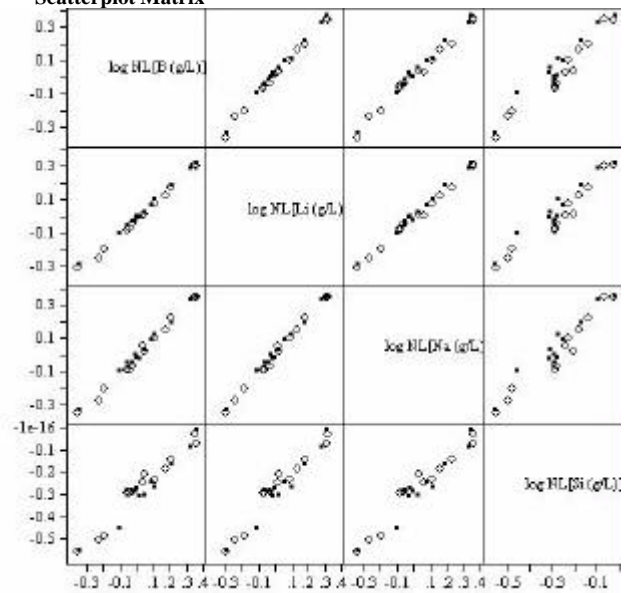


Exhibit A.12: Correlations and Scatter Plots of the Normalized PCTs after Screening for Solution Weight Loss Problems (continued)

Measured Bias-Corrected Compositions

Correlations of Screened PCTs

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9959	0.9917	0.9654
log NL[Li (g/L)]	0.9959	1.0000	0.9949	0.9578
log NL[Na (g/L)]	0.9917	0.9949	1.0000	0.9555
log NL[Si (g/L)]	0.9654	0.9578	0.9555	1.0000

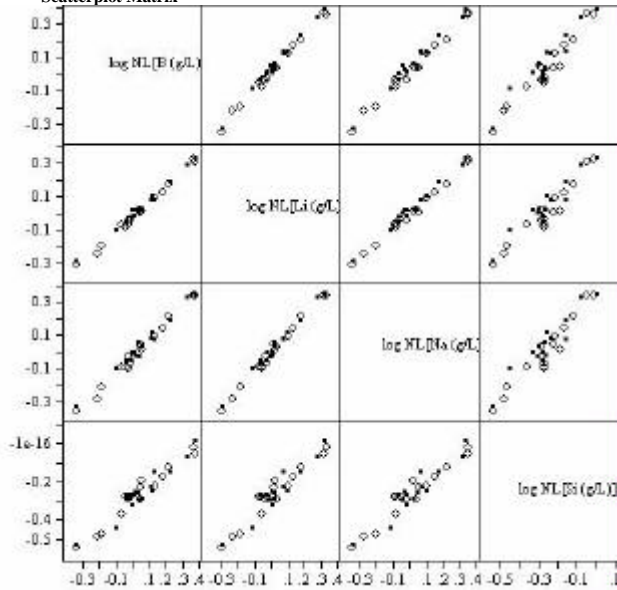
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Correlations of Valid PCTs

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9972	0.9940	0.9686
log NL[Li (g/L)]	0.9972	1.0000	0.9963	0.9657
log NL[Na (g/L)]	0.9940	0.9963	1.0000	0.9629
log NL[Si (g/L)]	0.9686	0.9657	0.9629	1.0000

2 rows not used due to missing values.

Scatterplot Matrix



Scatterplot Matrix

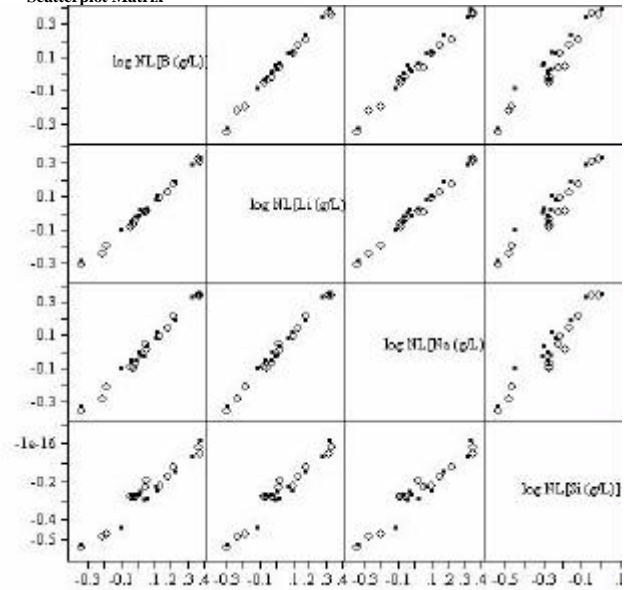


Exhibit A.12: Correlations and Scatter Plots of the Normalized PCTs after Screening for Solution Weight Loss Problems (continued)

Targeted Compositions

Correlations for Screened PCTs

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9873	0.9941	0.9530
log NL[Li (g/L)]	0.9873	1.0000	0.9907	0.9524
log NL[Na (g/L)]	0.9941	0.9907	1.0000	0.9412
log NL[Si (g/L)]	0.9530	0.9524	0.9412	1.0000

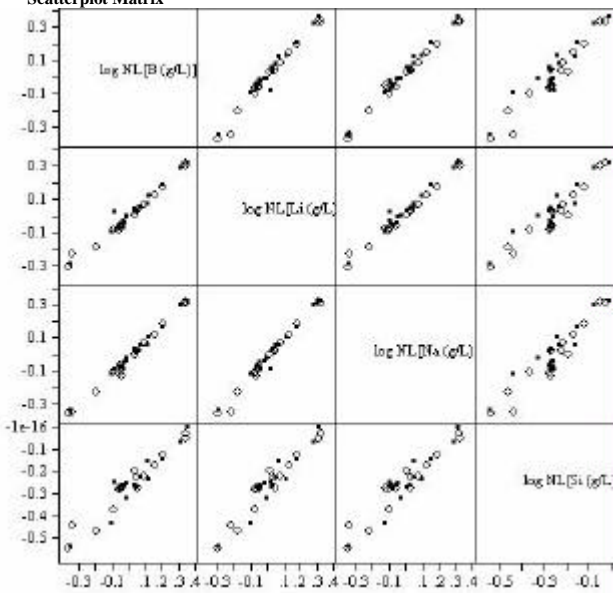
2 rows not used due to missing values.

Correlations for Screened PCTs

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9877	0.9953	0.9578
log NL[Li (g/L)]	0.9877	1.0000	0.9911	0.9608
log NL[Na (g/L)]	0.9953	0.9911	1.0000	0.9503
log NL[Si (g/L)]	0.9578	0.9608	0.9503	1.0000

8 rows not used due to missing values.

Scatterplot Matrix



Scatterplot Matrix

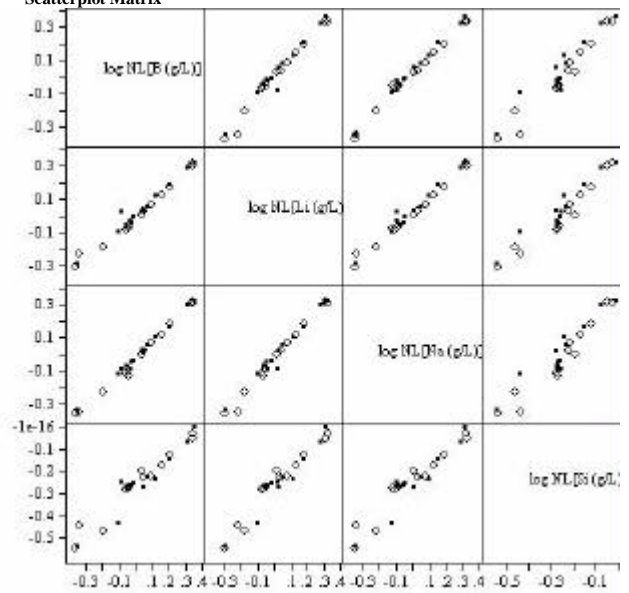
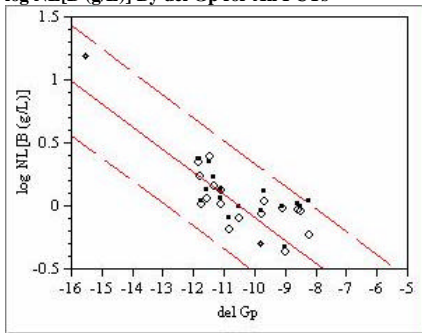
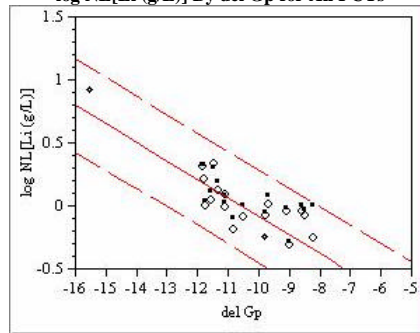


Exhibit A.13: Durability Model Predictions versus PCT Results
Based Upon Measured Glass Compositions

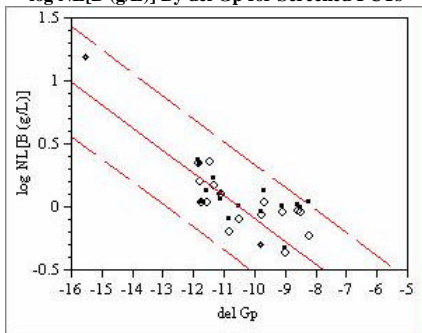
log NL[B (g/L)] By del Gp for All PCTs



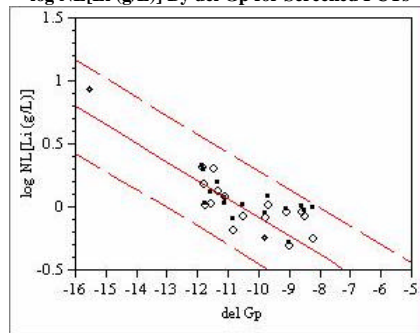
log NL[Li (g/L)] By del Gp for All PCTs



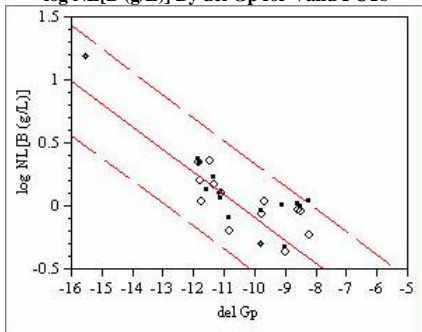
log NL[B (g/L)] By del Gp for Screened PCTs



log NL[Li (g/L)] By del Gp for Screened PCTs



log NL[B (g/L)] By del Gp for Valid PCTs



log NL[Li (g/L)] By del Gp for Valid PCTs

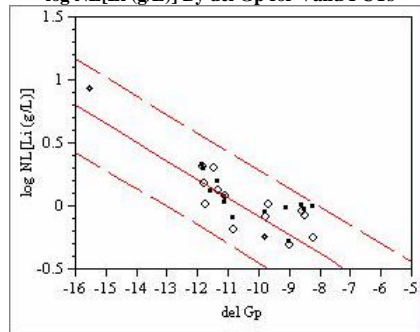
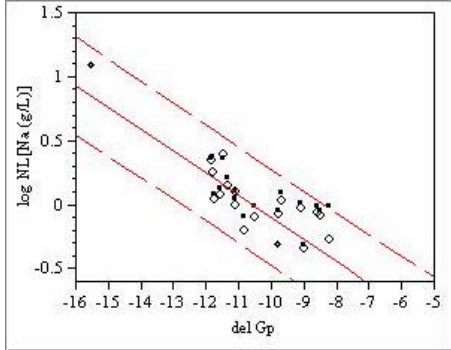
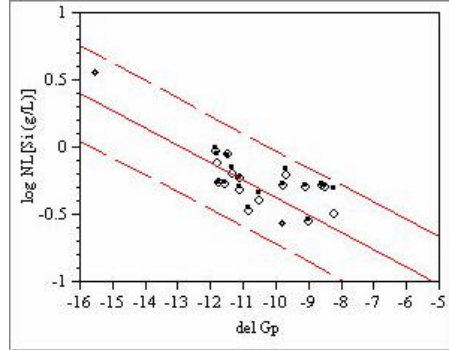


Exhibit A.13: Durability Model Predictions versus PCT Results
Based Upon Measured Glass Compositions (continued)

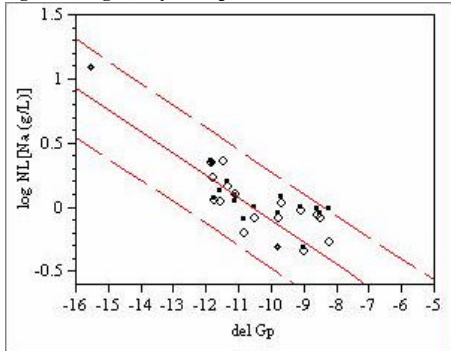
log NL[Na (g/L)] By del Gp for All PCTs



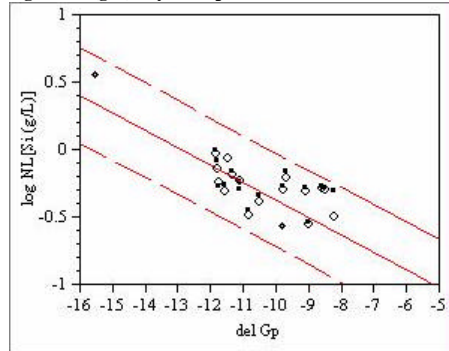
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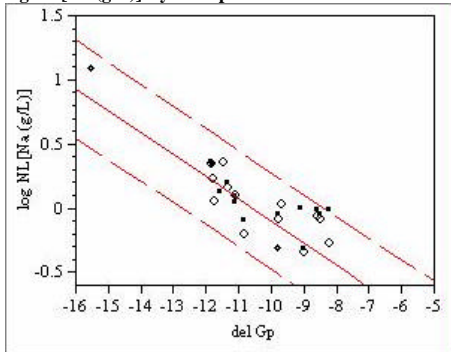
log NL[Na (g/L)] By del Gp for Screened PCTs



log NL[Si (g/L)] By del Gp for Screened PCTs



log NL[Na (g/L)] By del Gp for Valid PCTs



log NL[Si (g/L)] By del Gp for Valid PCTs

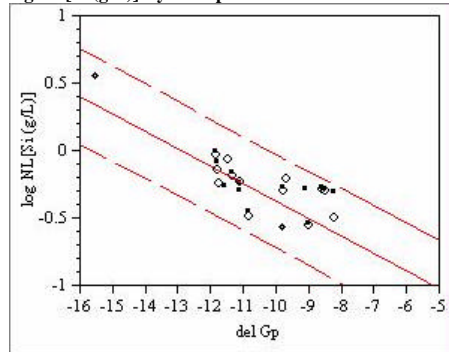
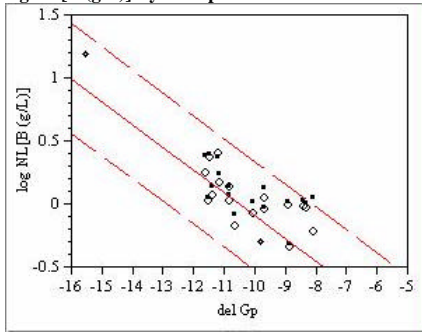
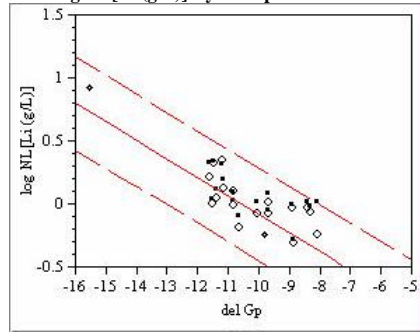


Exhibit A.13: Durability Model Predictions versus PCT Results
Based Upon Bias-Corrected, Measured Glass Compositions*(continued)*

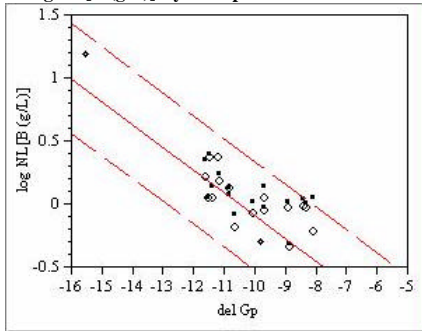
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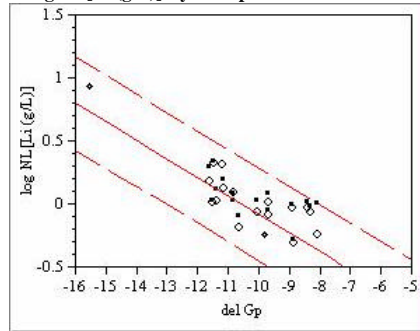
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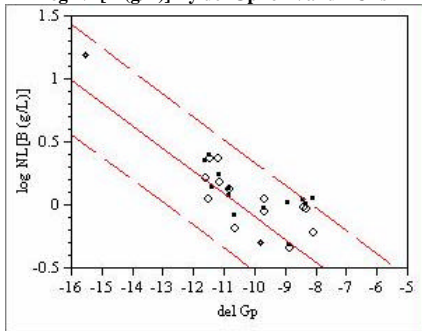
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log NL[Li (g/L)] By del Gp for Screened PCTs



log NL[B (g/L)] By del Gp for Valid PCTs



log NL[Li (g/L)] By del Gp for Valid PCTs

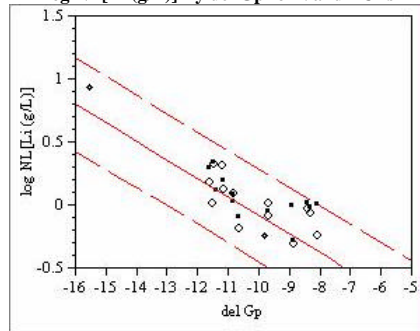
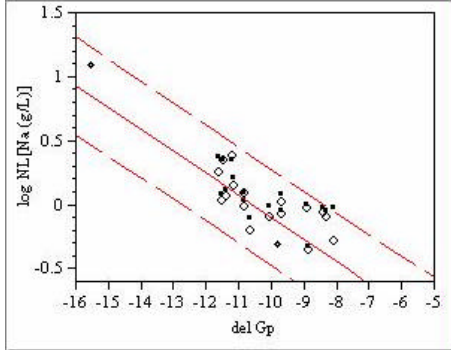
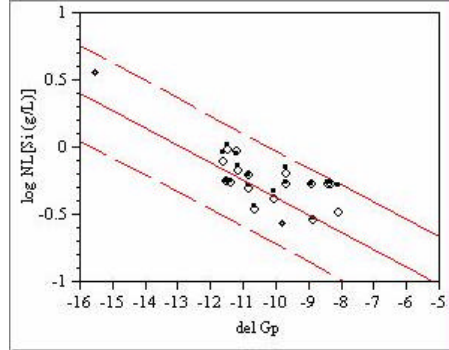


Exhibit A.13: Durability Model Predictions versus PCT Results
Based Upon Bias-Corrected, Measured Glass Compositions (continued)

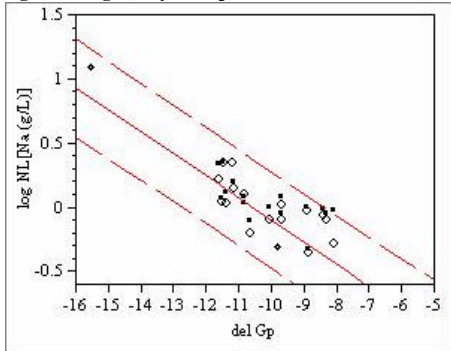
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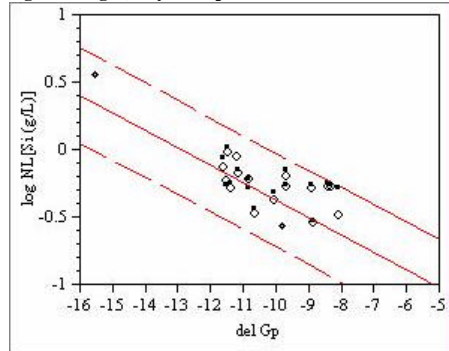
log NL[Si (g/L)] By del Gp for All PCTs



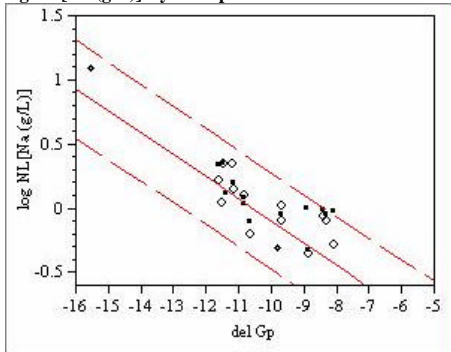
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log NL[Si (g/L)] By del Gp for Screened PCTs



log NL[Na (g/L)] By del Gp for Valid PCTs



log NL[Si (g/L)] By del Gp for Valid PCTs

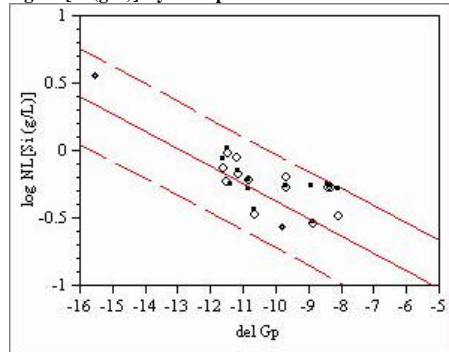


Exhibit A.13: Durability Model Predictions versus PCT Results
Based Upon Targeted Glass Compositions (continued)

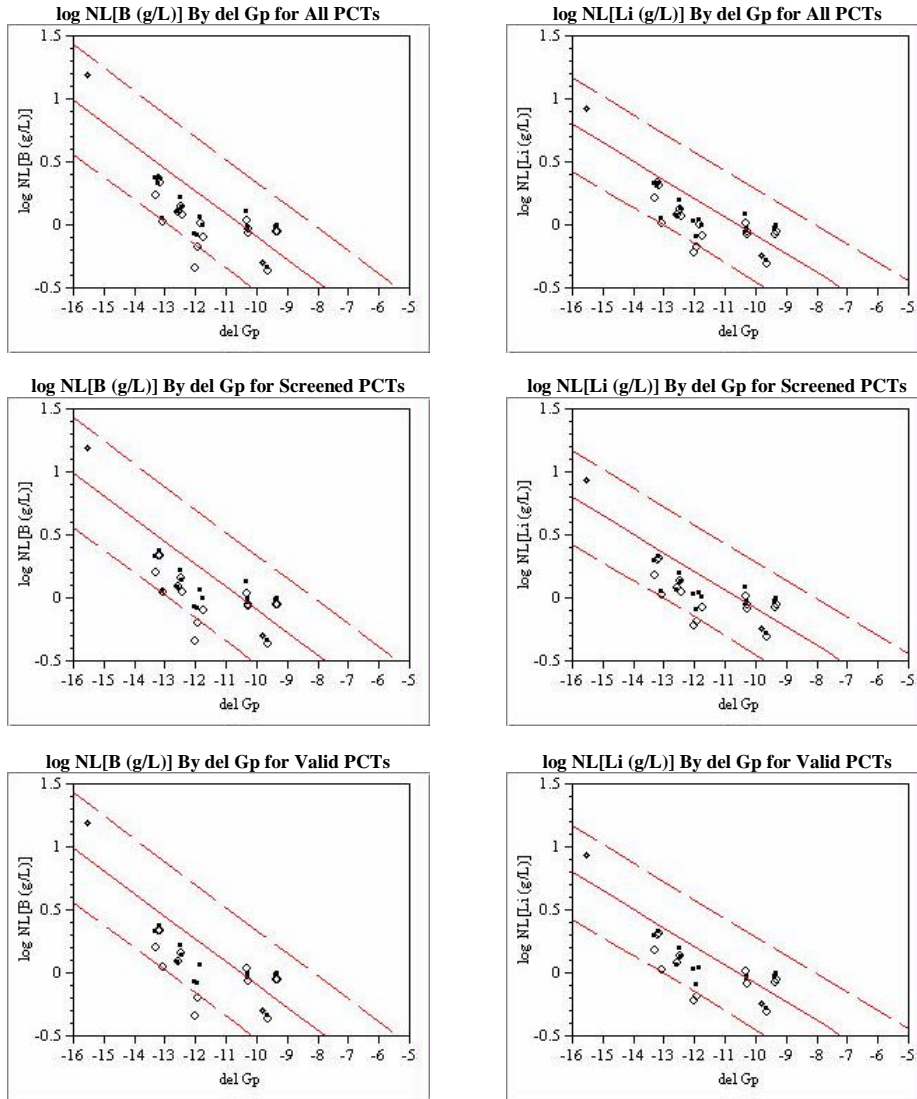
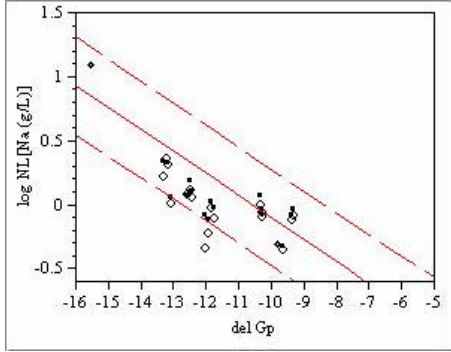
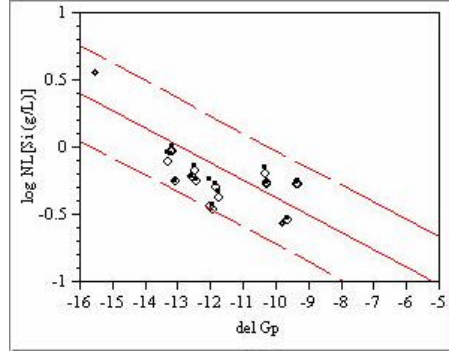


Exhibit A.13: Durability Model Predictions versus PCT Results
Based Upon Targeted Glass Compositions (continued)

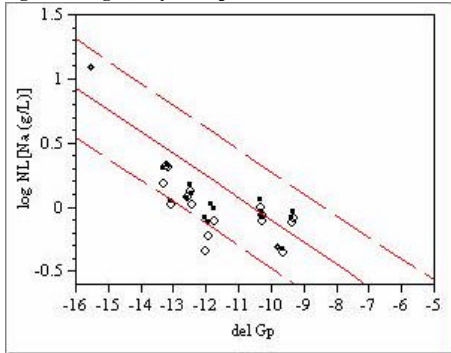
log NL[Na (g/L)] By del Gp for All PCTs



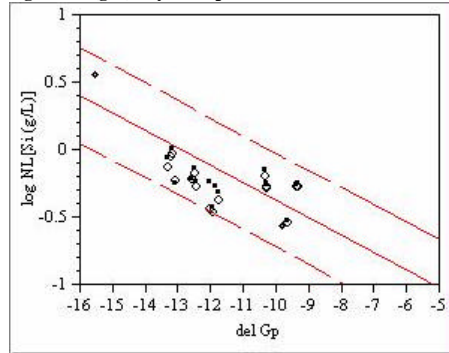
log NL[Si (g/L)] By del Gp for All PCTs



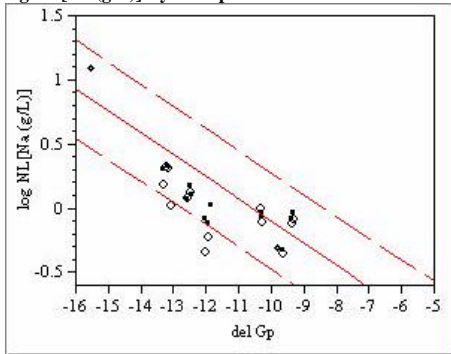
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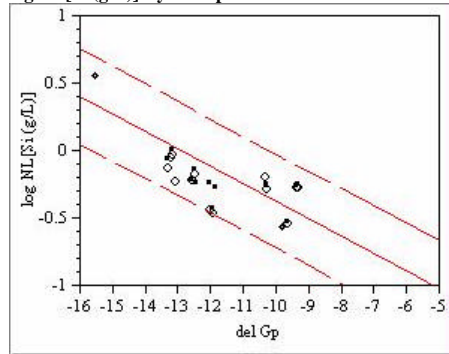
log NL[Si (g/L)] By del Gp for Screened PCTs



log NL[Na (g/L)] By del Gp for Valid PCTs

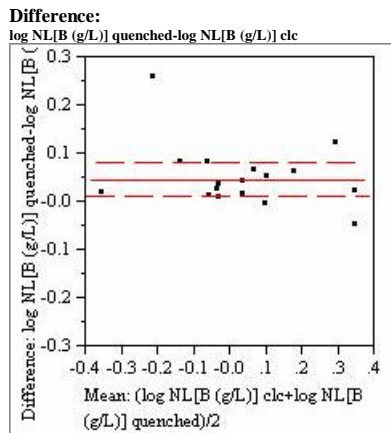


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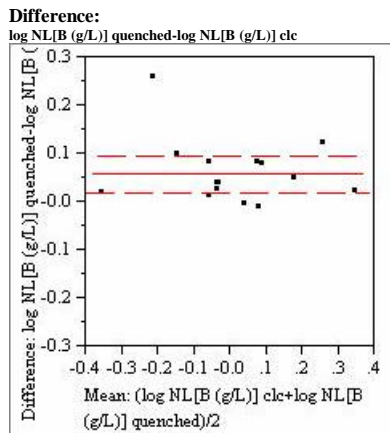
**Exhibit A.14: Paired Comparisons of Quenched versus Centerline Cooled PCTs
Using All of the Leachate Concentrations, Using Only Those Screened For Water Loss Problems, and Using Only Valid PCTs**

All of the PCT Results



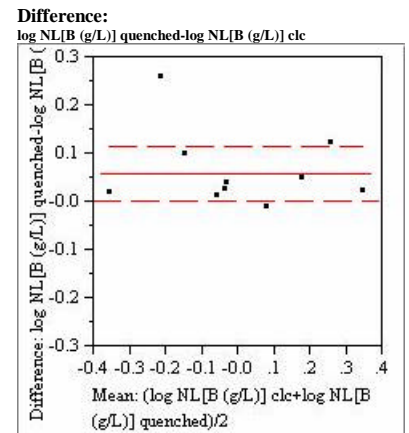
log NL[B (g/L)] quenched	0.05886	t-Ratio	2.883982
log NL[B (g/L)] clc	0.0126	DF	16
Mean Difference	0.04627	Prob > t	0.0108
Std Error	0.01604	Prob > t	0.0054
Upper95%	0.08027	Prob < t	0.9946
Lower95%	0.01226		
N	17		
Correlation	0.94532		

**Only Results for PCTs Passing
Water Loss (i.e. Screened)**



log NL[B (g/L)] quenched	0.03917	t-Ratio	3.280505
log NL[B (g/L)] clc	-0.0176	DF	14
Mean Difference	0.05682	Prob > t	0.0055
Std Error	0.01732	Prob > t	0.0027
Upper95%	0.09397	Prob < t	0.9973
Lower95%	0.01967		
N	15		
Correlation	0.93404		

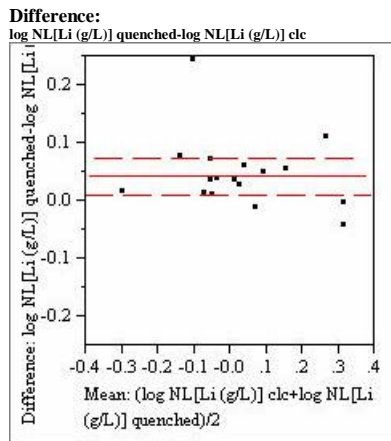
**Valid PCTs Only
(i.e., those with at least 2 of the triplicates OK).**



log NL[B (g/L)] quenched	0.03394	t-Ratio	2.36807
log NL[B (g/L)] clc	-0.0255	DF	9
Mean Difference	0.05942	Prob > t	0.0420
Std Error	0.02509	Prob > t	0.0210
Upper95%	0.11619	Prob < t	0.9790
Lower95%	0.00266		
N	10		
Correlation	0.93846		

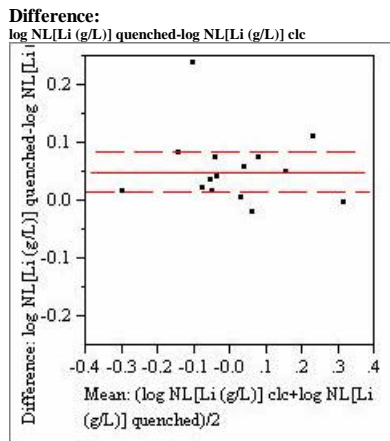
Exhibit A.14: Paired Comparisons of Quenched versus Centerline Cooled PCTs
Using All of the Leachate Concentrations and Using Only Those Screened For Water Loss Problems
(continued)

All of the PCT Results



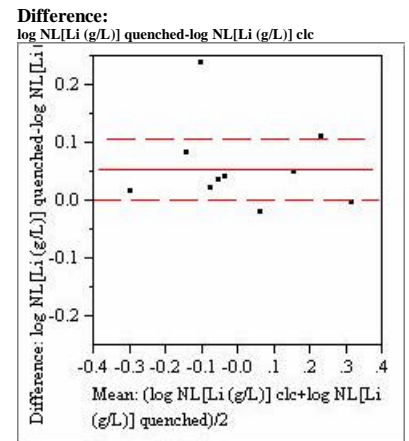
log NL[Li (g/L)] quenched	0.05326	t-Ratio	2.831674
log NL[Li (g/L)] clc	0.01041	DF	16
Mean Difference	0.04285	Prob > t	0.0120
Std Error	0.01513	Prob > t	0.0060
Upper95%	0.07493	Prob < t	0.9940
Lower95%	0.01077		
N	17		
Correlation	0.9337		

Only Results for PCTs Passing Water Loss (i.e. Screened)



log NL[Li (g/L)] quenched	0.03537	t-Ratio	3.086592
log NL[Li (g/L)] clc	-0.0145	DF	14
Mean Difference	0.04984	Prob > t	0.0080
Std Error	0.01615	Prob > t	0.0040
Upper95%	0.08447	Prob < t	0.9960
Lower95%	0.01521		
N	15		
Correlation	0.91985		

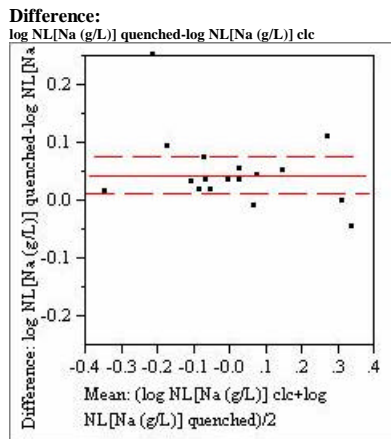
Valid PCTs Only (i.e., those with at least 2 of the triplicates OK).



log NL[Li (g/L)] quenched	0.03522	t-Ratio	2.268857
log NL[Li (g/L)] clc	-0.0182	DF	9
Mean Difference	0.05347	Prob > t	0.0495
Std Error	0.02356	Prob > t	0.0247
Upper95%	0.10677	Prob < t	0.9753
Lower95%	0.00016		
N	10		
Correlation	0.92417		

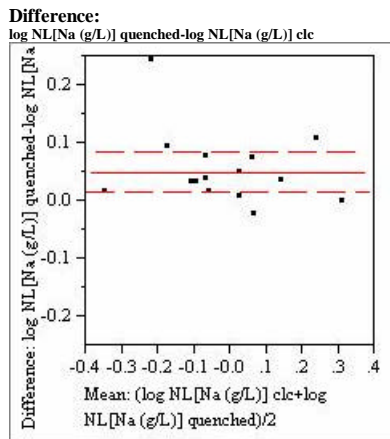
Exhibit A.14: Paired Comparisons of Quenched versus Centerline Cooled PCTs
Using All of the Leachate Concentrations and Using Only Those Screened For Water Loss Problems
(continued)

All of the PCT Results



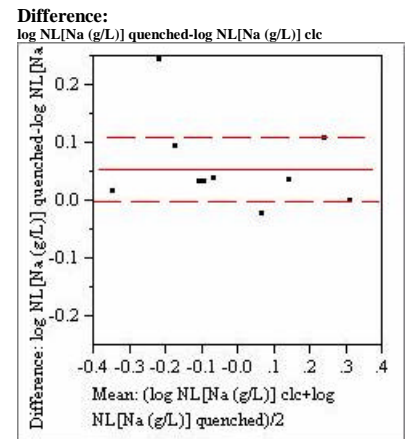
log NL[Na (g/L)] quenched	0.03236	t-Ratio	2.85998
log NL[Na (g/L)] clc	-0.012	DF	16
Mean Difference	0.04441	Prob > t	0.0113
Std Error	0.01553	Prob > t	0.0057
Upper95%	0.07732	Prob < t	0.9943
Lower95%	0.01149		
N	17		
Correlation	0.94867		

Only Results for PCTs Passing Water Loss (i.e. Screened)



log NL[Na (g/L)] quenched	0.01007	t-Ratio	3.031363
log NL[Na (g/L)] clc	-0.0399	DF	14
Mean Difference	0.04994	Prob > t	0.0090
Std Error	0.01647	Prob > t	0.0045
Upper95%	0.08527	Prob < t	0.9955
Lower95%	0.01461		
N	15		
Correlation	0.93724		

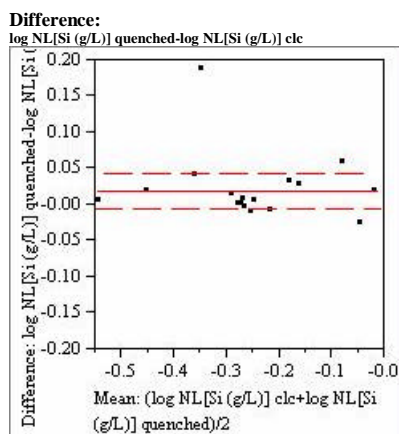
Valid PCTs Only (i.e., those with at least 2 of the triplicates OK).



log NL[Na (g/L)] quenched	0.00474	t-Ratio	2.259991
log NL[Na (g/L)] clc	-0.0497	DF	9
Mean Difference	0.05446	Prob > t	0.0502
Std Error	0.0241	Prob > t	0.0251
Upper95%	0.10897	Prob < t	0.9749
Lower95%	-5.3e-5		
N	10		
Correlation	0.94069		

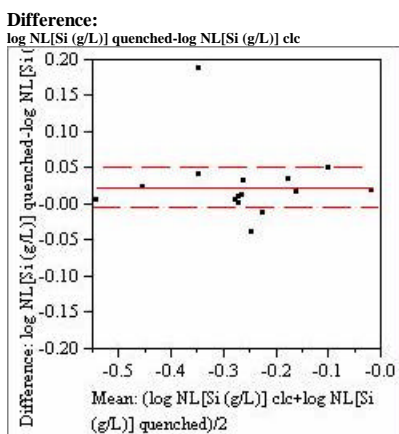
Exhibit A.14: Paired Comparisons of Quenched versus Centerline Cooled PCTs
Using All of the Leachate Concentrations and Using Only Those Screened For Water Loss Problems
(continued)

All of the PCT Results



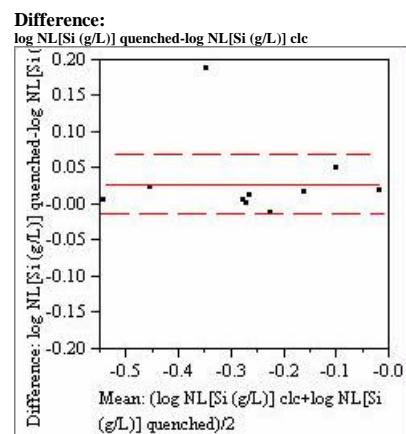
log NL[Si (g/L)] quenched	-0.2406	t-Ratio	1.712305
log NL[Si (g/L)] clc	-0.2604	DF	16
Mean Difference	0.01972	Prob > t	0.1061
Std Error	0.01152	Prob > t	0.0531
Upper95%	0.04413	Prob < t	0.9469
Lower95%	-0.0047		
N	17		
Correlation	0.94088		

Only Results for PCTs Passing Water Loss (i.e. Screened)



log NL[Si (g/L)] quenched	-0.2524	t-Ratio	1.837579
log NL[Si (g/L)] clc	-0.276	DF	14
Mean Difference	0.02361	Prob > t	0.0874
Std Error	0.01285	Prob > t	0.0437
Upper95%	0.05116	Prob < t	0.9563
Lower95%	-0.0039		
N	15		
Correlation	0.92956		

Valid PCTs Only (i.e., those with at least 2 of the triplicates OK).



log NL[Si (g/L)] quenched	-0.2519	t-Ratio	1.572331
log NL[Si (g/L)] clc	-0.2805	DF	9
Mean Difference	0.02855	Prob > t	0.1503
Std Error	0.01816	Prob > t	0.0752
Upper95%	0.06963	Prob < t	0.9248
Lower95%	-0.0125		
N	10		
Correlation	0.9353		

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Attachment I.

Edwards, T. B., "An Analytical Plan for the SRTC Mobile Laboratory to Follow in Measuring the Chemical Composition of the PHA Glasses of the FY01 Study (U), SRT-SCS-2001-00001, January 4, 2001.

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WESTINGHOUSE SAVANNAH RIVER COMPANY
INTEROFFICE MEMORANDUM

SRT-SCS-2001-00001

January 4, 2001

To: J. R. Harbour, 773-43A (wi)

cc: R. A. Baker, 773-42A (es) S. L. Marra, 704-T (wi)
D. R. Best, 773-41A (wo) R. C. Tuckfield, 773-42A (wi)
K. G. Brown, 773-43A (wi) R. J. Workman, 773-A (wi)
E. W. Holtzscheiter, 773-A (es)

From: T. B. Edwards, 773-42A (5-5148)
Statistical Consulting Section

wi - with sample identifiers
wo - without sample identifiers
es - executive summary only

R. A. Baker, Technical Reviewer

Date

R. C. Tuckfield, Manager
Statistical Consulting Section

Date

**An Analytical Plan for the
SRTC Mobile Laboratory to Follow
in Measuring the Chemical Compositions of the
PHA Glasses of the FY01 Study (U)**

EXECUTIVE SUMMARY

A task technical and quality assurance plan and an analytical plan have been prepared to direct activities associated with the FY01 PHA glass studies for the Salt Processing Program. Seventeen PHA glasses were fabricated as part of this study. The chemical compositions of these 17 glasses are to be determined by the SRTC Mobile Laboratory (SRTC-ML). This memorandum provides an analytical plan for the SRTC-ML to follow in measuring the chemical compositions of these 17 PHA glasses.

INTRODUCTION

A task technical and quality assurance (TT&QA) plan [1] and an analytical plan [2] have been prepared to direct activities associated with the FY01 PHA glass studies for the Salt Processing Program. Seventeen glasses have been fabricated as part of the PHA study [3]. The compositions of these 17 glasses are to be determined by the SRTC Mobile Laboratory (SRTC-ML). This memorandum provides an analytical plan for the SRTC-ML to follow in measuring the compositions of these 17 PHA glasses.

DISCUSSION

Table 1 provides the naming conventions for the 17 samples that are to be used by the SRTC-ML in conducting the compositional analyses and in reporting the measurements.¹

Table 1: Unique Sample ID's for the 17 Glasses

Original ID	Lab ID
pha01	T14
pha02	T11
pha03	T05
pha04	T03
pha05	T01
pha06	T17
pha07	T13
pha08	T10
pha09	T04
pha10	T09
pha11	T02
pha12	T16
pha13	T08
pha14	T06
pha15	T15
pha16	T12
pha17	T07

PREPARATION OF THE SAMPLES

The analytical procedures used by the SRTC-ML to determine cation concentrations for a glass sample include steps for sample preparation and for calibration of the Inductively Coupled Plasma (ICP) – Emission Spectrometer. These procedural steps are of primary concern in the development of this analytical plan.

The primary dissolution methods that are to be used by the SRTC-ML to complete this compositional study are lithium metaborate (LM) and peroxide fusion (pf). A third dissolution method (microwave fusion, mf) is to be used if necessary to assure

¹ Renaming these samples ensures that they will be processed as blind samples. This table is complete only for those on the distribution list with a “wi” following their names.

complete sample dissolution. All three dissolution methods are considered in this analytical plan.

The cation concentrations are to be measured (as weight percents) for the submitted samples prepared using one or more of the dissolution methods for the following elements: aluminum (Al), boron (B), calcium (Ca), chromium (Cr), copper (Cu), iron (Fe), potassium (K), lithium (Li), magnesium (Mg), manganese (Mn), sodium (Na), nickel (Ni), silicon (Si), titanium (Ti), uranium (U), and zirconium (Zr).

Each of the 17 samples submitted to the SRTC-ML is to be prepared twice by each of the dissolution methods utilized, and the prepared samples are to be read twice by Inductively Coupled Plasma – Emission Spectroscopy, with the instrument being calibrated before each of these two readings (for each of the prepared samples). This will lead to 4 measurements for each cation of interest for each of the 17 samples submitted to the lab. Table 2 provides unique identifiers for the 34 preparations for each dissolution method and a random sequencing scheme for conducting the dissolutions.

In Table 2, the sample identifier has been modified with a suffix consisting of a two-letter indicator for the preparation method (LM for lithium metaborate, pf for peroxide fusion, and mw for microwave) and a 1-digit indicator for preparation number.

Table 2: Preparation Blocks

Lithium Metaborate	Peroxide Fusion	Microwave Fusion (if necessary)
T11LM1	T11pf1	T09mf1
T10LM1	T04pf1	T17mf1
T03LM1	T11pf2	T06mf1
T10LM2	T07pf1	T09mf2
T16LM1	T12pf1	T16mf1
T16LM2	T04pf2	T02mf1
T11LM2	T10pf1	T03mf1
T12LM1	T07pf2	T17mf2
T17LM1	T05pf1	T05mf1
T13LM1	T09pf1	T15mf1
T17LM2	T03pf1	T06mf2
T12LM2	T05pf2	T08mf1
T14LM1	T13pf1	T05mf2
T01LM1	T12pf2	T14mf1
T03LM2	T10pf2	T02mf2
T04LM1	T06pf1	T16mf2
T04LM2	T08pf1	T04mf1
T05LM1	T13pf2	T10mf1
T06LM1	T01pf1	T04mf2
T06LM2	T17pf1	T15mf2
T15LM1	T03pf2	T14mf2
T13LM2	T14pf1	T08mf2
T15LM2	T06pf2	T07mf1
T08LM1	T15pf1	T11mf1
T07LM1	T09pf2	T03mf2
T08LM2	T02pf1	T01mf1
T09LM1	T16pf1	T12mf1
T05LM2	T15pf2	T11mf2
T14LM2	T08pf2	T13mf1
T02LM1	T17pf2	T10mf2

T02LM2	T01pf2	T01mf2
T01LM2	T14pf2	T07mf2
T07LM2	T02pf2	T13mf2
T09LM2	T16pf2	T12mf2

MEASUREMENT OF THE SAMPLES WITH THE ICP

The samples prepared by each of the dissolution methods employed are to be analyzed using ICP instrumentation calibrated for the particular preparation method. After the initial set of cation concentration measurements have been completed for a set of samples, the ICP instrumentation is to be recalibrated and a second set of concentration measurements for the appropriate cations determined. Two additional glasses are included in this analytical plan to provide an opportunity for checking the performance of the ICP instrumentation over the course of these analyses and for possible bias-correction of the measurements of the other glasses. One of these glasses is the standard, Batch 1, whose composition is provided in Table 3.

Table 3: Composition of Batch 1 in Weight Percent (wt%)

Oxide	Wt%	Oxide	Wt%
Al ₂ O ₃	4.877	MgO	1.419
B ₂ O ₃	7.777	MnO	1.726
BaO	0.151	Na ₂ O	9.003
CaO	1.220	Nd ₂ O ₃	0.147
Cr ₂ O ₃	0.107	NiO	0.751
Cs ₂ O	0.060	RuO ₂	0.0214
CuO	0.399	SiO ₂	50.22
Fe ₂ O ₃	12.839	TiO ₂	0.677
K ₂ O	3.327	ZrO ₂	0.098
Li ₂ O	4.429		

The second glass that will be used as a standard for these measurements is a uranium glass that is to be provided to the SRTC-ML along with other glass samples.

A randomized plan for measuring cation concentrations in the prepared samples by each dissolution method is provided in Tables 4-6. In these tables, the sample identifiers have been modified by the addition of a one-digit suffix to indicate whether the measurement is to be made during the first or second ICP calibration block for that sample.

Samples of the standards, Batch 1 and the uranium-bearing glasses, which are to be prepared using the appropriate dissolution method, have been added to Tables 4-6. The identifiers for the Batch 1 standard samples begin with the 3-letter designation "std" followed by the 2-letter dissolution indicator, then the 2-digit ICP block number, and finally, a number 1 through 3 for the three replicates of this glass per block. The identifiers for the uranium standard samples begin with the 4-letter designation "ustd" followed by the 2-letter dissolution indicator, then the 2-digit ICP block number, and finally, a number 1 through 2 for the duplicate measurements per block of this glass.

Table 4: ICP Blocks for Samples Prepared Using Lithium Metaborate (LM) Method

1-1	1-2	2-1	2-2
stdLM111	stdLM121	stdLM211	stdLM221
T07LM11	T14LM22	T08LM11	T11LM12
T13LM21	T14LM12	T11LM21	T02LM12
T05LM21	T16LM12	T03LM21	T03LM12
T07LM21	T04LM22	T02LM21	T08LM12
T17LM11	T01LM12	T12LM21	T11LM22
ustdLM111	ustdLM121	ustdLM211	ustdLM221
T14LM11	T05LM22	T10LM21	T15LM12
T06LM11	T13LM12	T10LM11	T12LM22
T01LM21	T07LM12	T08LM21	T10LM22
T14LM21	T07LM22	T12LM11	T08LM22
stdLM112	stdLM122	stdLM212	stdLM222
T06LM21	T17LM12	T15LM11	T15LM22
T04LM11	T16LM22	T02LM11	T02LM22
T05LM11	T06LM22	T15LM21	T12LM12
T16LM11	T01LM22	T03LM11	T03LM22
ustdLM112	ustdLM122	ustdLM212	ustdLM222
T01LM11	T06LM12	T09LM11	T09LM12
T16LM21	T17LM22	T09LM21	T09LM22
T13LM11	T04LM12	T11LM11	T10LM12
T17LM21	T05LM12	stdLM213	stdLM223
T04LM21	T13LM22		
stdLM113	stdLM123		

Table 5: ICP Blocks for Samples Prepared Using Peroxide Fusion (pf) Method

1-1	1-2	2-1	2-2
stdpf111	stdpf121	stdpf211	stdpf221
T05pf11	T17pf12	T08pf21	T02pf22
T11pf21	T06pf12	T15pf11	T02pf12
T17pf21	T10pf22	T03pf11	T07pf22
T13pf11	T05pf12	T07pf21	T12pf12
T16pf21	T09pf12	T02pf21	T08pf12
ustdpf111	ustdpf121	ustdpf211	ustdpf221
T17pf11	T09pf22	T04pf11	T15pf22
T01pf21	T17pf22	T14pf11	T08pf22
T05pf21	T11pf22	T02pf11	T12pf22
T09pf21	T05pf22	T15pf21	T04pf22
stdpf112	stdpf122	stdpf212	stdpf222
T06pf21	T10pf12	T07pf11	T03pf12
T16pf11	T16pf12	T12pf21	T14pf22
T13pf21	T01pf22	T04pf21	T07pf12
T11pf11	T06pf22	T08pf11	T14pf12
ustdpf112	ustdpf122	ustdpf212	ustdpf222
T10pf11	T01pf12	T03pf21	T15pf12
T10pf21	T13pf22	T12pf11	T04pf12
T06pf11	T16pf22	T14pf21	T03pf22
T01pf11	T13pf12	stdpf213	stdpf223
T09pf11	T11pf12		
stdpf113	stdpf123		

**Table 6: ICP Blocks for Samples Prepared Using Microwave Fusion (mf) Method
(if needed)**

1-1	1-2	2-1	2-2
stdmf111	stdmf121	stdmf211	stdmf221
T05mf11	T02mf22	T14mf11	T12mf22
T17mf11	T01mf12	T15mf11	T10mf12
T03mf11	T01mf22	T11mf21	T06mf12
T02mf11	T04mf12	T13mf21	T12mf12
T01mf11	T05mf12	T06mf21	T13mf12

ustdmf111	ustdmf121	ustdmf211	ustdmf221
T09mf21	T08mf22	T10mf21	T14mf22
T04mf11	T17mf12	T16mf11	T15mf22
T17mf21	T05mf22	T15mf21	T14mf12
T02mf21	T03mf12	T16mf21	T11mf22
stdmf112	stdmf122	stdmf212	stdmf222
T08mf11	T09mf22	T12mf11	T11mf12
T05mf21	T04mf22	T11mf11	T16mf12
T07mf21	T07mf12	T10mf11	T10mf22
T01mf21	T08mf12	T12mf21	T16mf22
ustdmf112	ustdmf122	ustdmf212	ustdmf222
T09mf11	T17mf22	T06mf11	T06mf22
T08mf21	T03mf22	T14mf21	T13mf22
T03mf21	T07mf22	T13mf11	T15mf12
T07mf11	T02mf12	stdmf213	stdmf223
T04mf21	T09mf12		
stdmf113	stdmf123		

CONCLUDING COMMENTS

This memorandum provides an analytical plan for the SRTC-ML to follow in measuring the chemical compositions of 17 PHA glasses. The analytical plan identifies several ICP calibration blocks in Tables 4-6 as well as preparation blocks in Table 2. The sequencing of the activities associated with each of the steps in the analytical procedures has been randomized. The size of each of the blocks is such that it should be completed in a single work shift.

If for some reason the measurements are not conducted in the sequences presented in this memorandum, the actual order used should be recorded along with any explanative comments.

The analytical plan provided in the preceding tables should be modified by the personnel of SRTC-ML to include any calibration check standards and/or other standards that are part of their routine operating procedures.

REFERENCES

- [1] Harbour, J. R. and T. B. Edwards, "Task Technical & QA Plan: Impact of Cooling Rates on the Durabilities of CST and PHA Glasses", WSRC-RP-2000-00996, Revision 0, December 2000.
- [2] Harbour, J. R. and T. B. Edwards, "Analytical Study Plan: Impact of Cooling Rates on the Durabilities of CST and PHA Glasses", WSRC-RP-2001-00080, Revision 0, January 3, 2001.
- [3] Edwards, T. B., "Selecting Test Compositions for the FY01 Glass Studies of the Salt Processing Program," SRT-SCS-2000-00069, December 15, 2000.

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Attachment II.

Edwards, T. B., "An Analytical Plan for the SRTC Mobile Laboratory to Follow in Measuring PCT Solutions for the PHA Glasses of the SDP FY01 Study (U), SRT-SCS-2001-00005, January 12, 2001.

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WESTINGHOUSE SAVANNAH RIVER COMPANY
INTEROFFICE MEMORANDUM

SRT-SCS-2001-00005

January 12, 2001

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From: T. B. Edwards, 773-42A (5-5148)
Statistical Consulting Section

wi - with sample identifiers
wo - without sample identifiers
es - executive summary only

R. A. Baker, Technical Reviewer

Date

R. C. Tuckfield, Manager
Statistical Consulting Section

Date

**An Analytical Plan for the
SRTC Mobile Laboratory to Follow in Measuring PCT
Solutions for the PHA Glasses of the SDP FY01 Study
(U)**

EXECUTIVE SUMMARY

The Immobilization Technology Section currently is conducting a study of the impact of cooling rate on glass durability as part of the Salt Disposition Program (SDP). The Product Consistency Test, or PCT, is used as a measure of glass durability, and its requirements are described in ASTM C1285-97 (Method A). Each PCT results in a leachate solution whose elemental concentrations must be measured to complete the determination of glass durability. In addition to the test glasses, PCTs are usually conducted on samples of Approved Reference Material (ARM) glass and samples of Environmental Assessment (EA) glass. One or more reagent blank samples are also included in these tests.

This memorandum addresses the PCTs that are being conducted for the seventeen Precipitate Hydrolysis Aqueous (PHA) glasses supporting the PHA portion of the SDP glass study. The glasses were cooled by both quenching and centerline cooling, yielding thirty-four glasses that are to be subjected to the PCT.

The Savannah River Technology Center-Mobile Laboratory (SRTC-ML) is to measure elemental concentrations of the resulting leachate solutions. This memorandum provides an analytical plan for the SRTC-ML to follow in measuring the compositions of the leachate solutions resulting from the PCT procedures for these PHA glasses.

INTRODUCTION

In support of the Salt Disposition Program (SDP), the Immobilization Technology Section currently is conducting a study [1] of the impact of cooling rate on glass durabilities. The Product Consistency Test, or PCT, is used as a measure of glass durability, and its requirements are described in ASTM C1285-97 (Method A) [2].

Seventeen Precipitate Hydrolysis Aqueous (PHA) glasses were batched and fabricated as part of the SDP glass study. Each of the seventeen glasses was both quenched and centerline cooled. This resulted in 34 PHA glasses for which PCTs are to be conducted.

The Savannah River Technology Center Mobile Laboratory (SRTC-ML) is to measure the compositions of the leachate solutions resulting from the PCTs for these glasses, and this memorandum provides an analytical plan for the SRTC-ML to follow in conducting the measurements.

DISCUSSION

Thirty-four glasses from the PHA portion of the SDP glass study are to be subjected to the PCT. Each of the tests is to be conducted in triplicate. In addition to the test glasses, triplicate PCTs are to be conducted on a sample of the Approved Reference Material (ARM) glass and a sample of the Environmental Assessment (EA) glass. Two reagent blank samples are also to be included in these tests. Thus, a total of 110 samples are required to complete these PCTs.

The leachates from these tests will be diluted by adding 6 mL of 0.4 HNO₃ to 4 mL of the leachate (a 4:10, volume to volume, v:v, dilution) before being submitted to the Mobile Laboratory. The EA leachates will be further diluted (1:10, v:v) with deionized water prior to submission to the Mobile Lab in order to prevent problems with the nebulizer.

Table 1 enumerates the PHA glasses and presents identifying codes, ab001 through ab110, for their PCTs as well as for the standards: EA, ARM, and blanks. The glass identifiers in Table 1 indicate glasses that were centerline cooled via a "clc" suffix. The naming convention of Table 1 is to be used by the SRTC-ML in analyzing these solutions and reporting the relevant concentration measurements.¹

¹ Renaming these samples ensures that they will be processed as blind samples by the SRTC-ML. This table is complete only for those on the distribution list with a "wi" following their names.

Table 1: Solution Identifiers

Original Sample	Solution Identifier	Original Sample	Solution Identifier	Original Sample	Solution Identifier	Original Sample	Solution Identifier	Original Sample	Solution Identifier
pha01	ab056	pha04clc	ab013	pha08	ab091	pha12	ab082	pha15clc	ab093
pha01	ab098	pha04clc	ab106	pha08clc	ab104	pha12	ab006	pha15clc	ab002
pha01	ab041	pha05	ab085	pha08clc	ab016	pha12	ab049	pha16	ab064
pha01clc	ab108	pha05	ab109	pha08clc	ab100	pha12clc	ab055	pha16	ab105
pha01clc	ab030	pha05	ab068	pha09	ab001	pha12clc	ab097	pha16	ab076
pha01clc	ab080	pha05clc	ab037	pha09	ab034	pha12clc	ab099	pha16clc	ab052
pha02	ab066	pha05clc	ab038	pha09	ab005	pha13	ab039	pha16clc	ab065
pha02	ab040	pha05clc	ab063	pha09clc	ab057	pha13	ab073	pha16clc	ab032
pha02	ab004	pha06	ab075	pha09clc	ab078	pha13	ab019	pha17	ab045
pha02clc	ab060	pha06	ab061	pha09clc	ab107	pha13clc	ab089	pha17	ab003
pha02clc	ab050	pha06	ab026	pha10	ab070	pha13clc	ab028	pha17	ab090
pha02clc	ab017	pha06clc	ab012	pha10	ab029	pha13clc	ab024	pha17clc	ab067
pha03	ab110	pha06clc	ab023	pha10	ab095	pha14	ab087	pha17clc	ab015
pha03	ab020	pha06clc	ab018	pha10clc	ab044	pha14	ab101	pha17clc	ab010
pha03	ab043	pha07	ab086	pha10clc	ab053	pha14	ab096	EA	ab047
pha03clc	ab069	pha07	ab025	pha10clc	ab062	pha14clc	ab036	EA	ab014
pha03clc	ab048	pha07	ab011	pha11	ab088	pha14clc	ab072	EA	ab092
pha03clc	ab083	pha07clc	ab042	pha11	ab084	pha14clc	ab094	ARM	ab031
pha04	ab035	pha07clc	ab027	pha11	ab077	pha15	ab008	ARM	ab102
pha04	ab058	pha07clc	ab033	pha11clc	ab059	pha15	ab074	ARM	ab007
pha04	ab021	pha08	ab022	pha11clc	ab054	pha15	ab079	blank	ab046
pha04clc	ab081	pha08	ab051	pha11clc	ab009	pha15clc	ab103	blank	ab071

ANALYTICAL PLAN

The analytical plan for the Mobile Lab is provided in this section. Each of the solution samples submitted to the SRTC-ML is to be analyzed only once for each of the following: boron (B), lithium (Li), sodium (Na), and silicon (Si). These measurements are to be made in parts per million (ppm). The analytical procedure used by the SRTC-ML to determine the relevant concentrations involves an Inductively Coupled Plasma (ICP) – Emission Spectrometer. The PCT solutions (as identified in Table 1) are grouped in six ICP blocks for processing by the Mobile Lab in Table 2. Each block will probably require a different calibration of the ICP.

Table 2: ICP Calibration Blocks for Leachate Measurements

1	2	3	4	5	6
std-b1-1	std-b2-1	std-b3-1	std-b4-1	std-b5-1	std-b6-1
ab059	ab053	ab083	ab103	ab023	ab021
ab108	ab003	ab009	ab035	ab027	ab106
ab064	ab065	ab099	ab089	ab028	ab005
ab085	ab054	ab077	ab075	ab025	ab002
ab069	ab084	ab041	ab086	ab102	ab107
ab044	ab105	ab100	ab008	ab078	ab017
ab082	ab097	ab068	ab042	ab014	ab007
ab052	ab038	ab080	ab031	ab093	ab018
ab067	ab109	ab090	ab001	ab058	ab019
std-b1-2	std-b2-2	std-b3-2	std-b4-2	std-b5-2	std-b6-2
ab104	ab048	ab076	ab066	ab040	ab071
ab045	ab015	ab062	ab012	ab072	ab094
ab037	ab098	ab032	ab047	ab013	ab033
ab070	ab016	ab063	ab081	ab101	ab092
ab046	ab030	ab091	ab057	ab034	ab096
ab055	ab006	ab095	ab036	ab050	ab026
ab110	ab051	ab010	ab039	ab061	ab079
ab088	ab029	ab049	ab060	ab073	ab024
ab022	ab020	ab043	ab087	ab074	ab004
ab056	std-b2-3	std-b3-3	std-b4-3	std-b5-3	ab011
std-b1-3					std-b6-3

A multi-element solution standard (denoted by "std-bi-j" where i=1, 2, 3, 4, 5, and 6 represents the block number and j=1, 2, 3 represents the position in the block) was added at the beginning, middle, and end of each of the six blocks. This standard may be useful in checking and correcting for bias in the concentration measurements resulting from the ICP calibrations.

CONCLUDING COMMENTS

In summary, this analytical plan identifies six ICP calibration blocks in Table 2 that are to be used by the SRTC-ML in conducting the boron (B), lithium (Li), sodium (Na), and silicon (Si) concentration measurements for the PCT study of the PHA test glasses. The sequencing of the activities associated with each of these steps in the analytical procedures has been randomized. The size of the blocks was selected so that each block could be completed in a single work shift. If for some reason the measurements are not conducted in the sequence presented in this memorandum, the actual order used should be recorded along with any explanative comments.

The analytical plan indicated in the preceding tables should be modified by the personnel of SRTC-ML to include any calibration check standards and/or other standards that are part of the standard operating procedures.

REFERENCES

- [1] Harbour, J. R. and T. B. Edwards, "Task Technical and QA Plan: Impact of Cooling Rate on the Durabilities of CST and PHA Glasses," WSRC-RP-2000-00996, December 11, 2000.
- [2] ASTM C1285-97, "Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT)," 1997.

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