

Composition and Property Measurements for PHA Phase 4 Glasses

by

T. B. Edwards

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

J. R. Harbour

R. J. Workman

DOE Contract No. DE-AC09-96SR18500

This paper was prepared in connection with work done under the above contract number with the U. S. Department of Energy. By acceptance of this paper, the publisher and/or recipient acknowledges the U. S. Government's right to retain a nonexclusive, royalty-free license in and to any copyright covering this paper, along with the right to reproduce and to authorize others to reproduce all or part of the copyrighted paper.

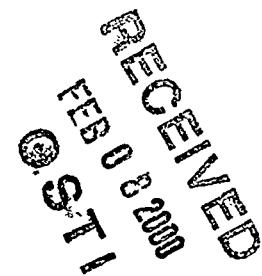
WSRC-TR-99-00294
Revision 0

Keywords: Coupled Operations,
DWPF, PCT, PCCS,
Liquidus Temperature,
Salt Disposition, Viscosity

Retention Time: Permanent

COMPOSITION AND PROPERTY MEASUREMENTS FOR PHA PHASE 4 GLASSES (U)

T. B. Edwards
J. R. Harbour
R. J. Workman



Westinghouse Savannah River Company
Savannah River Technology Center
Aiken, SC 29808



PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT NO. DE-AC09-96SR18500

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831; prices available from (615) 576-8401.

Available to the public from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161.

DISCLAIMER

**Portions of this document may be illegible
in electronic image products. Images are
produced from the best available original
document.**

COMPOSITION AND PROPERTY MEASUREMENTS FOR PHA PHASE 4 GLASSES (U)

August 18, 1999

Document Approvals

Thomas B Edwards

T. B. Edwards, Author
Statistical Consulting Section

9-16-99

Date

John N Harbour

J. R. Harbour, Author
Immobilization Technology Section

9-16-99

Date

Rhonda J. Workman

R. J. Workman, Author
Immobilization Technology Section

9-16-99

Date

K. G. Brown

K. G. Brown, Technical Review
Immobilization Technology Section

9/16/99

Date

R. C. Tuckfield

R. C. Tuckfield, Manager
Statistical Consulting Section

9/16/99

Date

E. W. Holtzscheiter

E. W. Holtzscheiter, Manager,
Immobilization Technology Section
Authorized Derivative Classifier

9-16-99

Date

This page intentionally left blank.

TABLE OF CONTENTS

Summary and Conclusions	1
Introduction	1
Results and Discussion	2
Chemical Compositions	2
PCT Results	7
Viscosity at 1150 °C	10
Liquidus Temperature (T _L)	12
Surface Crystallization	12
Phase Separation	13
Conclusions	14
References	14
Appendix A: Supplemental Tables and Exhibits	17
Appendix B: Two Additional Glasses	61

This page intentionally left blank.

SUMMARY AND CONCLUSIONS

The results presented in this report are for nine Precipitate Hydrolysis Aqueous (PHA) Phase 4 glasses. Three of the glasses contained HM sludge at 22, 26, and 30 wt% respectively, 10 wt% PHA and 1.25 wt% monosodium titanate (MST), all on an oxide basis. The remaining six glasses were selected from the Phase 1 and Phase 2 studies (Purex sludge) but with an increased amount of MST. The high-end target for MST of 2.5 wt% oxide was missed in Phases 1 and 2 due to ~30 wt% water content of the MST. A goal of this Phase 4 study was to determine whether this increase in titanium concentration from the MST had any impact on glass quality or processability. Two of the glasses, pha14c and pha15c, were rebatched and melted due to apparent batching errors with pha14 and pha15.

The models currently in the Defense Waste Processing Facility's (DWPF's) Product Composition Control System (PCCS) were used to predict durability, homogeneity, liquidus, and viscosity for these nine glasses. All of the HM glasses and half of the Purex glasses were predicted to be phase separated, and consequently prediction of glass durability is precluded with the current models for those glasses that failed the homogeneity constraint. If one may ignore the homogeneity constraint, the measured durabilities were within the 95% prediction limits of the model. Further efforts will be required to resolve this issue on phase separation (inhomogeneity).

The liquidus model predicted unacceptable liquidus temperatures for four of the nine glasses. The approximate, bounding liquidus temperatures measured for all had upper limits of 1000°C or less. Given the fact that liquidus temperatures were only approximated, the 30 wt% loading of Purex may be near or at the edge of acceptability for liquidus.

The measured viscosities were close to the predictions of the model. For the Purex glasses, pha12c and pha15c, the measured viscosities of 28 and 23 poise, respectively, indicate that DWPF processing may be compromised at the low end of the viscosity range (20 poise). Although the HM sludge glass examined (10 wt% PHA) had a measured viscosity of ~90 poise, the HM glasses at 7wt% PHA are predicted to be higher than the 100 poise limit for DWPF. Further work will be required to resolve these issues.

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 processability and product quality of the glass composition region for this alternative to the In-Tank Precipitation (ITP) Process. 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 obtain information on the feasibility of incorporating anticipated levels of PHA into DWPF glasses with and without doubling the nominal levels of monosodium titanate (MST).

A set of target compositions from which the glasses supporting this task are to be selected was provided in the memorandum appearing as Attachment I in [3]. Process and product property predictions for these glasses are also provided in that memorandum. The candidate glasses identified in that memorandum involved three sludge types: Purex, HM, and Blend; covered sludge loadings (in the glass) of 22, 26, and 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) at 1.25 and 2.5 wt%. For each composition, the remainder of the glass consisted of Frit 202. The glasses, batched and fabricated using the Purex sludge at a target loading of 26, 30 and 22 wt% of the glass, were selected to comprise Phase 1, 2, and 3, respectively, of this study and were reported in [3], [4], and [5], respectively. In Phase 4, the final phase of this study, some of the compositions (derived from Purex sludge) that were considered in the first three phases were revisited to resolve concerns that arose during these earlier investigations. In addition, three compositions derived from HM sludge were considered in this phase

of the study. The general, target compositions of the glasses initially considered in Phase 4 are provided in Table 1.

Table 1: General Composition of the PHA Phase 4 Glasses

Glass ID	Sludge Type	Sludge Loading	PHA	MST	Frit
pha20	HM	22%	10%	1.25%	66.75%
pha26	HM	26%	10%	1.25%	62.75%
pha32	HM	30%	10%	1.25%	58.75%
pha11c	Purex	26%	10%	2.5%	61.50%
pha12c	Purex	26%	13%	2.5%	58.50%
pha17c	Purex	30%	10%	2.5%	57.50%
pha18c	Purex	30%	13%	2.5%	54.50%

The glass identifiers used for these compositions include a "c" suffix for each of those glasses that are being re-batched for Phase 4 of this study. The MST used during the earlier phases of this study contained excess moisture that was not fully accounted for in the batching process resulting in TiO_2 levels in the glass falling somewhat short of their intended targets. Thus, one objective for Phase 4 is to ensure that no problems are seen for glasses fully loaded with (i.e., with targets met for) TiO_2 .

The properties of interest for these glasses included durability (as measured by the 7-day Product Consistency Test (PCT) [6]), viscosity at 1150°C, and liquidus temperature. The purpose of this report is to provide and investigate comparisons between

- the measured and target compositions of this set initial of Phase 4 PHA glasses and
- the property measurements and their predictions.

Phase 4 is the last of the investigative efforts for this PHA glass study

RESULTS AND DISCUSSION

The initial seven glasses comprising Phase 4 of the PHA study were designated as pha20, pha26, pha32, pha11c, pha12c, pha17c, and pha18c. Composition and property measurements of these glasses were conducted in parallel with the seven glasses comprising Phase 4 of the other ITP replacement alternative designated as the Crystalline Silicotitanate (CST) study [7]. This helps ensure that the CST and PHA glasses are fabricated, characterized, and analyzed under very similar conditions. The analytical plans that were used to generate the measurements required to support both (CST and PHA) studies appear as attachments to [7], [8], and [9]. These plans, which are identified in the discussion that follows, were prepared to support the overall Technical Task and QA plan [2] and the analytical study plan [10]. The results of these measurements (both composition and property) are presented in this section.

Chemical Compositions

Table 2 provides the target oxide compositions for all candidate PHA glasses using Purex sludge. Table 3 provides the composition of the three HM glasses included in Phase 4. See Attachment I of [3] for details on the development of these target compositions. The Glass ID column can be used to identify the rows of Table 2 corresponding to the compositions of the Phase 4 glasses, if the "c" suffix is ignored.

Table 2: Target Oxide Composition (in weight percents, wt%’s) of the PHA Glasses Fabricated with Purex Sludge

Glass																
Sludge	MST	PHA	Frit 202	ID	Al ₂ O ₃	B ₂ O ₃	BaO	CaO	Cr ₂ O ₃	CuO	Fe ₂ O ₃	K ₂ O	Li ₂ O	MgO	MnO	Na ₂ O
22	1.250	7	69.750	pha01	2.540	7.974	0.084	0.945	0.106	0.568	9.899	3.350	4.785	1.448	1.727	7.869
22	1.250	10	66.750	pha02	2.522	8.803	0.084	0.941	0.106	0.791	9.897	4.730	4.579	1.389	1.727	8.017
22	1.250	13	63.750	pha03	2.504	9.632	0.084	0.936	0.106	1.014	9.894	6.110	4.373	1.329	1.727	8.165
22	2.500	7	68.500	pha04	2.532	7.876	0.084	0.943	0.106	0.568	9.898	3.350	4.699	1.423	1.727	7.944
22	2.500	10	65.500	pha05	2.514	8.705	0.084	0.939	0.106	0.791	9.896	4.730	4.493	1.364	1.727	8.092
22	2.500	13	62.500	pha06	2.496	9.534	0.084	0.934	0.106	1.014	9.893	6.110	4.288	1.304	1.727	8.240
26	1.250	7	65.750	pha07	2.901	7.660	0.099	1.092	0.125	0.576	11.685	3.365	4.510	1.381	2.041	8.116
26	1.250	10	62.750	pha08	2.883	8.488	0.099	1.088	0.125	0.800	11.683	4.745	4.305	1.322	2.041	8.264
26	1.250	13	59.750	pha09	2.865	9.317	0.099	1.083	0.125	1.023	11.681	6.125	4.099	1.262	2.041	8.412
26	2.500	7	64.500	pha10	2.894	7.561	0.099	1.090	0.125	0.576	11.684	3.365	4.425	1.356	2.041	8.191
26	2.500	10	61.500	pha11	2.876	8.390	0.099	1.086	0.125	0.800	11.682	4.745	4.219	1.297	2.041	8.339
26	2.500	13	58.500	pha12	2.858	9.219	0.099	1.081	0.125	1.023	11.680	6.125	4.013	1.237	2.041	8.487
30	1.250	7	61.750	pha13	3.263	7.345	0.114	1.239	0.144	0.585	13.472	3.380	4.236	1.314	2.355	8.363
30	1.250	10	58.750	pha14	3.245	8.174	0.114	1.234	0.144	0.808	13.470	4.760	4.030	1.255	2.355	8.511
30	1.250	13	55.750	pha15	3.227	9.003	0.114	1.230	0.144	1.031	13.467	6.140	3.824	1.195	2.355	8.659
30	2.500	7	60.500	pha16	3.256	7.246	0.114	1.237	0.144	0.585	13.471	3.379	4.150	1.289	2.355	8.438
30	2.500	10	57.500	pha17	3.238	8.075	0.114	1.233	0.144	0.808	13.469	4.759	3.945	1.230	2.355	8.586
30	2.500	13	54.500	pha18	3.220	8.904	0.114	1.228	0.144	1.031	13.466	6.139	3.739	1.170	2.355	8.734

Table 2: Target Oxide Composition (in weight percents, wt%’s) of the PHA Glasses Fabricated with Purex Sludge (continued)

Glass															
Sludge	MST	PHA	Frit 202	ID	NiO	P ₂ O ₅	PbO	SiO ₂	TiO ₂	U ₃ O ₈	ZnO	ZrO ₂	F	Cl ⁻	(SO ₄) ⁻
22	1.250	7	69.750	pha01	0.930	0.030	0.096	53.684	1.128	2.003	0.086	0.109	0.032	0.240	0.173
22	1.250	10	66.750	pha02	0.930	0.030	0.096	51.404	1.127	2.003	0.086	0.109	0.032	0.240	0.173
22	1.250	13	63.750	pha03	0.930	0.030	0.096	49.124	1.125	2.003	0.086	0.109	0.032	0.240	0.173
22	2.500	7	68.500	pha04	0.930	0.030	0.096	52.734	2.226	2.003	0.086	0.109	0.032	0.240	0.173
22	2.500	10	65.500	pha05	0.930	0.030	0.096	50.454	2.225	2.003	0.086	0.109	0.032	0.240	0.173
22	2.500	13	62.500	pha06	0.930	0.030	0.096	48.174	2.224	2.003	0.086	0.109	0.032	0.240	0.173
26	1.250	7	65.750	pha07	1.099	0.036	0.114	50.766	1.126	2.367	0.102	0.129	0.038	0.283	0.205
26	1.250	10	62.750	pha08	1.099	0.036	0.114	48.486	1.125	2.367	0.102	0.129	0.038	0.283	0.205
26	1.250	13	59.750	pha09	1.099	0.036	0.114	46.206	1.124	2.367	0.102	0.129	0.038	0.283	0.205
26	2.500	7	64.500	pha10	1.099	0.036	0.114	49.816	2.224	2.367	0.102	0.129	0.038	0.283	0.205
26	2.500	10	61.500	pha11	1.099	0.036	0.114	47.536	2.223	2.367	0.102	0.129	0.038	0.283	0.205
26	2.500	13	58.500	pha12	1.099	0.036	0.114	45.256	2.222	2.367	0.102	0.129	0.038	0.283	0.205
30	1.250	7	61.750	pha13	1.268	0.041	0.132	47.849	1.125	2.731	0.118	0.149	0.043	0.327	0.236
30	1.250	10	58.750	pha14	1.268	0.041	0.132	45.569	1.123	2.731	0.118	0.149	0.043	0.327	0.236
30	1.250	13	55.750	pha15	1.268	0.041	0.132	43.289	1.122	2.731	0.118	0.149	0.043	0.327	0.236
30	2.500	7	60.500	pha16	1.268	0.041	0.132	46.899	2.223	2.731	0.118	0.149	0.043	0.327	0.236
30	2.500	10	57.500	pha17	1.268	0.041	0.132	44.619	2.221	2.731	0.118	0.149	0.043	0.327	0.236
30	2.500	13	54.500	pha18	1.268	0.041	0.132	42.339	2.220	2.731	0.118	0.149	0.043	0.327	0.236

Table 3: Target Compositions of PHA Phase 4 HM Glasses

Glass ID	pha20	pha26	pha32	Glass ID	pha20	pha26	pha32
Sludge	22	26	30	Sludge	22	26	30
MST	1.25	1.25	1.25	MST	1.25	1.25	1.25
PHA	10	10	10	PHA	10	10	10
Frit 202	66.75	62.75	58.75	Frit 202	66.75	62.75	58.75
Al ₂ O ₃	6.046	7.048	8.051	NiO	0.336	0.397	0.458
B ₂ O ₃	8.803	8.488	8.174	P ₂ O ₅	0.032	0.038	0.044
BaO	0.045	0.053	0.061	PbO	0.053	0.063	0.073
CaO	0.496	0.562	0.628	SiO ₂	54.086	51.656	49.226
Cr ₂ O ₃	0.073	0.086	0.099	TiO ₂	1.127	1.125	1.123
CuO	0.761	0.764	0.767	U ₃ O ₈	0.677	0.800	0.923
Fe ₂ O ₃	6.363	7.507	8.652	ZnO	0.014	0.016	0.019
K ₂ O	4.723	4.736	4.749	ZrO ₂	0.119	0.141	0.163
Li ₂ O	4.579	4.305	4.030	F ⁻	0.037	0.044	0.051
MgO	1.463	1.409	1.355	Cl ⁻	0.124	0.147	0.169
MnO	1.955	2.311	2.667	(SO ₄) ⁻	0.127	0.150	0.173
Na ₂ O	7.776	7.980	8.183				

Predictions for the properties of interest generated for these target compositions by the models utilized by the Defense Waste Processing Facility (DWPF) are also included in the discussion provided in Attachment I of [3]. 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. Comparisons between property predictions and property measurements are provided for these Phase 4 PHA glasses in the discussion that follows.

The initial seven glasses were batched and fabricated to the target compositions corresponding to the appropriate rows of Table 2 and to Table 3. In addition to the Phase 4 glasses (both CST and PHA), a standard glass (WCP 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) was provided to assist the SRTC-Mobile Laboratory (SRTC-ML) in conducting these analyses (see Attachment I of [8]). Due to outages at this laboratory, these glasses were subsequently submitted to the Analytical Development Section (ADS) of SRTC for chemical composition analysis.

Glasses were batched using the appropriate combinations of Purex sludge, glass formers, PHA, and MST. The simulated Purex and HM sludges were batched from dry chemicals and have oxide compositions provided in Attachment I of [3]. PHA was batched from chemicals and has an oxide composition provided in Table 2 of Attachment I of [3]. A basic MST solution was obtained from D. Hobbs. This material was washed and then dried. The composition of MST was determined by the Mobile Lab and is presented in Table 1 of Attachment I of [3]. Frit 202, Lot 14 was obtained from the DWPF. The Frit 202 composition is given in Table 7 of Attachment I of [3].

For each glass, the combined powders (~120 grams) were added to a 250 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.

Tables A.1 and A.2 in Appendix A provide the composition measurements obtained by ADS following an analytical plan similar to that provided in Attachment I of [8].¹ Two types of dissolutions (microwave and peroxide fusion) were used to generate these composition measurements. Table A.1 provides the peroxide fusion (pf) results and Table A.2 the microwave (MW) results. Calcium and silicon cation concentrations were measured using both preparation methods.

Exhibit A.1 in Appendix A provides a plot of the measurements by glass sample id by oxide. The results for pha26 call for a closer look at each of the microwave dissolutions. They are provided in Table 4.

Table 4: Comparison of MW Dissolutions for pha26

Oxide	Prep 1	Prep 2	Target
Al ₂ O ₃ (wt%)	2.777	6.453	7.048
CaO MW (wt%)	0.997	0.514	0.562
Cr ₂ O ₃ (wt%)	0.163	0.120	0.086
CuO (wt%)	0.795	0.691	0.764
Fe ₂ O ₃ (wt%)	10.137	7.348	7.507
K ₂ O (wt%)	4.485	4.445	4.736
Li ₂ O (wt%)	4.862	4.391	4.305
MgO (wt%)	1.442	1.286	1.409
MnO (wt%)	2.029	2.320	2.311
Na ₂ O (wt%)	8.374	7.952	7.980
Nb ₂ O ₅ (wt%)	0.074	0.072	0.000
NiO (wt%)	0.925	0.377	0.397
SiO ₂ MW (wt%)	49.471	49.100	51.656
TiO ₂ (wt%)	1.219	1.173	1.125
U ₃ O ₈ (wt%)	1.917	0.954	0.800
ZrO ₂ (wt%)	0.197	0.171	0.141

¹ The dissolved samples were prepared according to the plan, and then ADS LIMS numbers were randomly assigned to the dissolved samples.

Due to the inconsistency between the results for the two microwave dissolutions (and considering the target composition), the results from the first dissolution are deemed unrepresentative and excluded from further consideration in this report.

A review of the results from the standards was conducted for insight into the possibility that the ICP calibration contributes (in a systematic way) to the variation seen in the oxide measurements for the Phase 4 glasses. Exhibits A.2 and A.3 in Appendix A provide plots of the oxide measurements per analytical block by oxide by dissolution method. Table 5 provides the average measured composition for the two types of standards included in this analytical plan by analytical block. The reference values for the standards are also provided in this table.

Table 5: Measurements from Glass Standards from Phase 4

Oxide	std (Batch 1)			Ustd (Uranium-bearing Standard)		
	Analytical Block		Reference	Analytical Block		Reference
	1	2		1	2	
Al ₂ O ₃	4.853	4.895	4.877	4.002	4.021	4.100
B ₂ O ₃	7.640	7.899	7.777	10.015	9.937	9.209
CaO (pf)	1.621	1.588	1.220	1.644	1.747	1.301
CaO (MW)	1.240	1.244	1.220	1.309	1.312	1.301
CaO (avg)	1.431	1.416	1.220	1.477	1.530	1.301
Cr ₂ O ₃	0.105	0.108	0.107	0.261	0.259	0.000
CuO	0.400	0.403	0.399	0.013	0.014	0.000
Fe ₂ O ₃	13.384	13.493	12.839	13.836	13.863	13.196
K ₂ O	3.002	3.348	3.327	2.813	3.013	2.999
Li ₂ O	4.581	4.608	4.429	3.133	3.136	3.057
MgO	1.442	1.450	1.419	1.197	1.199	1.210
MnO	1.737	1.741	1.726	2.809	2.798	2.892
Na ₂ O	9.223	9.265	9.003	12.083	12.083	11.795
Nb ₂ O ₅	0.070	0.070	0.000	0.073	0.073	0.000
NiO	0.780	0.786	0.751	1.104	1.114	1.120
SiO ₂ (pf)	50.906	52.733	50.220	51.893	51.505	45.353
SiO ₂ (MW)	47.740	47.807	50.220	43.466	43.463	45.353
SiO ₂ (avg)	49.323	50.270	50.220	47.680	47.484	45.353
TiO ₂	0.700	0.704	0.677	1.014	1.015	1.049
U ₃ O ₈	0.340	0.340	0.000	2.404	2.377	2.406
ZrO ₂	0.121	0.123	0.098	0.009	0.010	0.000
Sum of Oxides	99.132	100.919	98.869	103.922	103.927	99.687

The analytical results from the Batch 1 samples were used to bias-correct for a possible ICP calibration effect (a block effect) in the other measurements.² This was accomplished for each oxide in turn by taking the original oxide measurement, noting its block, and then multiplying the measurement by the ratio of the corresponding reference value for Batch 1 divided by the average oxide measurement for Batch 1 in that block. The calcium and silicon values for each dissolution method were adjusted via this process. This approach was used to bias-correct the composition measurements of the Phase 3 and standard glasses.

Exhibit A.4 in Appendix A provides plots of these measurements for each oxide over all of the glasses (including the standards), and Table 6 provides summary information for these measurements. The sums of oxides for the target, measured, and measured bias-corrected (bc) compositions are also provided. A review of these sums shows that they are all within the interval of 95 to 105 weight percent with the smallest value being 96.8 wt% for the bias-corrected composition of pha32 and the largest being 103.9 for the uranium-bearing glass standard.

² Bias corrections of this type have been advantageous (see for example "A Statistical Review of Data from the SRTC Mobile Laboratory," WSRC-RP-98-00430, Revision 0, June 15, 1998) but not always. In some instances, bias correction does not improve the accuracy of the results. Measurements are bias-corrected in this report, and bias-corrected values are considered in the comparisons that follow. Conclusions, developed from these comparisons, that are insensitive to the way the glass compositions are represented (target, measured, or bias-corrected) demonstrate robustness to which representation might be nearer the true composition for each glass.

Another observation from this exhibit and table is the closeness of the TiO_2 measurements to their respective targets for these PHA glasses. A problem was seen in Phases 1 [3] and 2 [4]. Namely, the TiO_2 measurements for the PHA glasses fell short of their respective target values for the first two phases even though the measurements for the standards compared very favorably to their targets. This behavior prompted a re-evaluation a source of TiO_2 , the MST. A subsequent analysis of MST revealed a larger than expected moisture content (~30wt%). However, as discussed in [3], glasses for Phases 1 and 2 were batched and fabricated prior to this discovery. Batching formulations were subsequently modified to account for the additional loss that would be expected for this situation. Glasses for Phases 3 and 4 were batched in a manner fully accounting for the additional moisture. In addition, Phase 4 included selected glasses from Phases 1 (pha11c and pha12c) and 2 (pha17c and pha18c) that were re-batched using the new formulations. The results from Table 6 and Exhibit A.4 indicate that better coverage was achieved for the higher MST loadings for these phases and that the MST loadings were successfully targeted for the glasses of this phase.

Table 6: Target, Measured and Bias-Corrected Compositions (in wt%) for the Phase 4 Glasses

	Batch 1			Uranium Standard (Ustd)			pha11c		
	Target	Measured	Measured Bias-cor.	Target	Meas.	Measured Bias-cor.	Target	Measured	Measured Bias-cor.
Al_2O_3	4.877	4.874	4.877	4.100	4.011	4.014	2.876	2.783	2.784
B_2O_3	7.777	7.769	7.777	9.209	9.976	9.990	8.390	8.420	8.429
CaO	1.220	1.423	1.220	1.301	1.503	1.289	1.086	1.118	0.966
Cr_2O_3	0.107	0.107	0.107	0.000	0.260	0.261	0.125	0.156	0.156
CuO	0.399	0.402	0.399	0.000	0.013	0.013	0.800	0.754	0.749
Fe_2O_3	12.839	13.439	12.839	13.196	13.850	13.232	11.682	11.171	10.672
K_2O	3.327	3.175	3.327	2.999	2.913	3.055	4.745	4.109	4.307
Li_2O	4.429	4.594	4.429	3.057	3.135	3.022	4.219	4.403	4.245
MgO	1.419	1.446	1.419	1.210	1.198	1.175	1.297	1.295	1.270
MnO	1.726	1.739	1.726	2.892	2.804	2.783	2.041	2.067	2.052
Na_2O	9.003	9.244	9.003	11.795	12.083	11.768	8.339	8.310	8.094
NiO	0.751	0.783	0.751	1.120	1.109	1.064	1.099	0.971	0.932
SiO_2	50.220	49.796	50.220	45.353	47.582	47.906	47.536	48.730	49.149
TiO_2	0.677	0.702	0.677	1.049	1.014	0.978	2.223	2.219	2.140
U_3O_8	0.000	0.340	0.340	2.406	2.390	2.390	2.367	2.101	2.101
ZrO_2	0.098	0.122	0.098	0.000	0.009	0.008	0.129	0.173	0.139
Sum of Oxides	98.869	100.026	99.280	99.687	103.924	103.022	98.954	98.854	98.262
	pha12c			pha17c			pha18c		
	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.	Target	Measured	Measured Bias-cor.
Al_2O_3	2.858	2.705	2.707	3.238	3.065	3.067	3.220	3.458	3.460
B_2O_3	9.219	9.283	9.297	8.075	7.763	7.770	8.904	8.998	9.007
CaO	1.081	1.159	0.999	1.233	1.270	1.093	1.228	1.361	1.179
Cr_2O_3	0.125	0.146	0.146	0.144	0.182	0.183	0.144	0.180	0.180
CuO	1.023	0.991	0.985	0.808	0.773	0.768	1.031	1.081	1.074
Fe_2O_3	11.680	11.348	10.842	13.469	13.470	12.869	13.466	13.985	13.361
K_2O	6.125	5.397	5.661	4.759	4.510	4.733	6.139	5.399	5.662
Li_2O	4.013	4.280	4.126	3.945	4.024	3.879	3.739	3.907	3.767
MgO	1.237	1.248	1.224	1.230	1.091	1.070	1.170	1.202	1.180
MnO	2.041	1.984	1.969	2.355	2.659	2.639	2.355	2.617	2.598
Na_2O	8.487	8.510	8.289	8.586	8.208	7.994	8.734	9.046	8.810
NiO	1.099	0.980	0.940	1.268	1.200	1.152	1.268	1.207	1.158
SiO_2	45.256	46.927	47.333	44.619	44.409	44.795	42.339	42.936	43.322
TiO_2	2.222	2.222	2.143	2.221	2.291	2.210	2.220	2.319	2.237
U_3O_8	2.367	2.328	2.328	2.731	2.111	2.111	2.731	2.274	2.274
ZrO_2	0.129	0.155	0.125	0.149	0.181	0.145	0.149	0.181	0.146
Sum of Oxides	98.962	99.737	99.187	98.830	97.280	96.551	98.837	100.226	99.490

Table 6: Target, Measured and Bias-Corrected Compositions (in wt%) for the Phase 4 Glasses
(continued)

	pha20			pha26			pha32		
	Target	Measured	Measured	Target	Measured	Measured	Target	Measured	Measured
Al ₂ O ₃	6.046	5.457	5.461	7.048	6.453	6.457	8.051	7.112	7.117
B ₂ O ₃	8.803	8.684	8.693	8.488	8.365	8.373	8.174	7.996	8.004
CaO	0.496	0.553	0.472	0.562	0.660	0.531	0.628	0.697	0.597
Cr ₂ O ₃	0.073	0.113	0.113	0.086	0.120	0.120	0.099	0.126	0.127
CuO	0.761	0.706	0.702	0.764	0.691	0.687	0.767	0.790	0.786
Fe ₂ O ₃	6.363	6.983	6.671	7.507	7.348	7.020	8.652	8.286	7.916
K ₂ O	4.723	4.388	4.601	4.736	4.445	4.662	4.749	4.356	4.567
Li ₂ O	4.579	4.585	4.420	4.305	4.391	4.233	4.030	4.080	3.933
MgO	1.463	1.202	1.179	1.409	1.286	1.262	1.355	1.104	1.083
MnO	1.955	2.024	2.009	2.311	2.320	2.303	2.667	2.738	2.717
Na ₂ O	7.776	7.509	7.314	7.980	7.952	7.745	8.183	8.030	7.821
NiO	0.336	0.341	0.327	0.397	0.377	0.362	0.458	0.439	0.421
SiO ₂	54.086	53.279	53.711	51.656	52.378	52.548	49.226	49.049	49.453
TiO ₂	1.127	1.146	1.105	1.125	1.173	1.131	1.121	1.157	1.116
U ₃ O ₈	0.677	0.763	0.763	0.800	0.954	0.954	0.923	0.923	0.923
ZrO ₂	0.119	0.155	0.124	0.141	0.171	0.138	0.163	0.205	0.165
Sum of Oxides	99.383	97.959	97.738	99.315	99.102	98.814	99.246	97.165	96.822

PCT Results

The seven PHA glasses making up Phase 4, after being batched and fabricated, were subjected to the 7-day Product Consistency Test (PCT) as an assessment of their durabilities [6]. More specifically, Method A of the PCT (ASTM C1285) was used for these measurements. Since durability is the critical product quality metric for vitrified nuclear waste, a review of the PCTs for these glasses was seen as a prerequisite for additional testing of these glasses. The PCTs were to be conducted in triplicate for the Phase 4 glasses. In addition, PCTs were also conducted in triplicate for samples of the Environmental Assessment (EA) glass, the ARM glass, and a blank (ASTM Type I water). An analytical plan supporting these tests was provided in the form of a memorandum (see Attachment II of [9]). This plan was intended to assist the SRTC-ML in measuring the compositions of the solutions resulting from the PHA and CST Phases 3 and 4 PCTs. Due to equipment problems, the SRTC-ML was not able to complete these analyses; they were subsequently completed by the Analytical Development Section (ADS) of SRTC. 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 this 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. Any measurement determined to be below detection was replaced by its detection limit in subsequent analyses. PCT leachate concentrations are typically normalized using the cation composition (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 target cation compositions 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 comparisons. 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 (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.

As a preliminary step to completing these normalizations of the PCTs, a review of the data was conducted. Exhibit A.5 in Appendix A provides plots of the leachate concentrations by sample id and by element with and without the EA and the blank samples. No problems are seen in these data, in that the results are reasonably consistent across all Phase 4 and standard glasses. Table 7 provides a look at the results from the three analyses of the multi-element standard solution that were included in the analytical plan. These results also indicate consistent and reasonably accurate results from these analyses.

Table 7: Measurements of Standard Solution

Block	Sequence	B (ppm)	Si (ppm)	Na (ppm)	Li (ppm)
1	1	19.5	48.9	82.7	9.9
1	1	19.8	49.5	84.2	10.1
1	1	19.7	49.0	83.5	10.0
Block 1	average	19.6	49.1	83.5	10.0
2	2	19.4	49.6	81.8	9.7
2	2	19.5	49.3	81.6	9.7
3	2	19.4	49.2	81.4	9.7
Block 2	average	19.4	49.4	81.6	9.7
3	3	19.1	48.8	80.6	9.5
3	3	19.3	49.0	81.5	9.6
3	3	19.3	49.3	82.1	9.7
Block 3	average	19.2	49.0	81.4	9.6
4	4	19.2	49.0	81.8	9.7
4	4	19.4	49.1	82.4	9.8
4	4	19.3	49.1	82.2	9.7
Block 4	average	19.3	49.1	82.1	9.7
5	5	19.7	49.0	83.4	10.0
5	5	19.8	49.0	83.7	10.1
5	5	19.8	49.0	83.7	10.1
Block 5	average	19.7	49.0	83.6	10.0
6	6	19.5	48.6	83.0	10.0
6	6	19.6	48.6	83.3	10.0
6	6	19.7	49.0	83.7	10.1
Block 6	average	19.6	48.7	83.3	10.0
Overall	average	19.5	49.0	82.6	9.9
Reference	Value	20	50	81	10
% Difference		-2.6%	-1.9%	2.0%	-1.5%

Table 8 provides the results from the normalization process using the information in Table 6 and Table A.3. Exhibit A.6 in Appendix A provides scatter plots for these results offering an opportunity to investigate the consistency in the leaching across the elements for the glasses of this study. This consistency is typically demonstrated by a high degree of linear correlation among the values. For the PCTs normalized using the either target, measured, or bias-corrected compositions, each of these correlations is at least 99%.

Table 8: Normalized PCTs for Phase 4

Glass ID	Composition	log NL [B (g/L)]	log NL [Si (g/L)]	log NL [Na (g/L)]	log NL [Li (g/L)]	NL B (g/L)	NL Si (g/L)	NL Na (g/L)	NL Li (g/L)
ARM	reference comp. [11]	-0.29085	-0.54854	-0.27396	-0.21652	0.51	0.28	0.53	0.61
EA	reference comp. [11]	1.28207	0.63353	1.18838	1.04022	19.15	4.30	15.43	10.97
pha11c	measured	0.00658	0.27729	-0.00643	0.00240	1.02	0.53	0.99	1.01
pha11c	measured, bias-cor.	0.00612	-0.28101	0.00504	0.01831	1.01	0.52	1.01	1.04
pha11c	target	0.00815	-0.26651	-0.00793	0.02095	1.02	0.54	0.98	1.05
pha12c	measured	0.17737	-0.19919	0.15490	0.15261	1.50	0.63	1.43	1.42
pha12c	measured, bias-cor.	0.17672	-0.20293	0.16637	0.16851	1.50	0.63	1.47	1.47
pha12c	target	0.18039	-0.18344	0.15610	0.18061	1.51	0.66	1.43	1.52
pha17c	measured	0.08084	-0.26107	0.07914	0.06536	1.20	0.55	1.20	1.16
pha17c	measured, bias-cor.	0.08047	-0.26483	0.09060	0.08127	1.20	0.54	1.23	1.21
pha17c	target	0.06374	-0.26312	0.05958	0.07393	1.16	0.55	1.15	1.19
pha18c	measured	0.24209	-0.19310	0.19460	0.20194	1.75	0.64	1.57	1.59
pha18c	measured, bias-cor.	0.24166	-0.19699	0.20606	0.21784	1.74	0.64	1.61	1.65
pha18c	target	0.24667	-0.18702	0.20982	0.22103	1.76	0.65	1.62	1.66
pha20	measured	-0.30200	-0.52025	-0.27890	-0.24396	0.50	0.30	0.53	0.57
pha20	measured, bias-cor.	-0.30245	-0.52376	-0.26743	-0.22806	0.50	0.30	0.54	0.59
pha20	target	-0.30793	-0.52678	-0.29405	-0.24342	0.49	0.30	0.51	0.57
pha26	measured	-0.33060	-0.54519	-0.31561	-0.26829	0.47	0.28	0.48	0.54
pha26	measured, bias-cor.	-0.33098	-0.54660	-0.30414	-0.25238	0.47	0.28	0.50	0.56
pha26	target	-0.33691	-0.53916	-0.31714	-0.25972	0.46	0.29	0.48	0.55
pha30	measured	-0.33341	-0.53492	-0.30177	-0.25942	0.46	0.29	0.50	0.55
pha30	measured, bias-cor.	-0.33383	-0.53848	-0.29031	-0.24352	0.46	0.29	0.51	0.57
pha30	target	-0.34298	-0.53649	-0.30997	-0.25411	0.45	0.29	0.49	0.56

As seen in Table 8, the durabilities for the PHA Phase 4 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.). Figure 1 provides an opportunity for a closer look at these results using measured and bias-corrected compositions. Figure 1 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 and bias-corrected) compositions [11]. 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), ARM (shown as a “z”), and the PHA glasses (each shown as an “x”) are presented on this plot. Note that the PHA results reveal acceptable PCTs that are well predicted by the current DWPF durability model. Figure 2 provides a plot of the boron results based target compositions. Exhibit A.7 in Appendix A provides similar plots of the PHA durability measurements versus the DWPF durability models for B, Si, Na, and Li. The behaviors seen in the plots for Si, Na, and Li are similar to that demonstrated by the B results: acceptable and predictable PCTs.

Figure 1.
Log NL(B) (g/L) By del Gp
(Using PHA Measured & Bias-corrected
EA and ARM reference compositions)

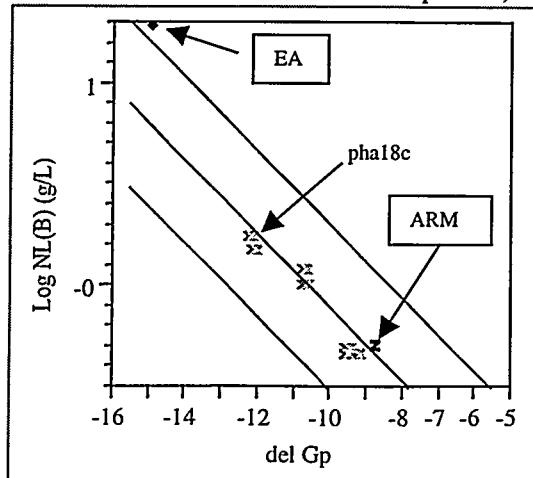
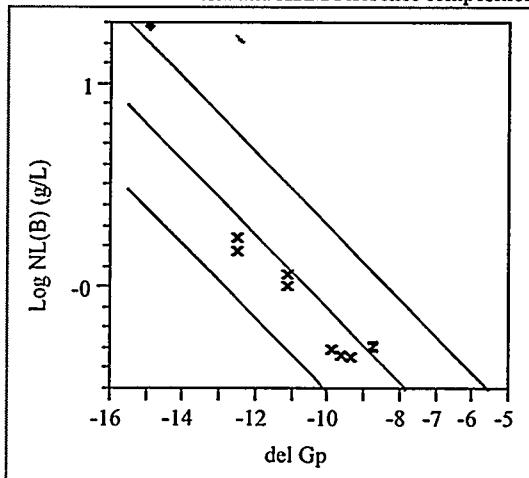


Figure 2.
Log NL(B) (g/L) By del Gp
(Using PHA target compositions &
EA and ARM reference compositions)



An additional point regarding Figures 1 and 2 should be made, specifically about pha18c. Recall that this composition was a re-batch of an earlier Phase 2 glass. The batched composition of Phase 2 pha18 missed its original target due to a batching problem. The Phase 4 pha18c hit its target composition, as was noted in the previous section. There was one other feature of pha18 in Phase 2: its PCT result was unpredictable by the DWPF model and unacceptable as compared to DWPF's acceptance region for this property [4]. Glass pha15 of Phase 2 demonstrated the same behavior [4]. The PCT for pha18c of Phase 4 was both predictable and acceptable.

This improvement in the PCT outcome for pha18c prompted a desire to take a re-look at pha15. This glass, pha15, and another Phase 2 glass, pha14, were re-batched; these glasses were designated as pha15c and pha14c. Their chemical compositions were measured and they were subjected to the 7-day PCT. See Appendix B for a discussion of these measurements. The normalized PCTs for these two glasses are provided in Table 9.

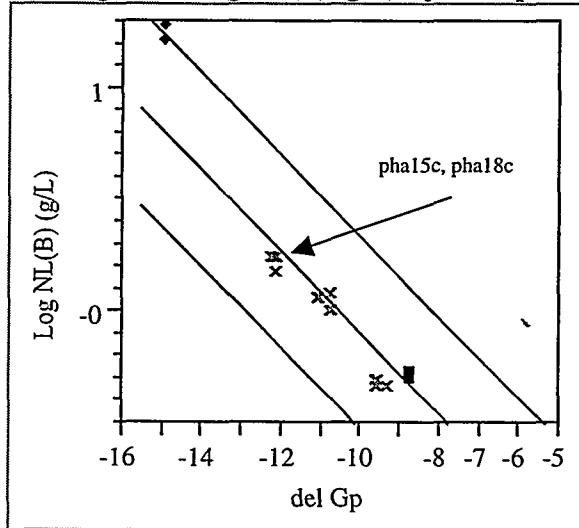
Table 9: Normalized PCTs for pha14c and pha15c

Glass ID	Composition	log NL [B (g/L)]	log NL [Si (g/L)]	log NL [Na (g/L)]	log NL [Li (g/L)]	NL B (g/L)	NL Si (g/L)	NL Na (g/L)	NL Li (g/L)
pha14c	measured	0.06317	-0.25322	0.03246	0.03802	1.16	0.56	1.08	1.09
	target	0.03885	-0.26697	0.02967	0.03252	1.09	0.54	1.07	1.08
pha15c	measured	0.24023	-0.19416	0.17513	0.18018	1.74	0.64	1.50	1.51
	target	0.21601	-0.20994	0.17687	0.17711	1.64	0.62	1.50	1.50

Figure 3 provides a plot of the DWPF durability model based upon ΔG_p (showing all nine of the Phase 4 glasses) using measured compositions for the nine glasses. The EA results are indicated with a diamond, the ARM results are indicated by a "z", and the PHA Phase 2 results by an "x".

The results of Table 9 and Figure 3 indicate that the PCT for the re-batched pha15c was both predictable and durable, the same improvement as seen for pha18c.

Figure 3: Log NL(B) (g/L) By del Gp



Viscosity at 1150°C

Viscosity measurements were made on several of the Phase 4 PHA glasses at SRTC using a Harrop, high-temperature viscometer [12]. The viscosity (in Poise) of each of these glasses at 1150°C was estimated from a Fulcher equation fitted to a set of viscosity measurements taken over an appropriate

range of temperatures. The functional form of the (three-parameter) Fulcher equation (expressed in Poise) used to fit these data is given by equation (1):

$$\ln \hat{\eta} = A + \frac{B}{(T - C)} \quad (1)$$

where A, B, and C represent the parameters of the model that were determined from the available measurements (represented by η , expressed in Poise) at various temperatures (represented by T). The fitted model was then used to predict the viscosity of the given glass at 1150°C.

Although no definitive error analysis has been completed on the use of the Harrop viscometer, SRTC has conducted several sets of viscosity measurements using this viscometer with good results [13]. Two crucible/spindle sets were used in conducting these measurements, which were sequenced according to the plan provided in Attachment IV of [9]. This plan covered the CST and PHA Phase 4 glasses and called for measurements of several of the PHA glasses to be followed by measurements of the Batch 1 standard glass with both crucible/spindle sets. Measurements of Batch 1 conducted before the planned measurements were reported in [13] and [3]. Exhibit A.8 of Appendix A provides the measured viscosities, the results of the Fulcher fit, and the prediction at 1150°C for each of the PHA Phase 4 glasses that were tested. The details of the measurements of the final two samples of Batch 1 were presented in [7]. The information presented in this exhibit (along with predictions from the DWPF viscosity model and the Batch 1 results from [3], [10] and [7]) is summarized in Table 10.

Table 10: Viscosity Results (in Poise) By Glass ID

Glass ID	Viscosity (Poise) @ 1150 °C	Predicted (measured composition)	Predicted (bias-corrected composition)	Predicted (target composition)
Batch 1	48.6, 49.7, 46.4	44.2	(Sharp-Schurtz) ³	56.2
	48.9, 47.3			
pha11c	Not Measured	45.6	51.2	40.4
pha12c	27.6	32.7	36.7	28.5
pha17c	Not Measured	32.7	37.1	31.3
pha18c	Not Measured	21.7	24.8	21.3
pha20	Not Measured	96.7	105.8	102.0
pha26	Not Measured	98.4	106.3	97.0
pha32	89.7	90.4	99.5	91.9
pha14c	Not Measured	31.4	N/A	33.3
pha15c	22.8	22.0	N/A	23.0
Batch 1	49.9, 49.5	44.2	(Sharp-Schurtz)	

The melt viscosities at 1150°C for the three PHA glasses (pha12c, pha15c, and pha32) are within the DWPF operating range. However, the HM glass with 10 wt% PHA (pha32) had a measured viscosity of ~90 poise which is close to the upper limit for DWPF operations. Predictions indicate that decreasing the PHA to 7 wt% (pha31) would increase the viscosity to 120 poise while increasing the PHA to 13 wt% (pha33) would decrease the viscosity to 69 poise. Since the model prediction for pha32 is very close to the measured viscosity, the expectation is that lower levels of PHA would lead to glasses with viscosities too high for operations at DWPF. On the other hand, at higher levels of PHA, the viscosities drop to levels comfortably within the operating range.

For the Purex glasses, pha12c and pha15c, the measured viscosities of 28 and 23 poise, respectively, indicate that DWPF processing may be compromised at the low end of the viscosity range (20 poise). Further work will be required to resolve this issue.

³ Sharp-Schurtz is the analytic arm of Owens Corning Fiberglas and is now known as Owens Corning Testing (OCT).

Liquidus Temperature (T_L)

The standard ASTM practice for measuring liquidus temperature uses a gradient furnace. The equipment for determining liquidus temperature by this method is being installed and tested within SRTC in a clean laboratory. Due to the presence of depleted uranium in the glass samples (as well as the early stage of equipment setup), we were not able to use this method for liquidus determination. A decision was therefore made to perform isothermal holds using reasonable quantities of the glass to bound the liquidus temperature.

XRD was selected as the method of detection for crystal formation in the glasses. It is estimated that the sensitivity of XRD (non-quantitative) is ~ 0.7 to 1 wt% for a crystalline phase (in this case, Trevorite). Therefore, for this type of measurement, absence of detection of a crystalline phase was evidence that the liquidus temperature is less than the temperature of that isothermal hold. On the other hand, detection of Trevorite (or any other primary crystalline phase) indicates that the liquidus temperature is higher than the temperature of the isothermal hold.

The liquidus temperature for each glass composition was estimated by performing an isothermal hold starting at 900°C and increasing in 50°C increments as required. Approximately 5 grams of each glass were placed in small platinum crucibles and transferred to a furnace already heated to 1150°C. After a four-hour hold period, the temperature was reduced to the desired temperature and held at that temperature for 24 hours. The crucibles were then removed from the furnace and the glass allowed to cool within the crucibles at room temperature. The PHA glasses at the hold temperature were submitted for XRD analysis. Care was taken to obtain glass that was not part of the top glass surface. The glass pieces, although mainly from the bulk, usually included part of the bottom surface (i.e., that surface in contact with the crucible). Those glasses containing Trevorite in the bulk glass were then taken to higher temperatures and analyzed by XRD. The approximate, bounding liquidus temperatures for the 9 PHA glasses were as given in Table 11.

Table 11: Liquidus Temperatures

Glass ID	Liquidus Temperature	T_L Prediction Using Glass Composition as Represented by	Measured	Measured bias-cor.
pha11c	<900°C	1001.5	987.3	977.5
pha12c	<900°C	1012.2	997.5	987.1
pha17c	<1000°C	1051.0	1050.9	1037.6
pha18c	<950°C	1065.7	1073.8	1059.2
pha20	<900°C	906.3	916.2	910.2
pha26	<900°C	935.6	928.3	922.3
pha32	<900°C	970.7	959.2	950.8
pha14c	<1000°C	1045.4	1059.8	N/A ⁴
pha15c	<1000°C	1059.3	1073.9	N/A

The bounding liquidus temperatures are below the nominal PAR value of 1025°C [14]. Given the fact that liquidus temperatures were only approximated, the 30 wt% loading of Purex may be near or at the edge of acceptability for liquidus. The predictions based upon measured, bias-corrected, and target compositions from DWPF's liquidus model are also provided in Table 11. The model predicts unacceptable liquidus temperatures for the glasses at 30 wt% Purex (i.e., pha14c, pha15c, pha17c, and pha18c).

Surface Crystallization

For liquidus measurements, crystal formation is considered only in the interior or bulk glass region. Therefore, samples submitted for XRD analysis were bulk samples. However, crystals can form at the interface of the glass and the crucible and/or the glass and air. For completeness, the detection of these

⁴ No opportunity for bias correction was available for these glasses. Not enough standards were in the analytical runs for these glasses.

surface crystals on the top of the glass is provided in Table 12 as a function of temperature. The crystals detected in the PHA glasses were located at the center of the top glass surface.

Table 12. Surface Crystals for the Nine PHA Glasses as a Function of Temperature

---after the 24 hour heat treatment---

	pha11c	pha12c	pha14c	pha15c	pha17c	pha18c	pha20	pha26	pha32
1150°C	No test	No test	No test	No test	No test	No test	No test	No test	No test
1000°C	No test	No test	none	none	none	No test	No test	No test	No test
950°C	No test	No test	none	No test	none	No test	No test	No test	No test
900°C	none	none	crystals	crystals	crystals	crystals	none	none	none

This composition of the surface crystals determined using EDS is consistent with the Trevorite (NiFe_2O_4) crystal structure and in agreement with crystals identified during previous studies on these types of systems. The chemical composition indicates that other cations are substituted into the crystal structure in small amounts and these include Ti, Cr, and Mn.

Phase Separation

The formation of separate amorphous phases in glass is referred to as amorphous phase separation or inhomogeneity. Crystal formation, as determined by liquidus measurements on the other hand, is also a type of phase separation, 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 property acceptance region (PAR) limit for the homogeneity constraint in the PCCS is nominally a value of 211 [14]. For the measurement acceptance region (MAR), the value will be even higher. In order to pass this constraint, the calculated value must be greater than the MAR value. The homogeneity values calculated using the measured chemical compositions reveal that all of the HM glasses and two of the Purex glasses are below the PAR value. These values are provided in Table 13.

Table 13: Homogeneity Property Predictions

Glass ID	Homogeneity Property Prediction based on (Acceptability Requires a Value > 211)		
	Target Composition	Measured Composition	Bias-Corrected Composition
pha11c	205.8	203.7	200.5
pha12c	205.4	205.2	202.0
pha17c	213.5	211.1	207.1
pha18c	213.2	218.9	214.8
pha20	201.1	199.2	197.4
pha26	209.1	206.0	203.5
pha32	217.1	208.8	206.6
pha14c	215.3	214.5	N/A
pha15c	214.9	215.6	N/A

The homogeneity constraint was developed for glasses that do contain PHA. Therefore, the predictability of phase separation by this model should be applicable. A significant search for phase separation in these glasses is beyond the scope of work for this task, except when routine SEM analysis is performed. For these six glasses, no SEM analyses were performed.

CONCLUSIONS

The results presented in this report are for nine PHA Phase 4 glasses. Three of the glasses contained HM sludge at 22, 26, and 30 wt% respectively, 10 wt% PHA and 1.25 wt% MST, all on an oxide basis. The remaining six glasses were selected from the Phase 1 and Phase 2 studies (Purex sludge) but with an increased amount of MST. The high-end target for MST of 2.5 wt% oxide was missed in Phases 1 and 2 due to ~30 wt% water content of the MST. A goal of this Phase 4 study was to determine whether this increase in titanium concentration from the MST had any impact on glass quality or processability. Two of the glasses, pha14c and pha15c, were rebatched and melted due to apparent batching errors with pha14 and pha15.

The models currently in DWPF's PCCS were used to predict durability, homogeneity, liquidus, and viscosity for these nine glasses. All of the HM glasses and half of the Purex glasses were predicted to be phase separated, and consequently prediction of glass durability is precluded with the current models for those glasses that failed the homogeneity constraint. If one may ignore the homogeneity constraint, the measured durabilities were within the 95% prediction limits of the model. Further efforts will be required to resolve this issue on phase separation (inhomogeneity).

The liquidus model predicted unacceptable liquidus temperatures for four of the nine glasses. The approximate, bounding liquidus temperatures measured for all had upper limits of 1000°C or less. Given the fact that liquidus temperatures were only approximated, the 30 wt% loading of Purex may be near or at the edge of acceptability for liquidus.

The measured viscosities were close to the predictions of the model. For the Purex glasses, pha12c and pha15c, the measured viscosities of 28 and 23 poise, respectively, indicate that DWPF processing may be compromised at the low end of the viscosity range (20 poise). Although the HM sludge glass examined (10 wt% PHA) had a measured viscosity of ~90 poise, the HM glasses at 7wt% PHA are predicted to be higher than the 100 poise limit for DWPF. Further work will be required to resolve these issues.

REFERENCES

- [1] Elder, H. H., "Technical Task Request: DWPF Waste Qualification – DWPF Coupled Operation Chemistry," HLW-SDT-TTR-99-07.0, February 2, 1999.
- [2] Harbour, J. R. and T. B. Edwards, "Technical Task and QA Plan: DWPF Coupled Operation Chemistry – PHA Glass Testing," WSRC-RP-99-00218, Revision 1, April 23, 1999.
- [3] Edwards, T. B., J. R. Harbour, and R. J. Workman, "Composition and Property Measurements for PHA Phase 1 Glasses (U)," WSRC-TR-99-00262, Revision 0, August 4, 1999.
- [4] Edwards, T. B., J. R. Harbour, and R. J. Workman, "Composition and Property Measurements for PHA Phase 2 Glasses (U)," WSRC-TR-99-00290, Revision 0, August 18, 1999.
- [5] Edwards, T. B., J. R. Harbour, and R. J. Workman, "Composition and Property Measurements for PHA Phase 3 Glasses (U)," WSRC-TR-99-00292, Revision 0, August 18, 1999.
- [6] ASTM C1285-97, "Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT)," 1997.
- [7] Edwards, T. B., J. R. Harbour, and R. J. Workman, "Composition and Property Measurements for CST Phase 4 Glasses (U)," WSRC-TR-99-00293, Revision 0, August 18, 1999.
- [8] Edwards, T. B., J. R. Harbour, and R. J. Workman, "Composition and Property Measurements for CST Phase 3 Glasses (U)," WSRC-TR-99-00291, Revision 0, August 18, 1999.

- [9] Edwards, T. B., J. R. Harbour, and R. J. Workman, "Composition and Property Measurements for CST Phase 2 Glasses (U)," WSRC-TR-99-00289, Revision 0, August 18, 1999.
- [10] Harbour, J. R. and T. B. Edwards, "Analytical Study Plan-PHA: DWPF Coupled Operation Chemistry – PHA Glass Testing," WSRC-RP-99-00315, Revision 0, April 14, 1999.
- [11] Jantzen, C. M., J. B. Pickett, K. G. Brown, T. B. Edwards, and D. C. Beām, "Process/Product Models for the Defense Waste Processing Facility (DWPF): Part I. Predicting Glass Durability from Composition Using a Thermodynamic Hydration Energy Reaction Model (THERMO) (U)," WSRC-TR-93-672, Rev. 1, September 28, 1995.
- [12] Schumacher, R. F. and D. K. Peeler, "Establishment of Harrop, High-Temperature Viscometer," WSRC-RP-98-00737, Revision 0, September 1998.
- [13] Schumacher, R. F., R. J. Workman, J. R. Harbour, and T. B. Edwards, "Measurements of DWPF Glass Viscosity – Interim Report," WSRC-RP-99-00350, Revision 0, May 5, 1999.
- [14] Brown, K. G. and R. L. Postles, "SME Acceptability Determination for DWPF Process Control (U)," WSRC-TR-95-0364, Revision 3, February 21, 1996.

This page intentionally left blank.

Appendix A:

Supplemental Tables and Exhibits

This page intentionally left blank.

**Table A.1: Composition Measurements Using Peroxide Fusion Dissolutions
(expressed as cation weight fractions)**

Glass ID	Block	Seq	LIMS #	Lab ID	B	Ca	Si
Batch 1	1	1	130865a	NA2O2 - Batch 1 std 1	0.02373	0.01109	0.23817
pha17c	1	3	130839a	NA2O2 - F14pf1	0.02288	0.00976	0.20299
pha18c	1	4	130853a	NA2O2 - F09pf1	0.02723	0.01004	0.20124
pha18c	1	6	130854a	NA2O2 - F09pf2	0.02780	0.00988	0.20499
pha20	1	7	130862a	NA2O2 - F10pf2	0.02664	0.00394	0.25885
Ustd	1	9	130868a	NA2O2 - U std -1	0.03186	0.01313	0.25186
pha26	1	12	130841a	NA2O2 - F02pf2	0.02611	0.00536	0.25223
pha32	1	13	130847a	NA2O2 - F05pf1	0.02469	0.00551	0.23859
pha20	1	15	130845a	NA2O2 - F10pf1	0.02657	0.00449	0.25647
Batch 1	1	17	130866a	NA2O2 - Batch 1 std 2	0.02396	0.01199	0.23984
pha11c	1	19	130848a	NA2O2 - F08pf1	0.02563	0.00825	0.23199
pha12c	1	21	130863a	NA2O2 - F12pf2	0.02922	0.00927	0.22949
pha12c	1	22	130863a	NA2O2 - F12pf2	0.02922	0.00927	0.22949
pha17c	1	23	130855a	NA2O2 - F14pf2	0.02413	0.01005	0.21383
Ustd	1	25	130869a	NA2O2 - U std-2	0.03035	0.01038	0.23328
pha32	1	27	130860a	NA2O2 - F05pf2	0.02411	0.00552	0.23136
pha26	1	31	130840a	NA2O2 - F02pf1	0.02469	0.00469	0.23931
pha11c	1	32	130858a	NA2O2 - F08pf2	0.02610	0.00867	0.23482
Batch 1	1	33	130867a	NA2O2 - Batch 1 std 3	0.02349	0.01167	0.23586
Batch 1	2	1	130865b	NA2O2 - BATCH 1 STD-1	0.02467	0.01076	0.24624
pha26	2	5	130841b	NA2O2 - F02pf2	0.02719	0.00511	0.26373
Ustd	2	9	130868b	NA2O2 - U std-1	0.03198	0.01369	0.25368
pha11c	2	10	130848b	NA2O2 - F08pf1	0.02646	0.00834	0.23996
pha20	2	11	130862b	NA2O2 - F10pf2	0.02725	0.00407	0.26503
pha11c	2	12	130858b	NA2O2 - F08pf2	0.02642	0.00865	0.23999
pha20	2	14	130845b	NA2O2 - F10pf1	0.02741	0.00592	0.26616
pha26	2	16	130840b	NA2O2 - F02pf1	0.02593	0.00526	0.25254
Batch 1	2	17	130866b	NA2O2 - Batch 1 std-2	0.02396	0.01125	0.24023
pha18c	2	19	130854b	NA2O2 - F09pf2	0.02832	0.01029	0.20940
pha18c	2	20	130853b	NA2O2 - F09pf1	0.02843	0.01049	0.21038
pha17c	2	22	130855b	NA2O2 - F14pf2	0.02507	0.00989	0.22353
pha32	2	23	130860b	NA2O2 - F05pf2	0.02506	0.00548	0.24274
Ustd	2	25	130869b	NA2O2 - U std-2	0.02975	0.01128	0.22782
pha32	2	26	130847b	NA2O2 - F05pf1	0.02546	0.00619	0.24724
pha17c	2	27	130839b	NA2O2 - F14pf1	0.02436	0.00999	0.21797
pha12c	2	31	130863b	NA2O2 - F12pf2	0.02815	0.00912	0.22712
pha12c	2	32	130852b	NA2O2 - F12pf1	0.02873	0.00843	0.22811
Batch 1	2	33	130867b	NA2O2 - Batch 1 std-3	0.02497	0.01203	0.25302

Values at their detection limits were set to their detection limits.

The CST Phase 4 glasses were analyzed with these PHA glasses. Their values are not presented here.

See [8] for a complete listing which includes the CST glasses.

**Table A.2: Composition Measurements From Microwave Preparation
(expressed as cation weight fractions)**

Glass	ID	Block	Seq	LIMS #	Lab ID	ICP-ES												AA ⁵			
						Al	Ca	Cr	Cu	Fe	Li	Mg	Mn	Na	Nb	Ni	Si	Ti	U	Zr	
Batch 1	1	1	1	130803a	MW - BATCH 1 STD-1	0.02572	0.00879	0.00074	0.00318	0.09252	0.02105	0.00861	0.01336	0.06798	0.00048	0.00615	0.21578	0.00417	0.00280	0.00118	0.02592
pha18c	1	4	130800a	MW - F09mw2	0.01893	0.00957	0.00127	0.00878	0.09968	0.01852	0.00751	0.02075	0.06882	0.00054	0.00967	0.19823	0.01416	0.01830	0.00141	0.04356	
pha20	1	5	130798a	MW - F10mw2	0.02836	0.00325	0.00077	0.00566	0.04860	0.02134	0.00698	0.01574	0.05468	0.00053	0.00271	0.23294	0.00691	0.00802	0.00117	0.03524	
pha26	1	6	130781a	MW - F02mw2	0.03413	0.00365	0.00084	0.00551	0.05134	0.02028	0.00773	0.01803	0.05887	0.00050	0.00300	0.22782	0.00703	0.00912	0.00131	0.03565	
pha18c	1	8	130786a	MW - F09mw1	0.01773	0.00906	0.00120	0.00846	0.09557	0.01774	0.00698	0.01990	0.06548	0.00052	0.00925	0.18943	0.01362	0.01939	0.00131	0.04283	
Ustd	1	9	130806a	MW - U STD-1	0.02131	0.00936	0.00181	0.00011	0.09711	0.01460	0.00725	0.02188	0.08988	0.00052	0.00876	0.20346	0.00610	0.02112	0.00007	0.02324	
phn32	1	11	130792a	MW - F05mw1	0.03832	0.00436	0.00089	0.00634	0.05818	0.01905	0.00681	0.02139	0.05998	0.00053	0.00346	0.21810	0.00694	0.00742	0.00141	0.03477	
pha26	1	14	130775a	MW - F02mw1	0.01467	0.00712	0.00112	0.00634	0.07076	0.02253	0.00868	0.01575	0.06201	0.00052	0.00724	0.23196	0.00730	0.01629	0.00112	0.03518	
pha12c	1	15	130782a	MW - F12mw2	0.01379	0.00738	0.00100	0.00789	0.07854	0.01981	0.00716	0.01530	0.06235	0.00048	0.00769	0.20602	0.01327	0.01983	0.00111	0.04284	
Batch 1	1	17	130804a	MW - Batch 1 Std-2	0.02610	0.00909	0.00072	0.00326	0.09527	0.02171	0.00887	0.01370	0.06977	0.00048	0.00618	0.22852	0.00428	0.00285	0.00078	0.02319	
pha32	1	19	130801a	MW - F05mw2	0.03696	0.00422	0.00084	0.00629	0.05769	0.01885	0.00652	0.02114	0.05938	0.00054	0.00344	0.21782	0.00694	0.00728	0.00162	0.03460	
pha20	1	20	130780a	MW - F10mw1	0.02949	0.00339	0.00079	0.00562	0.04902	0.02123	0.00753	0.01572	0.05678	0.00050	0.00266	0.23737	0.00682	0.00666	0.00117	0.03477	
pha12c	1	21	130787a	MW - F08mw2	0.01465	0.00744	0.00107	0.00605	0.07832	0.02048	0.00778	0.01610	0.06104	0.00052	0.00768	0.21885	0.01335	0.01839	0.00130	0.03262	
pha11c	1	22	130779a	MW - F12mw1	0.01478	0.00771	0.00100	0.00791	0.08007	0.01987	0.00787	0.01545	0.06381	0.00053	0.00770	0.21211	0.01333	0.02046	0.00114	0.04347	
pha11c	1	24	130784a	MW - F08mw1	0.01463	0.00750	0.00106	0.00594	0.07736	0.02026	0.00776	0.01582	0.06170	0.00053	0.00750	0.21780	0.01314	0.01644	0.00125	0.03245	
Ustd	1	25	130807a	MW - U Std-2	0.02104	0.00935	0.00176	0.00010	0.09645	0.01451	0.00719	0.02163	0.08938	0.00050	0.00860	0.20290	0.00605	0.01965	0.00006	0.02346	
pha17c	1	28	130794a	MW - F14mw1	0.01526	0.00773	0.00125	0.00620	0.09406	0.01863	0.00615	0.02055	0.05856	0.00050	0.00949	0.20195	0.01384	0.01630	0.00132	0.03637	
Batch 1	1	33	130805a	MW - Batch 1 Std-3	0.02523	0.00871	0.00070	0.00315	0.09305	0.02107	0.00861	0.01330	0.06751	0.00051	0.00605	0.22517	0.00414	0.00302	0.00073	0.02566	
Batch 1	2	1	130803b	MW - BATCH 1 STD-1	0.02594	0.00885	0.00074	0.00322	0.09412	0.02140	0.00873	0.01346	0.06887	0.00048	0.00617	0.21817	0.00421	0.00280	0.00117	0.02710	
pha17c	2	3	130802b	MW - F14mw2	0.01717	0.00871	0.00125	0.00616	0.09447	0.01877	0.00701	0.02069	0.06325	0.00051	0.00942	0.19869	0.01368	0.01821	0.00139	0.03789	
pha12c	2	4	130784b	MW - F08mw1	0.01496	0.00758	0.00107	0.00602	0.07818	0.02048	0.00787	0.01601	0.06248	0.00053	0.00762	0.21977	0.01331	0.01849	0.00131	0.03338	
pha18c	2	5	130782b	MW - F12mw2	0.01373	0.00741	0.00099	0.00792	0.07897	0.01988	0.00720	0.01530	0.06232	0.00048	0.00768	0.21252	0.01334	0.01956	0.00114	0.04544	
pha11c	2	6	130800b	MW - F09mw2	0.01892	0.00964	0.00125	0.00888	0.10065	0.01875	0.00758	0.02075	0.06922	0.00054	0.00968	0.20125	0.01427	0.01973	0.00134	0.04762	
pha26	2	7	130787b	MW - F08mw2	0.01467	0.00747	0.00106	0.00607	0.07869	0.02059	0.00782	0.01611	0.06138	0.00052	0.00773	0.21911	0.01342	0.01795	0.00127	0.03798	
pha26	2	8	130781b	MW - F02mw2	0.03417	0.00369	0.00080	0.00553	0.05144	0.02051	0.00778	0.01791	0.05911	0.00050	0.00293	0.23121	0.00704	0.00706	0.00123	0.03814	
Ustd	2	9	130806b	MW - U STD-1	0.02116	0.00935	0.00176	0.00009	0.09698	0.01460	0.00725	0.02168	0.08964	0.00052	0.00872	0.20418	0.00608	0.01907	0.00005	0.02507	
Batch 1	2	17	130804b	MW - BATCH 1 STD-2	0.02629	0.00903	0.00075	0.00327	0.09604	0.02180	0.00890	0.01372	0.06992	0.00048	0.00345	0.21213	0.00698	0.00829	0.00141	0.03724	
pha26	2	18	130775b	MW - F02mw1	0.01472	0.00714	0.00111	0.00636	0.07106	0.02264	0.00871	0.01568	0.06223	0.00052	0.00729	0.23053	0.00731	0.01623	0.00180	0.03928	
pha32	2	19	130792b	MW - F05mw1	0.03809	0.00432	0.00086	0.00629	0.05778	0.01890	0.00675	0.02106	0.05930	0.00053	0.00344	0.21714	0.00688	0.00832	0.00163	0.03804	
pha12c	2	20	130779b	MW - F12mw1	0.01497	0.00770	0.00099	0.00794	0.07992	0.01997	0.00788	0.01540	0.06406	0.00053	0.00775	0.21001	0.01334	0.01911	0.00122	0.04747	
pha20	2	22	130780b	MW - F10mw1	0.02945	0.00331	0.00079	0.00561	0.04894	0.02123	0.00747	0.01560	0.05660	0.00050	0.00267	0.23709	0.00683	0.00601	0.00114	0.03758	
pha18c	2	24	130786b	MW - F09mw1	0.01762	0.00883	0.00120	0.00841	0.09538	0.01758	0.00693	0.01968	0.06490	0.00052	0.00931	0.19069	0.01356	0.01971	0.00132	0.04526	
Ustd	2	25	130807b	MW - U STD-2	0.02140	0.00941	0.00178	0.00012	0.09695	0.01454	0.00722	0.02167	0.08964	0.00050	0.00879	0.20215	0.00609	0.02124	0.00010	0.02495	
pha20	2	29	130798b	MW - F10mw2	0.02822	0.00328	0.00074	0.00567	0.04880	0.02139	0.00700	0.01564	0.05476	0.00053	0.00268	0.23846	0.00692	0.00521	0.00110	0.03811	
pha17c	2	30	130794b	MW - F14mw1	0.01532	0.00779	0.00125	0.00621	0.09385	0.01867	0.00615	0.02042	0.05866	0.00050	0.00943	0.20112	0.01379	0.01902	0.00131	0.03893	
Batch 1	2	33	130805b	MW - BATCH 1 STD-3	0.02549	0.00879	0.00073	0.00317	0.09298	0.02101	0.00861	0.01326	0.06740	0.00051	0.00606	0.22352	0.00416	0.00302	0.00077	0.02795	

Values at their detection limits were set to their detection limits. The CST Phase 4 glasses were analyzed with these PHA glasses. Their values are not presented here. See [8] for a complete listing which includes the CST glasses.

Table A.3: Composition of PCT Leachate Solutions

Glass	ID	Blk	Seq	LIMS #	Sample ID	Concentrations in ppm (as reported)					Concentrations in ppm (after correcting for dilution)					Common Logarithm of ppm Concentrations			
						B	Li	Na	Pb	Si	B	Si	Na	Li	Pb	log[B]	log[Si]	log[Na]	log[Li]
std	1	1	1	130358	PCT - Std-b1-1	19.467	9.920	82.739	0.029	48.894	19.467	48.894	82.739	9.920	0.029	1.28930	1.68926	1.91771	0.99651
pha20	1	2	130359		PCT - U15	7.695	7.064	16.958	0.029	43.149	12.825	71.916	28.264	11.774	0.048	1.10807	1.85683	1.45123	1.07091
ARM	1	6	130363		PCT - U25	10.666	8.746	22.974	0.029	36.623	17.777	61.040	38.291	14.377	0.048	1.24986	1.78561	1.58309	1.16367
pha01	1	8	130365		PCT - U70	25.880	18.770	52.431	0.029	98.211	43.134	163.688	87.387	31.284	0.048	1.63482	2.21402	1.94145	1.49532
std	1	9	130366		PCT - Std-b1-2	19.758	10.106	84.244	0.029	49.510	19.758	49.510	84.244	10.106	0.029	1.29574	1.69469	1.92554	1.00458
blank	1	12	130369		PCT - U22	0.040	0.004	0.140	0.029	0.049	0.067	0.082	0.233	0.007	0.048	-1.17608	-1.08795	-0.63201	-2.17608
pha17c	1	15	130372		PCT - U53	18.321	14.002	46.087	0.029	67.873	30.536	113.124	76.813	23.337	0.048	1.48481	2.05355	1.88544	1.36805
pha11c	1	16	130373		PCT - U80	16.104	12.604	36.821	0.029	72.007	26.841	120.014	61.370	21.007	0.048	1.42879	2.07923	1.78795	1.32237
pha05	1	17	130374		PCT - U21	19.500	15.079	40.172	0.029	83.599	32.501	139.335	66.955	25.132	0.048	1.51189	2.14406	1.82578	1.40023
std	1	18	130375		PCT - std-b1-3	19.669	10.002	83.454	0.029	48.968	19.669	48.968	83.454	10.002	0.029	1.29378	1.68991	1.92145	1.00009
std	2	1	130376		STD-B2-1	19.376	9.692	81.811	0.044	49.598	19.376	49.598	81.811	9.692	0.044	1.28726	1.69546	1.91281	0.98641
pha05	2	4	130379		U78	19.103	14.526	39.357	0.060	84.950	31.839	141.586	65.596	24.210	0.100	1.50296	2.15102	1.81688	1.38400
pha01	2	5	130380		U61	25.208	18.027	50.859	0.058	99.572	42.014	165.957	84.767	30.046	0.097	1.62340	2.22000	1.92823	1.47778
blank	2	6	130381		U79	0.046	0.015	0.223	0.033	0.114	0.077	0.190	0.372	0.025	0.055	-1.11538	-0.72124	-0.42984	-1.60205
pha17c	2	8	130383		U71	16.346	12.096	41.316	0.054	68.290	27.244	113.819	68.861	20.160	0.090	1.43527	2.05622	1.83798	1.30450
std	2	9	130384		STD-B2-2	19.467	9.705	81.562	0.046	49.333	19.467	49.333	81.562	9.705	0.046	1.28930	1.69314	1.91149	0.98700
ARM	2	11	130386		U31	10.834	8.533	22.828	0.056	37.276	18.057	62.128	38.047	14.222	0.093	1.25665	1.79329	1.58033	1.15296
pha11c	2	13	130388		U56	16.630	12.671	37.466	0.063	73.560	27.717	122.603	62.445	21.119	0.105	1.44275	2.08850	1.79550	1.32467
pha20	2	15	130390		U39	8.791	7.793	18.940	0.056	47.755	14.652	79.593	31.567	12.989	0.093	1.16590	1.90088	1.49924	1.11356
std	2	18	130393		STD-B2-3	19.419	9.658	81.390	0.049	49.189	19.419	49.189	81.390	9.658	0.049	1.28823	1.69187	1.91057	0.98489
std	3	1	130394		STD-B3-1	19.113	9.522	80.567	0.047	48.760	19.113	48.760	80.567	9.522	0.047	1.28133	1.68806	1.90616	0.97873
pha05	3	2	130395		U18	19.146	14.629	39.472	0.060	84.914	31.911	141.526	65.788	24.382	0.100	1.50394	2.15084	1.81815	1.38707
pha01	3	5	130398		U60	24.418	17.562	49.704	0.049	96.916	40.697	161.530	82.842	29.271	0.082	1.60957	2.20825	1.91825	1.46643
pha20	3	6	130399		U72	7.776	7.025	16.932	0.055	44.519	12.960	74.200	28.221	11.709	0.092	1.11261	1.87040	1.45057	1.06850
std	3	9	130402		STD-B3-2	19.265	9.648	81.451	0.053	49.003	19.265	49.003	81.451	9.648	0.053	1.28477	1.69022	1.91090	0.98444
pha17c	3	11	130404		U81	17.668	13.075	44.236	0.045	68.667	29.447	114.447	73.728	21.792	0.075	1.46905	2.05861	1.86763	1.33830
pha11c	3	13	130406		U26	15.094	11.763	35.089	0.051	70.979	25.157	118.301	58.483	19.605	0.085	1.40066	2.07299	1.76703	1.29238
ARM	3	15	130408		U49	10.834	8.521	22.911	0.043	36.743	18.057	61.240	38.186	14.202	0.072	1.25665	1.78703	1.58190	1.15235
std	3	17	130410		STD-B3-3	19.339	9.711	82.099	0.035	49.310	19.339	49.310	82.099	9.711	0.035	1.28643	1.69294	1.91434	0.98726
std	4	1	130411		STD-B4-1	19.165	9.690	81.752	0.042	48.956	19.165	48.956	81.752	9.690	0.042	1.28251	1.68981	1.91250	0.98632
pha03	4	2	130412		PCT - U24	30.246	19.709	57.031	0.045	96.535	50.411	160.895	95.054	32.849	0.075	1.70253	2.20654	1.97797	1.51652
pha04	4	3	130413		PCT - U55	25.802	20.743	53.426	0.089	113.181	43.004	188.639	89.045	34.572	0.148	1.63351	2.27563	1.94961	1.53873
pha06	4	5	130415		PCT - U29	26.071	17.434	51.640	0.029	89.886	43.453	149.813	86.068	29.057	0.048	1.63802	2.17555	1.93484	1.46325
pha18c	4	8	130418		PCT - U23	26.878	15.967	58.565	0.032	74.546	44.798	124.246	97.610	26.612	0.053	1.65125	2.09428	1.98950	1.42508
std	4	9	130419		PCT - STD-B4-2	19.377	9.759	82.363	0.029	49.126	19.377	49.126	82.363	9.759	0.029	1.28729	1.69131	1.91573	0.98941
pha26	4	11	130421		PCT - U62	6.956	6.290	16.621	0.030	41.026	11.594	68.378	27.702	10.484	0.050	1.06422	1.83492	1.44252	1.02051
EA	4	13	130423		PCT - U07	38.600	12.142	111.398	0.039	57.005	643.346	950.102	1856.670	202.371	0.065	2.80845	2.97777	3.26874	2.30615
pha02	4	14	130424		PCT - U36	19.714	15.355	41.745	0.038	85.842	32.857	143.073	69.576	25.592	0.063	1.51663	2.15556	1.84246	1.40811
pha12c	4	15	130425		PCT - U75	28.647	18.380	58.580	0.041	88.212	47.746	147.023	97.635	30.634	0.068	1.67894	2.16739	1.98961	1.48620
pha32	4	16	130426		PCT - U74	6.784	6.091	17.489	0.052	39.855	11.307	66.426	29.149	10.152	0.087	1.05334	1.82234	1.46462	1.00655
std	4	17	130427		PCT - STD-B4-3	19.309	9.719	82.229	0.034	49.077	19.309	49.077	82.229	9.719	0.034	1.28576	1.69088	1.91503	0.98762

Notes:

- (1). Values that are below detection (indicated by a "<") were converted to their detection limit.
- (2) The CST Phase 3 and PHA Phases 3 and 4 glasses are also shown in this table (since they were part of the analytical plan). However, no analyses of these data are included in this report.

Table A.3: Composition of PCT Leachate Solutions
(continued)

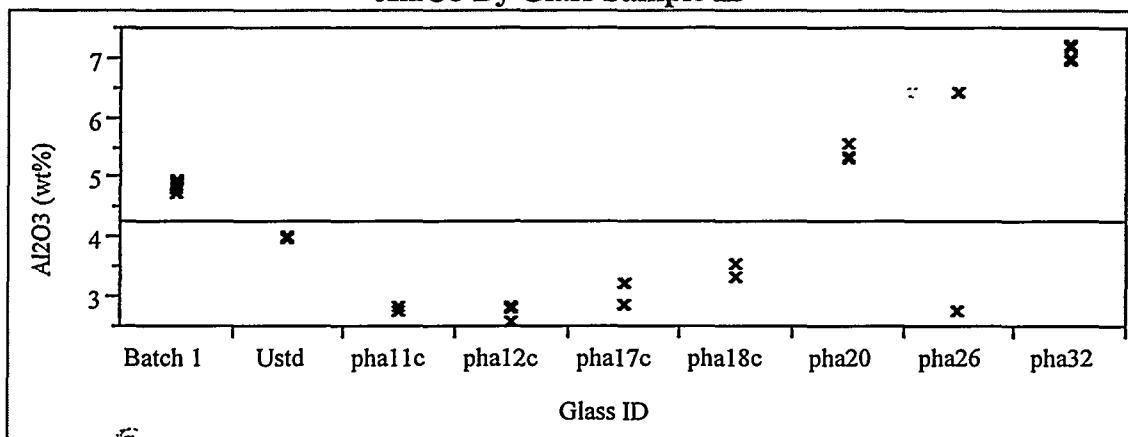
Glass ID	Blk	Seq	LIMS #	Sample ID	Concentrations in ppm (as reported)						Concentrations in ppm (after correcting for dilution)						Common Logarithm of ppm Concentrations			
					B	Li	Na	Pb	Si	B	Si	Na	Li	Pb	log[B]	log[Li]	log[Na]	log[Pb]		
std	5	1	130428	PCT - STD-B5-1	19.670	10.009	83.367	0.029	49.025	19.670	49.025	83.367	10.009	0.029	1.293380	1.69042	1.92099	1.00039		
phia03	5	2	130429	PCT - U19	30.785	20.231	57.501	0.029	96.080	51.309	160.157	95.837	33.719	0.048	1.71020	2.20449	1.10964	1.9853	1.522788	
phia26	5	3	130430	PCT - U63	7.723	6.989	17.924	0.030	43.292	12.872	72.155	29.874	11.649	0.050	1.85827	1.47529	1.06627		1.42200	
phia02	5	4	130431	PCT - U46	20.360	15.854	42.292	0.029	83.835	33.994	139.728	70.488	26.424	0.048	1.53064	2.14528	1.84812			
phia18c	5	5	130432	PCT - U14	29.831	17.363	64.045	0.029	78.043	49.719	130.074	106.744	29.222	0.048	1.69653	2.11419	2.02834			
phia12	5	6	130433	PCT - U40	6.981	6.327	18.006	0.029	40.552	11.635	67.388	30.011	10.545	0.048	1.06578	1.82987	1.47728		1.02306	
phia04	5	8	130435	PCT - U10	25.970	21.137	54.134	0.029	114.372	43.284	190.624	90.225	35.229	0.048	1.63633	2.28018	1.95533		1.54690	
std	5	9	130436	PCT - STD-B5-2	19.787	10.053	83.700	0.029	48.985	19.787	48.985	83.700	10.053	0.029	1.29638	1.69006	1.92273		1.00230	
phia12c	5	12	130439	PCT - U09	23.641	15.633	49.987	0.029	78.468	39.402	130.783	83.313	26.036	0.048	1.59552	2.11655	1.92072		1.41590	
phia06	5	14	130441	PCT - U69	26.835	18.083	52.672	0.029	90.100	44.726	150.170	87.788	30.139	0.048	1.65036	2.17638	1.94344			
EA	5	16	130443	PCT - U77	43.471	14.619	123.358	0.029	63.060	724.531	103.1021	243.655	0.048	2.86006	3.02161	3.31303		2.38678		
blank	5	17	130444	PCT - U88	0.099	0.013	0.188	0.029	0.087	0.165	0.145	0.313	0.022	0.048	-0.78251	-0.83862	-0.50398		-1.66420	
std	5	18	130445	PCT - STD-B5-3	19.762	10.069	83.741	0.029	48.953	19.762	48.953	83.741	10.069	0.029	1.29533	1.68978	1.92294		1.00299	
phia04	6	1	130446	PCT - STD-B6-1	19.528	9.889	82.971	0.029	48.559	19.528	48.559	82.971	9.989	0.029	1.29066	1.68637	1.91893		0.99552	
phia32	6	3	130448	PCT - U44	24.807	20.383	51.824	0.029	110.133	41.346	183.559	86.375	33.972	0.048	1.61643	2.26378	1.93639			
EA	6	4	130449	PCT - U30	6.980	6.354	18.030	0.029	40.017	11.634	66.696	30.051	10.590	0.048	1.06571	1.82410	1.47785		1.31113	
phia06	6	5	130450	PCT - U58	39.043	12.446	111.786	0.029	56.382	650.730	941.386	1863.137	207.428	0.048	2.81340	2.97377	3.27025		2.31689	
phia26	6	6	130451	PCT - U68	27.686	18.652	54.244	0.029	91.908	46.144	153.183	90.408	31.087	0.048	1.66412	2.18321	1.92621		1.49258	
std	6	9	130453	PCT - U84	7.185	6.532	16.821	0.029	41.306	11.975	68.845	28.036	10.887	0.048	1.07828	1.83787	1.44771		1.03690	
phia12c	6	11	130454	PCT - STD-B6-2	19.632	10.025	83.330	0.029	48.630	19.632	83.330	10.025	0.029	1.29297	1.68690	1.92080				
phia02	6	13	130455	PCT - U50	5.797	4.220	12.956	0.029	20.378	21.594	7.033	0.048	0.98806	1.53102	1.33433		1.02491			
phia18c	6	14	130459	PCT - U34	20.470	16.126	43.177	0.029	86.547	34.117	144.248	71.963	26.877	0.048	1.53298	1.85711	1.42938			
phia03	6	15	130460	PCT - U01	31.304	18.573	66.733	0.029	79.072	52.174	131.789	111.224	30.956	0.048	1.71746	2.11988	2.04620		1.49074	
blank	6	17	130462	PCT - U87	0.061	0.006	0.332	0.029	0.052	0.102	0.087	0.553	0.010	0.048	1.71442	2.20566	1.98339		1.53181	
std	6	18	130463	PCT - STD-B6-3	19.707	10.052	83.703	0.029	48.974	19.707	48.974	83.703	10.052	0.029	1.29462	1.68897	1.92274		1.00225	

Notes:

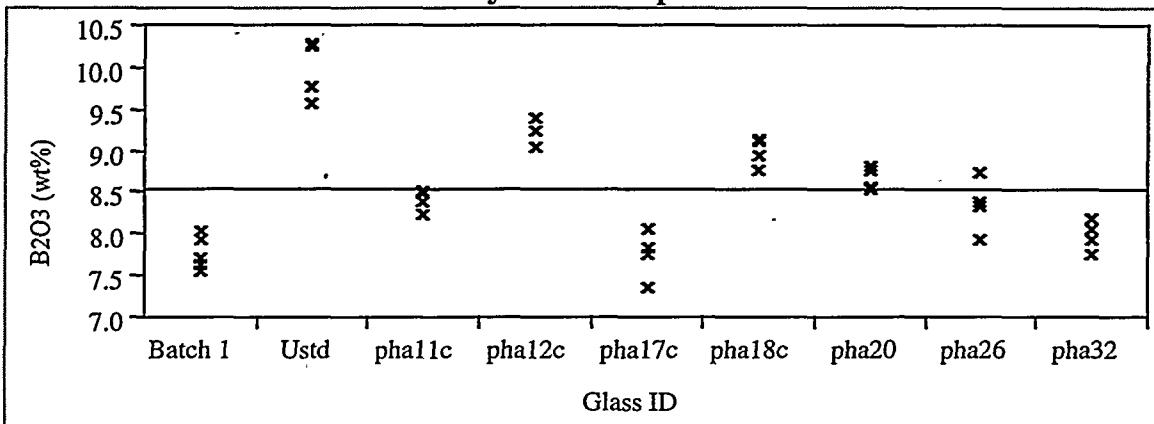
- (1). Values that are below detection (indicated by a "<") were converted to their detection limit.
- (2). The CST Phase 3 and PHA Phases 3 and 4 glasses are also shown in this table (since they were part of the analytical plan). However, no analyses of these data are included in this report.

Exhibit A.1: Measurements by Glass Sample ID by Oxide

Al₂O₃ By Glass Sample ID



B₂O₃ By Glass Sample ID



CaO MW By Glass Sample ID

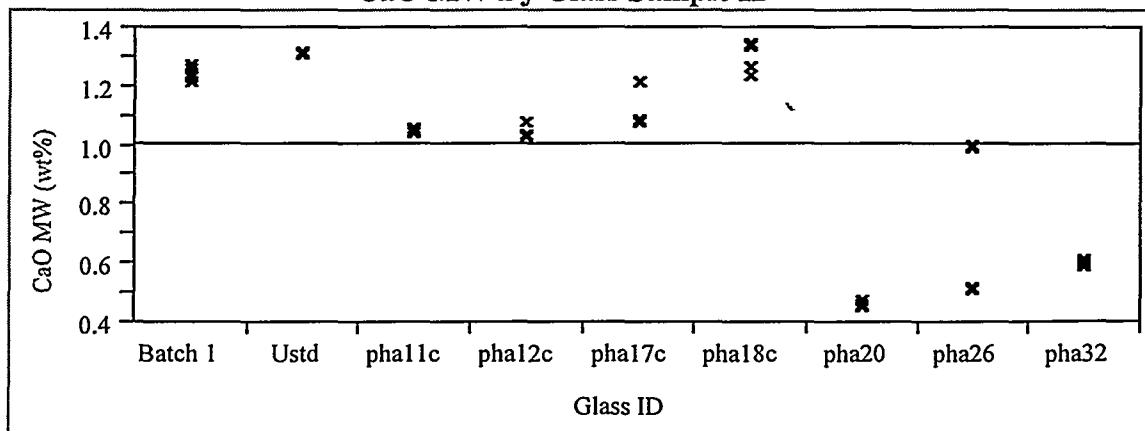
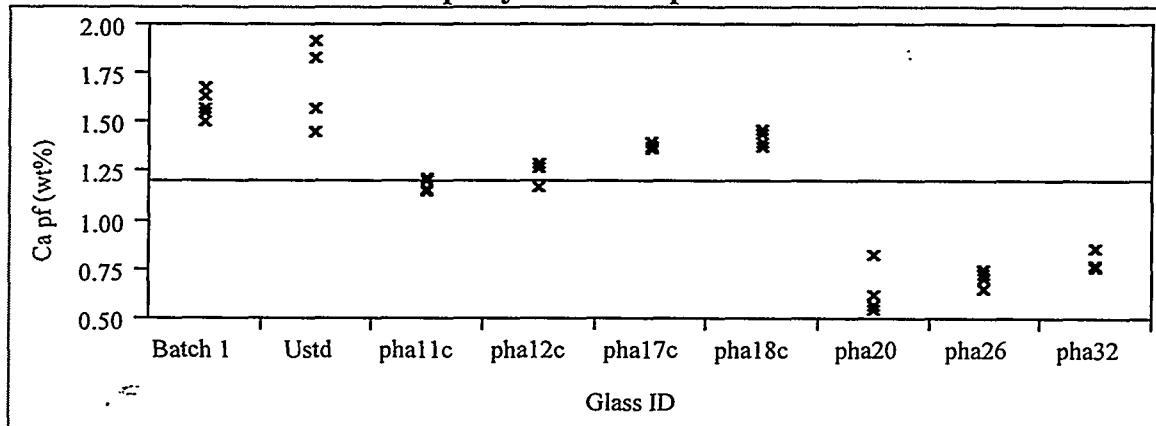
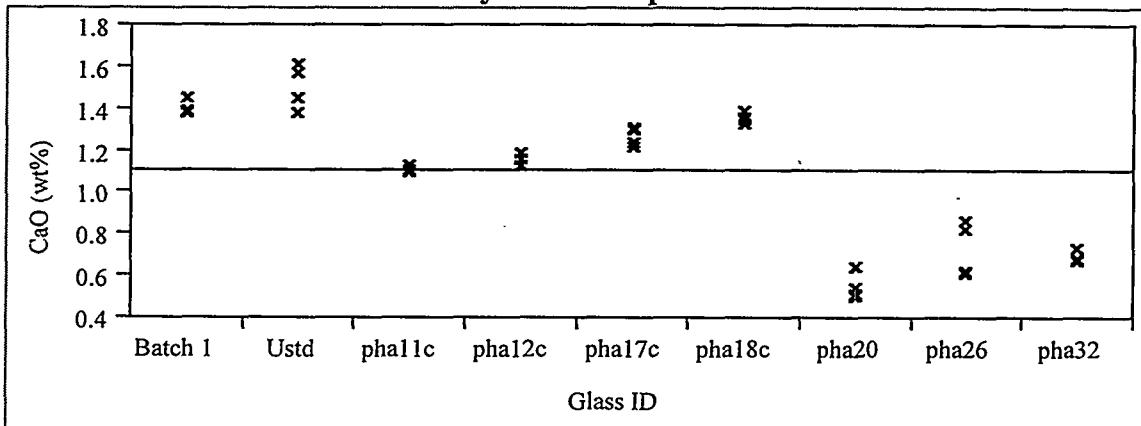


Exhibit A.1: Measurements by Glass Sample ID by Oxide
 (continued)

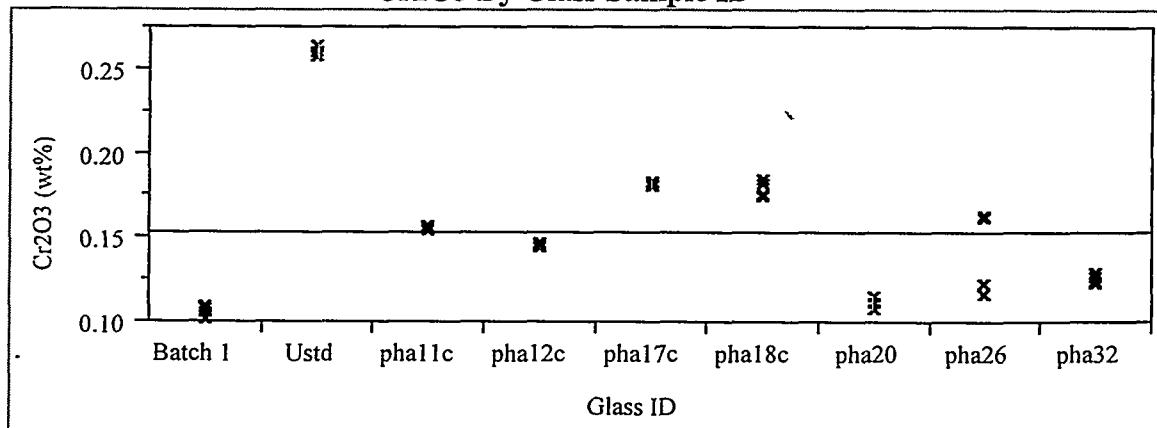
CaO pf By Glass Sample ID



CaO By Glass Sample ID

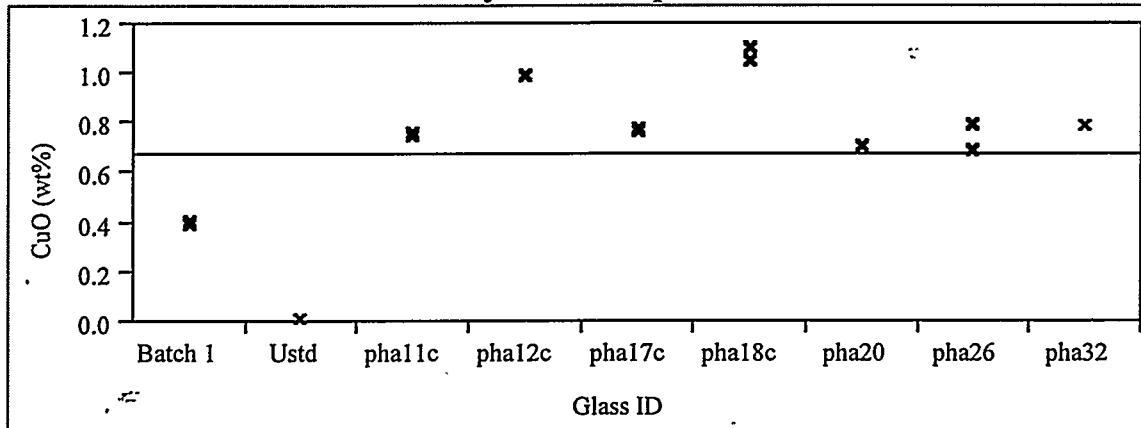


Cr₂O₃ By Glass Sample ID

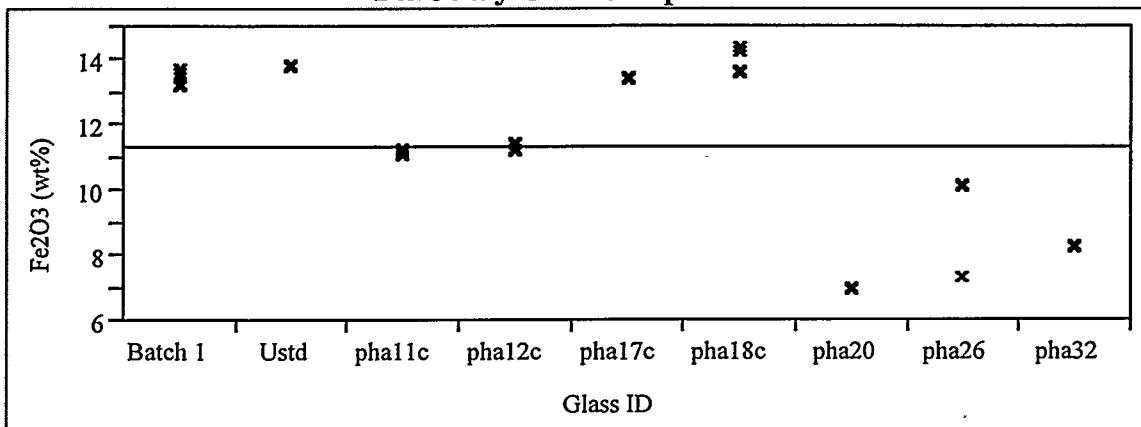


**Exhibit A.1: Measurements by Glass Sample ID by Oxide
(continued)**

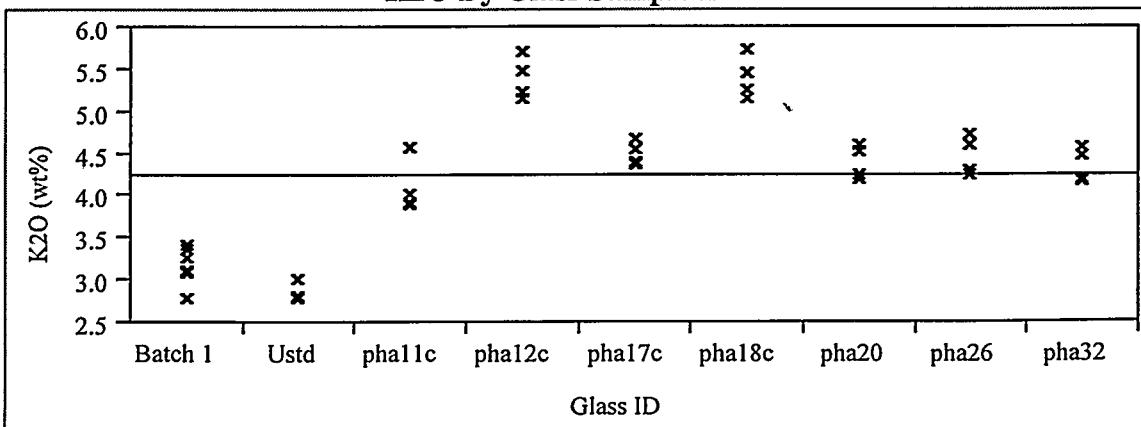
CuO By Glass Sample ID



Fe₂O₃ By Glass Sample ID

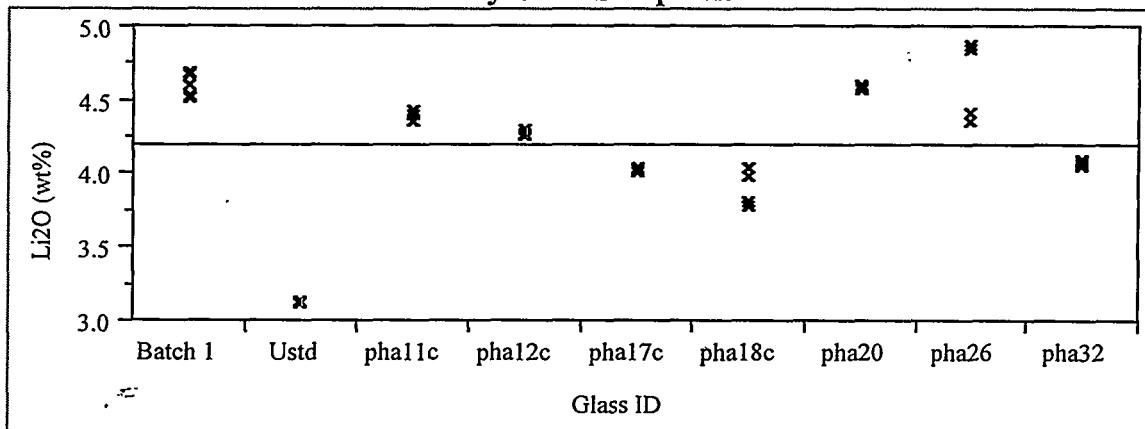


K₂O By Glass Sample ID

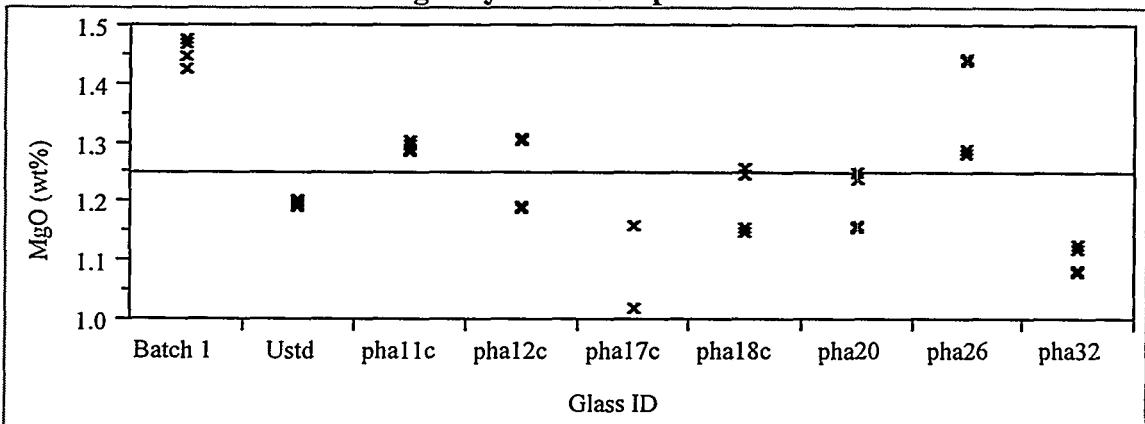


**Exhibit A.1: Measurements by Glass Sample ID by Oxide
(continued)**

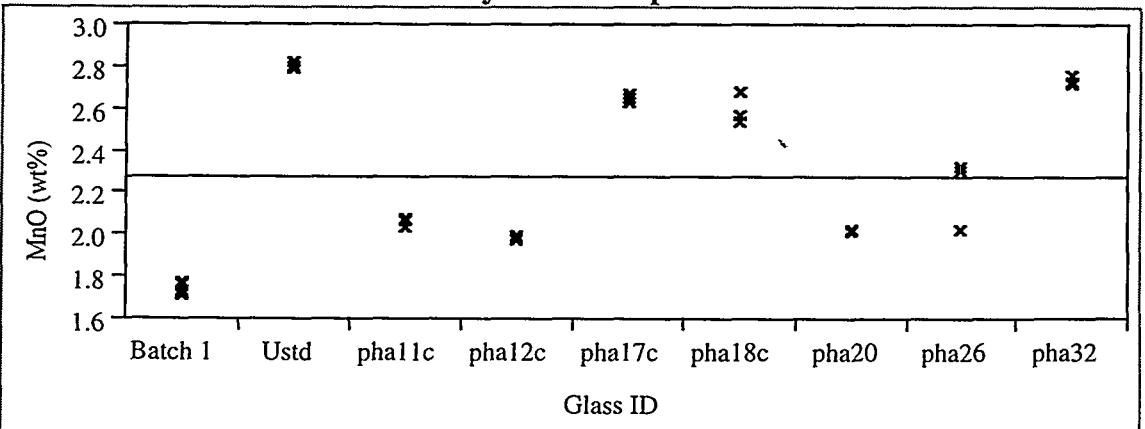
Li₂O By Glass Sample ID



MgO By Glass Sample ID

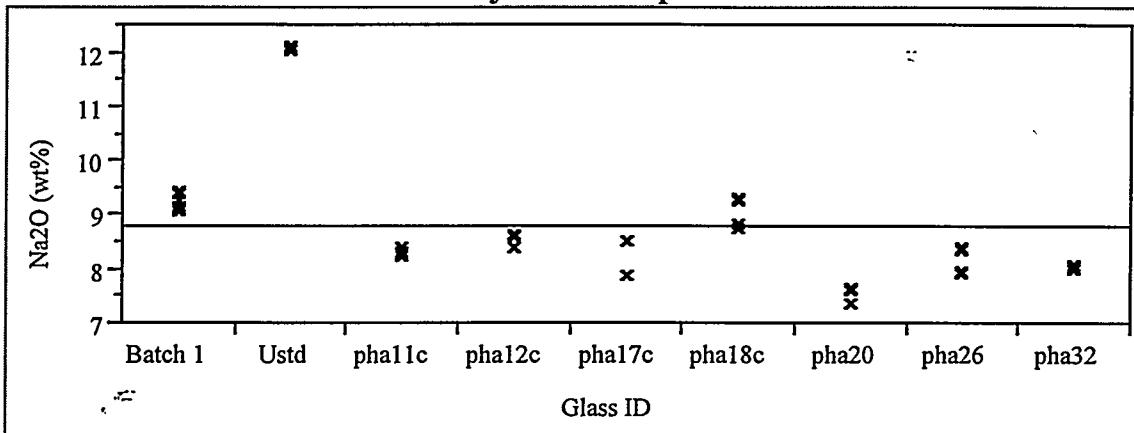


MnO By Glass Sample ID

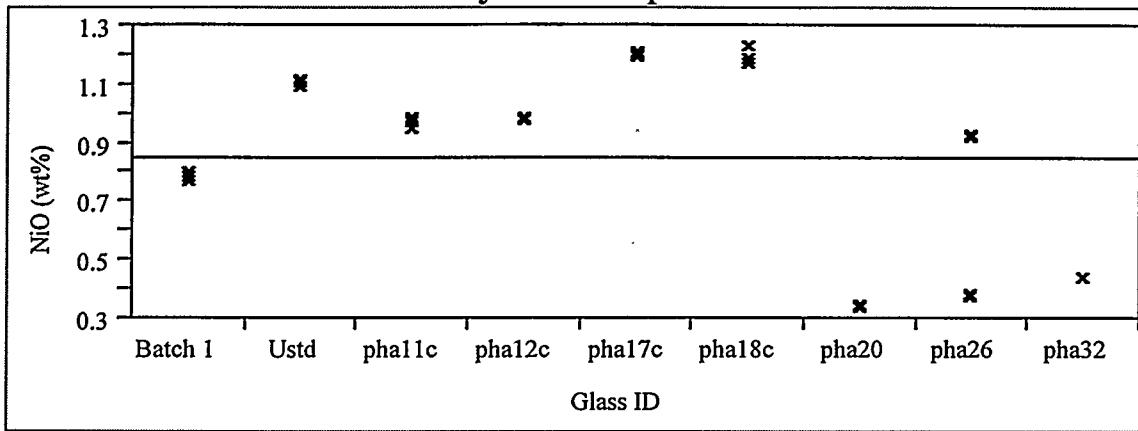


**Exhibit A.1: Measurements by Glass Sample ID by Oxide
(continued)**

Na₂O By Glass Sample ID



NiO By Glass Sample ID



SiO₂ MW By Glass Sample ID

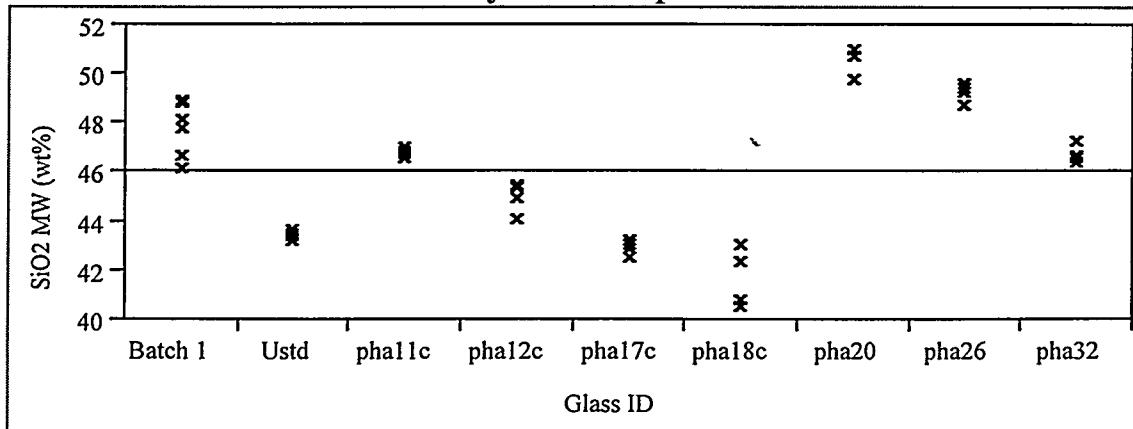
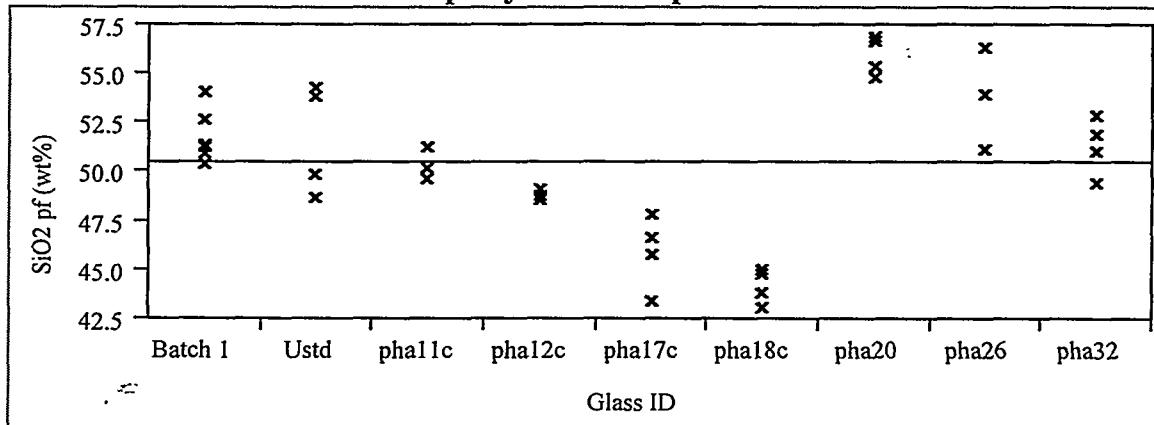
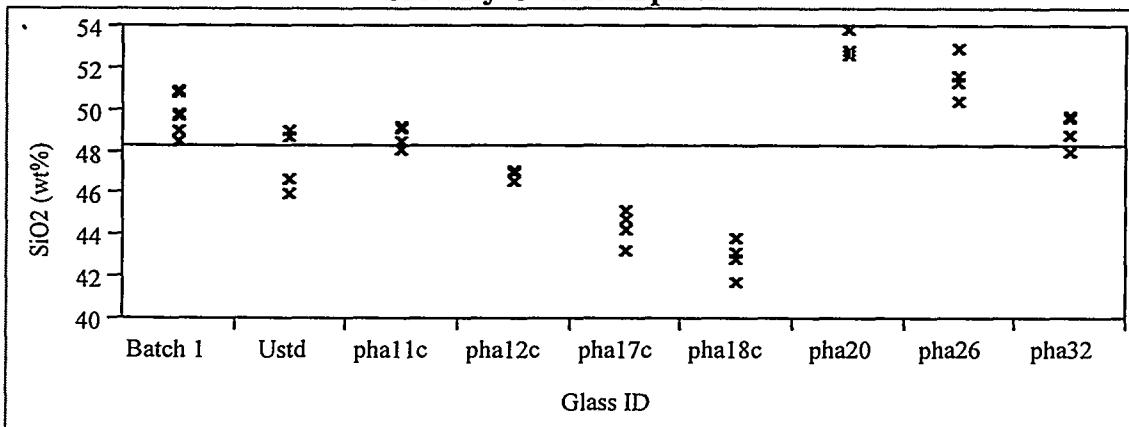


Exhibit A.1: Measurements by Glass Sample ID by Oxide
(continued)

SiO₂ pf By Glass Sample ID



SiO₂ By Glass Sample ID



TiO₂ By Glass Sample ID

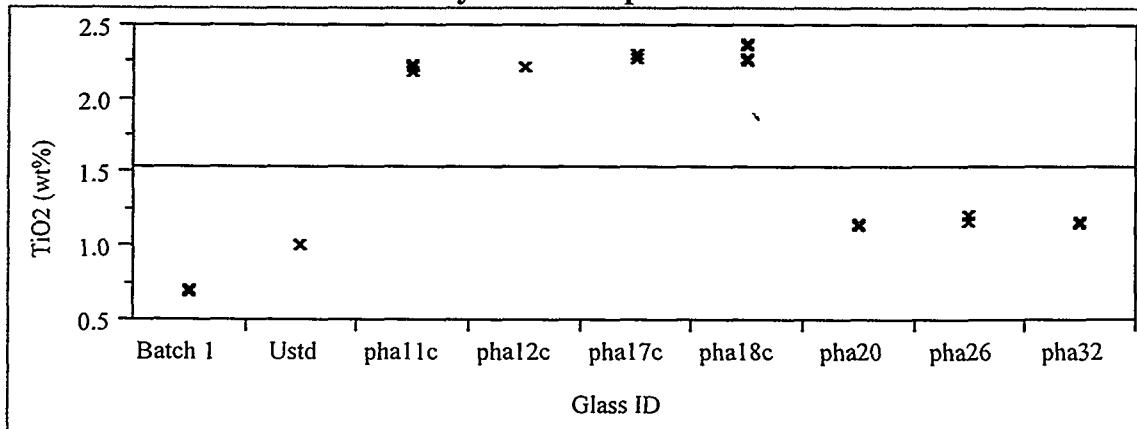
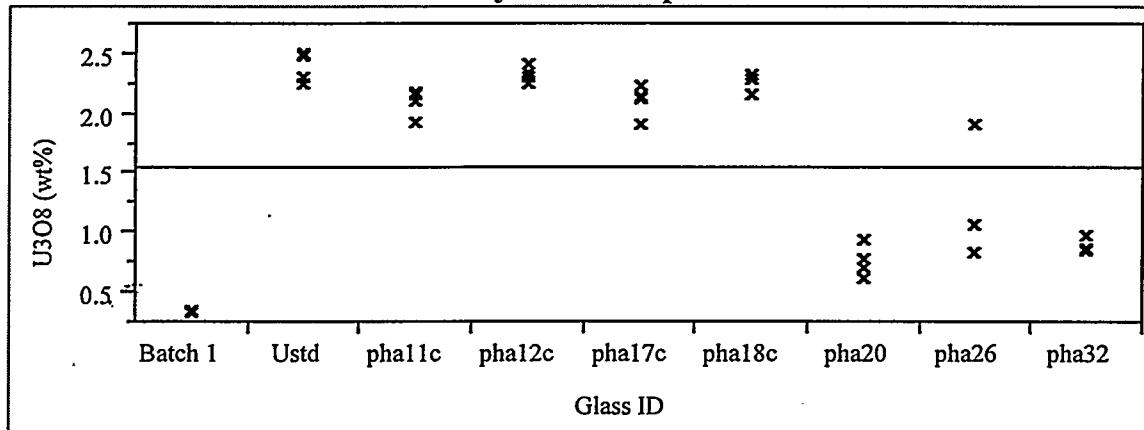
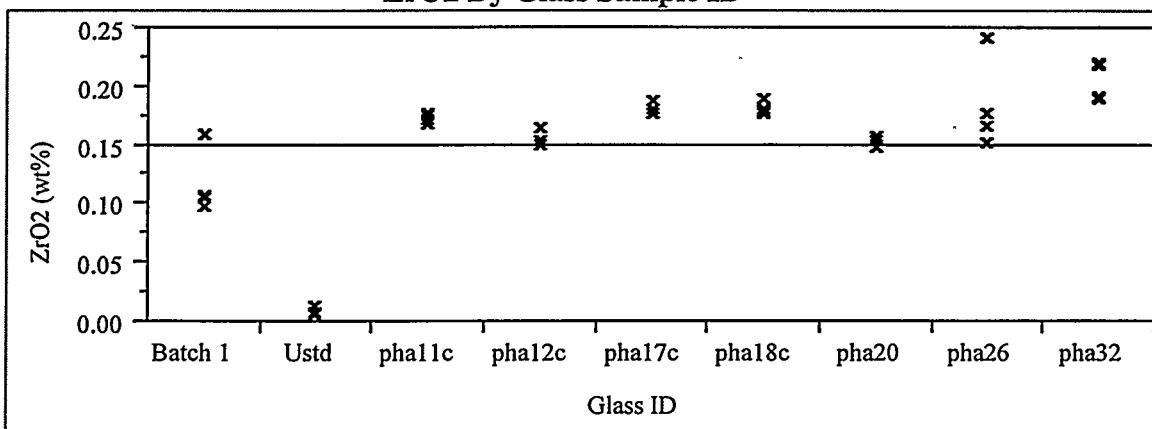


Exhibit A.1: Measurements by Glass Sample ID by Oxide
 (continued)

U₃O₈ By Glass Sample ID



ZrO₂ By Glass Sample ID



Sum of Oxides By Glass Sample ID

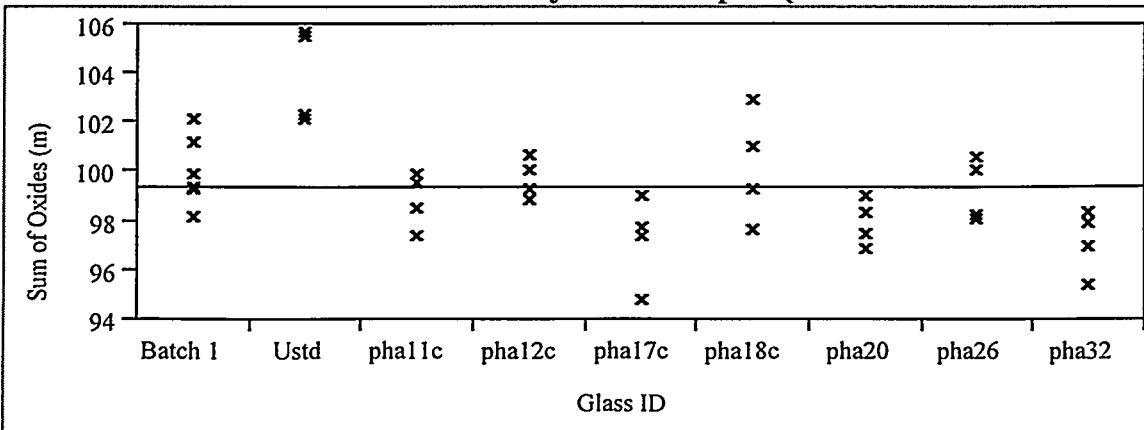
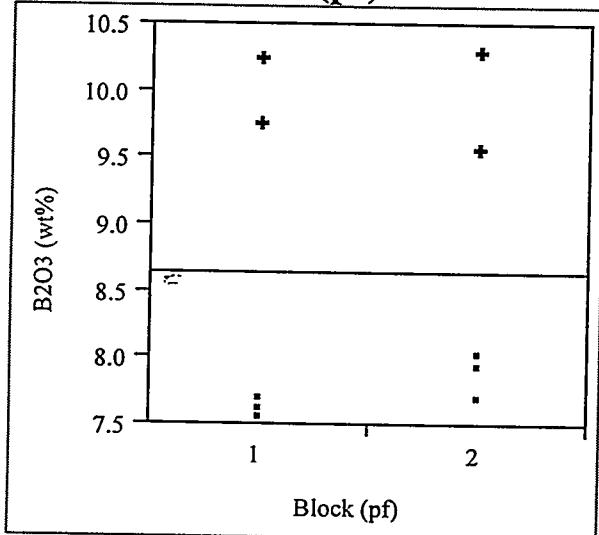
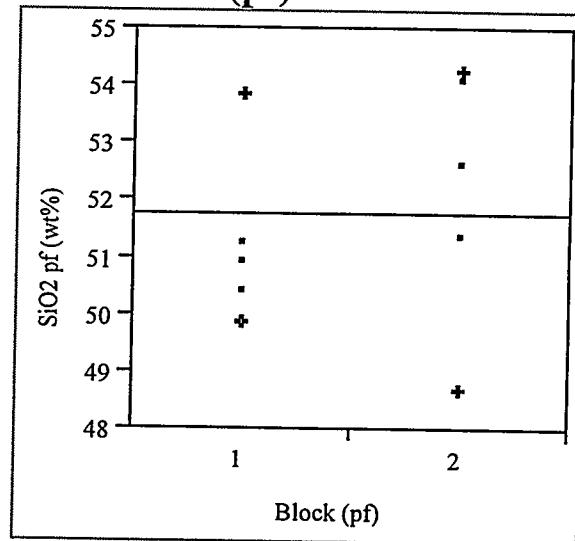


Exhibit A.2: Peroxide Fusion Measurements of Glass Standards by Oxide
(+ u-std; small square Batch 1 standard)

B₂O₃ (wt%) By Block (pf)



SiO₂ pf (wt%) By Block (pf)



Ca pf (wt%) By Block (pf)

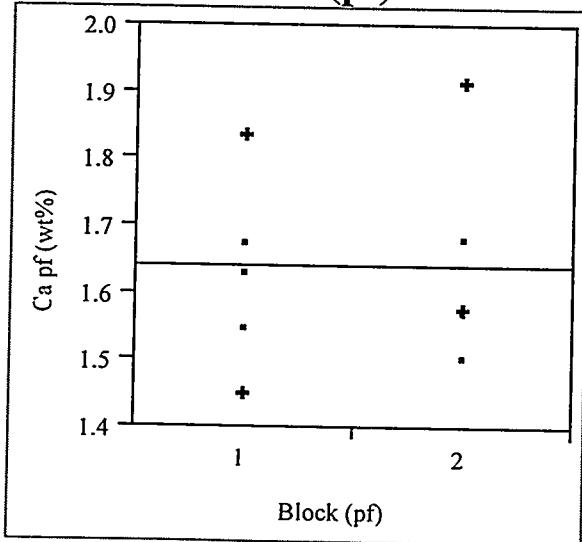
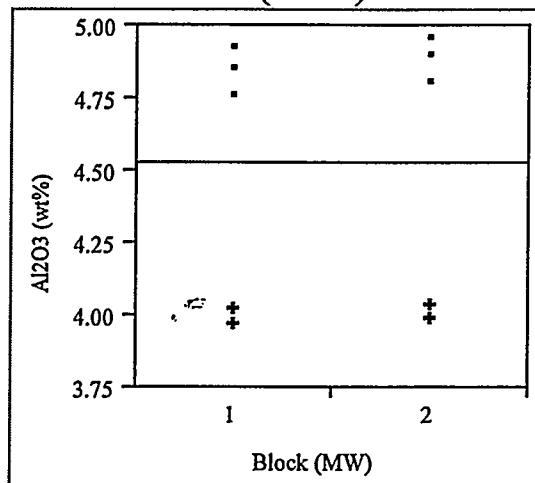
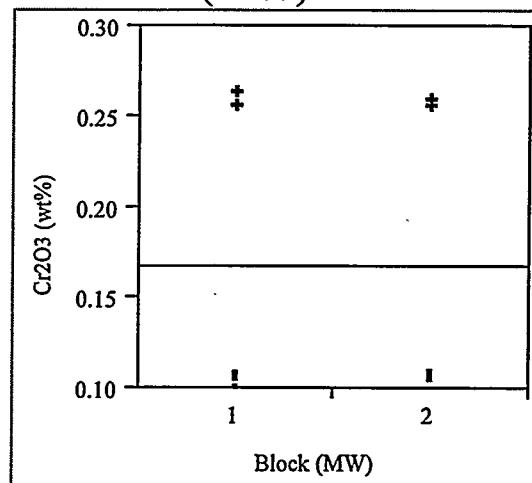


Exhibit A.3: Measurements of Glass Standards by Oxide
(+ u-std; small square Batch 1 standard)

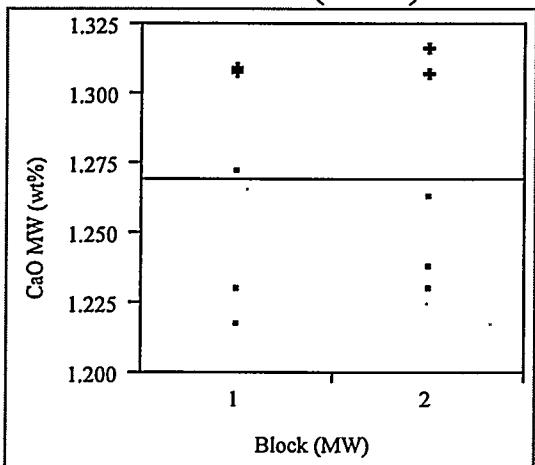
Al₂O₃ (wt%) By Block (MW)



Cr₂O₃ (wt%) By Block (MW)



CaO MW (wt%) By Block (MW)



CuO (wt%) By Block (MW)

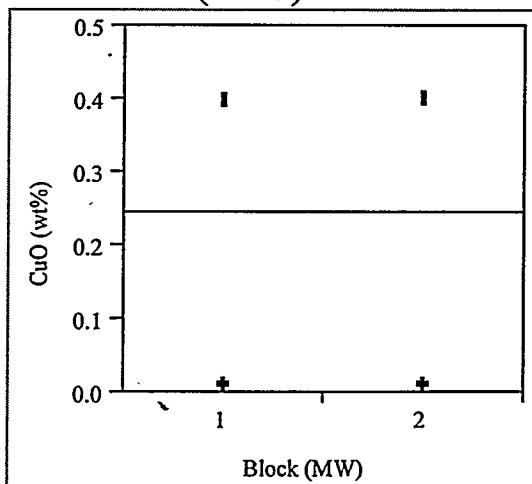
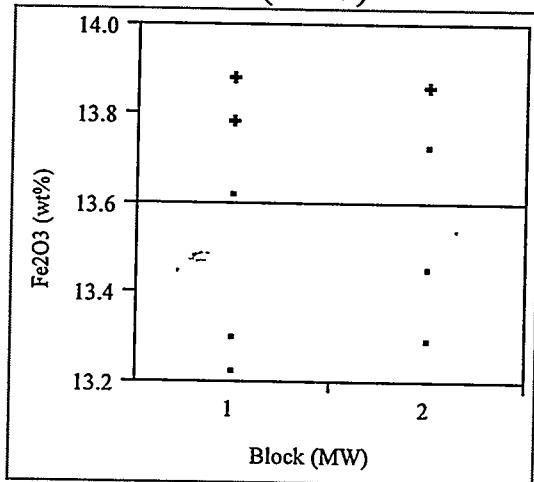
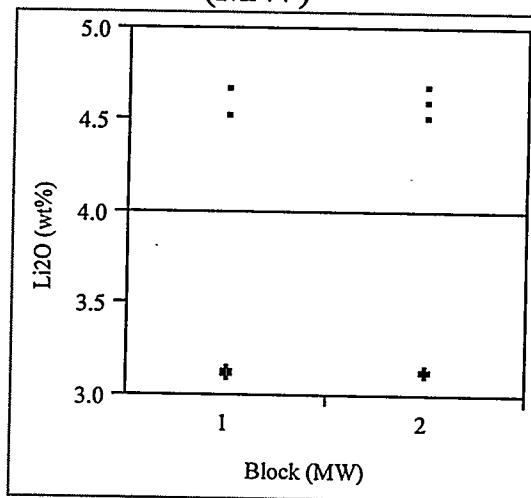


Exhibit A.3: Measurements of Glass Standards by Oxide
(+ u-std; small square Batch 1 standard)
(continued)

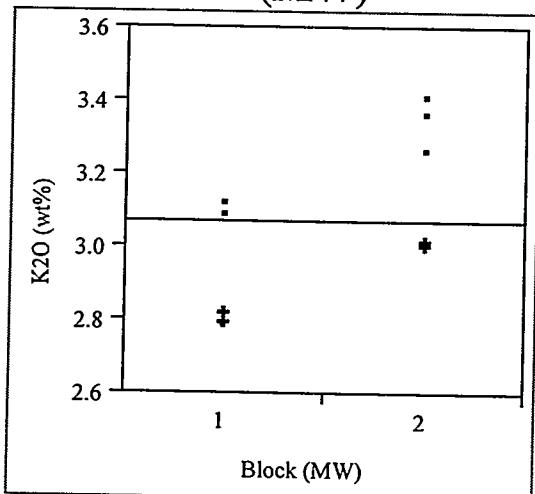
Fe₂O₃ (wt%) By Block (MW)



Li₂O (wt%) By Block (MW)



K₂O (wt%) By Block (MW)



MgO (wt%) By Block (MW)

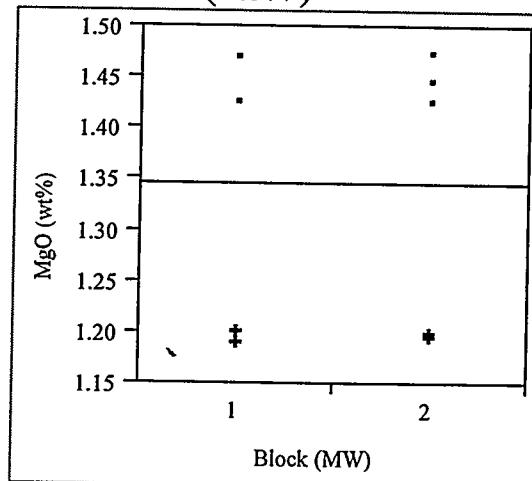


Exhibit A.3: Measurements of Glass Standards by Oxide

(+ u-std; small square Batch 1 standard)

(continued)

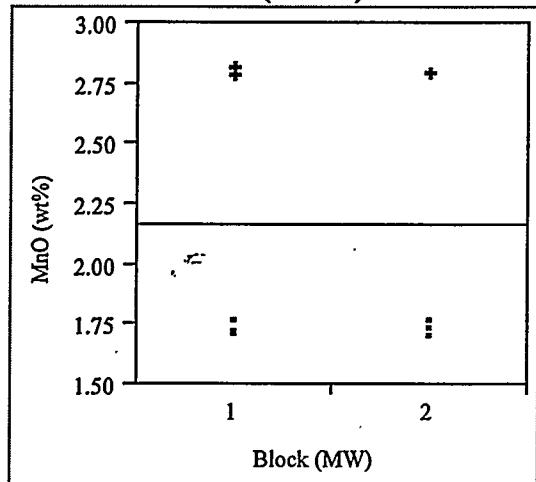
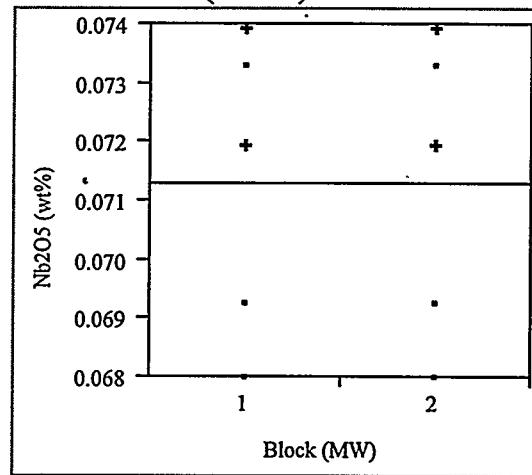
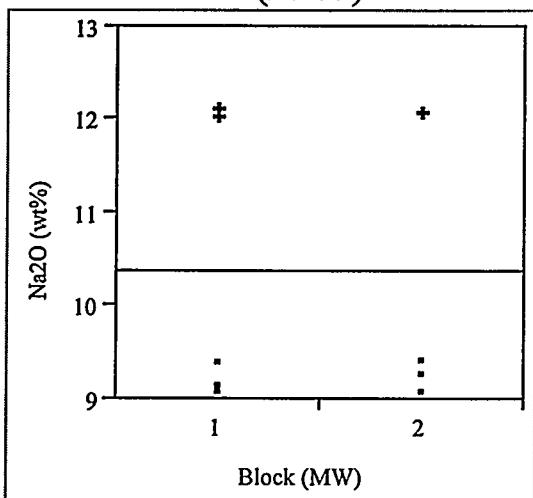
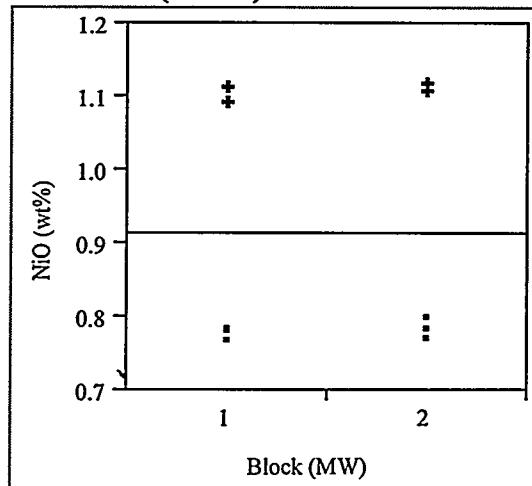
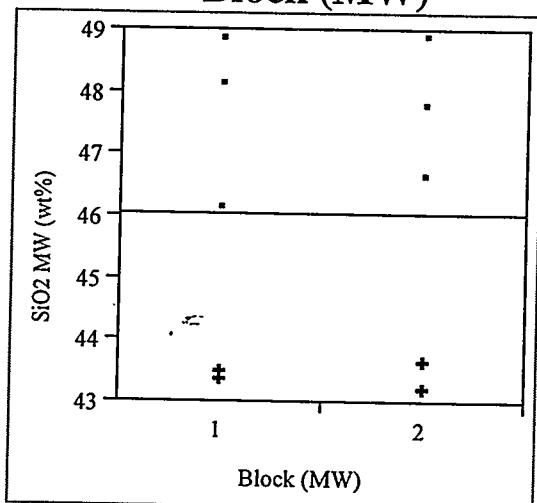
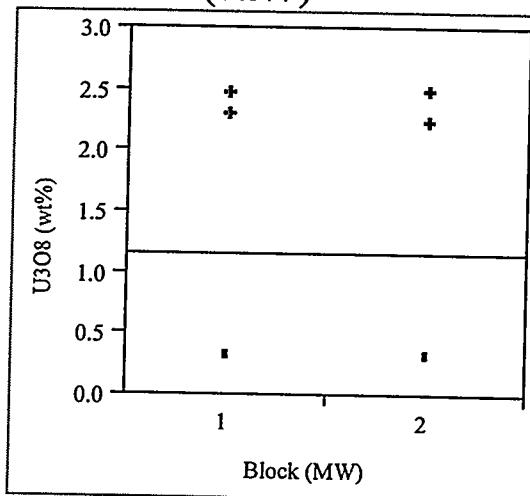
MnO (wt%) By Block (MW)**Nb₂O₅ (wt%) By Block (MW)****Na₂O (wt%) By Block (MW)****NiO (wt%) By Block (MW)**

Exhibit A.3: Measurements of Glass Standards by Oxide
 (+ u-std; small square Batch 1 standard)
 (continued)

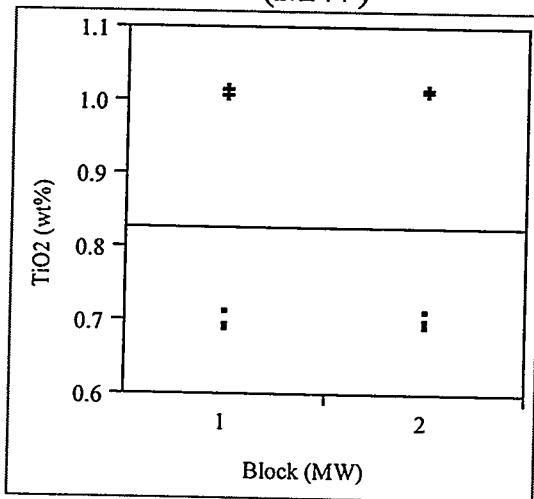
SiO₂ MW (wt%) By Block (MW)



U₃O₈ (wt%) By Block (MW)



TiO₂ (wt%) By Block (MW)



ZrO₂ (wt%) By Block (MW)

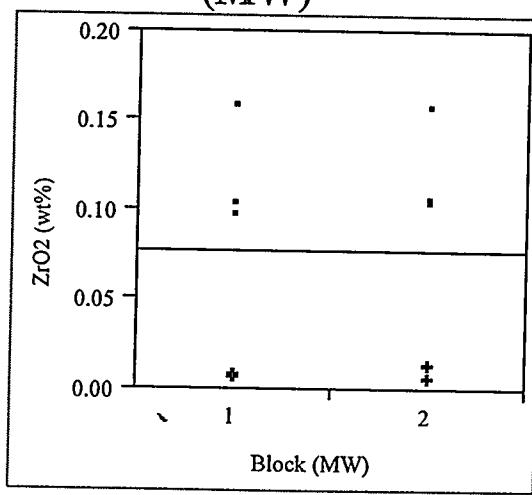


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

Al₂O₃

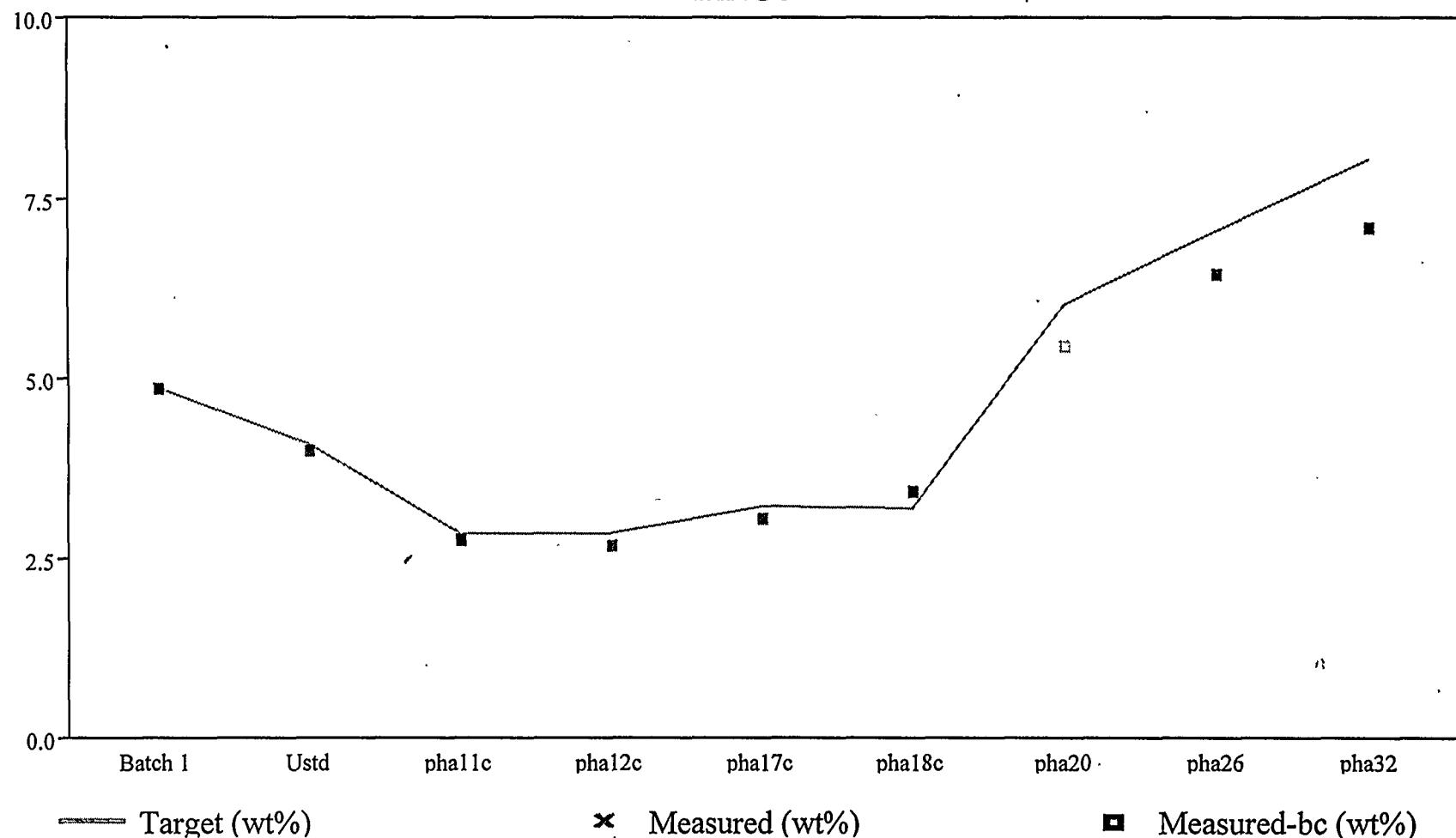


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

B2O3

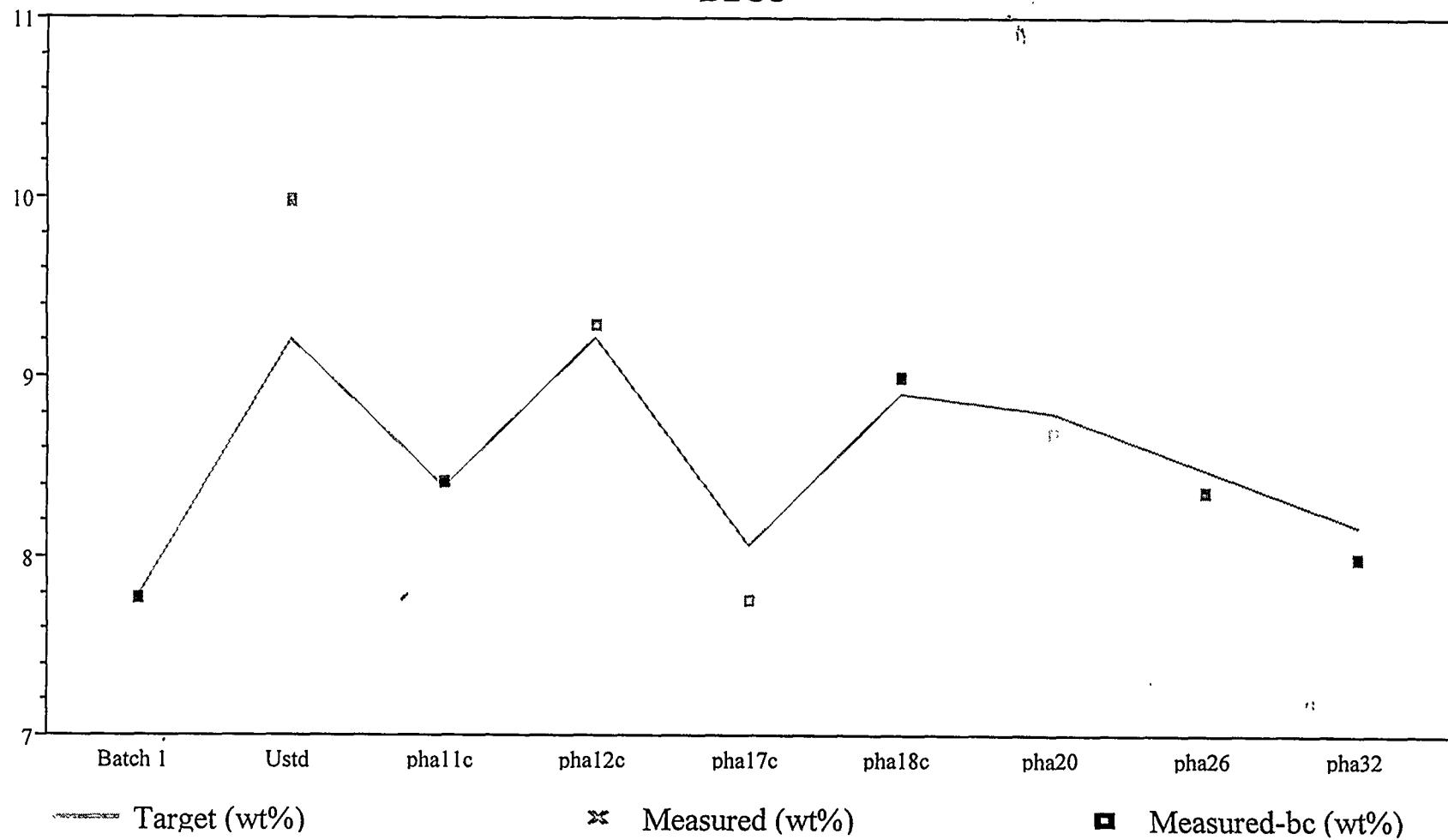


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

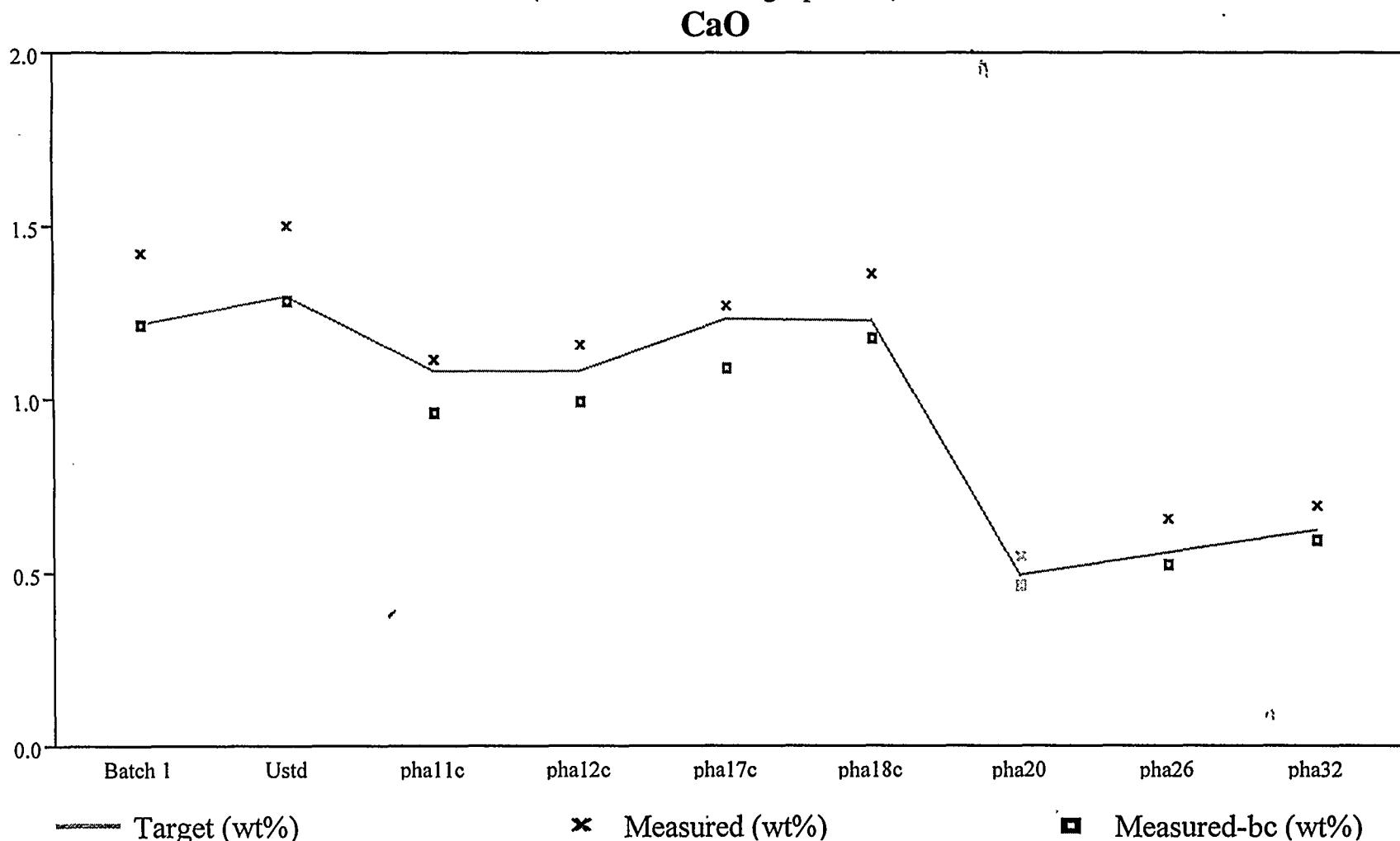


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

Cr₂O₃

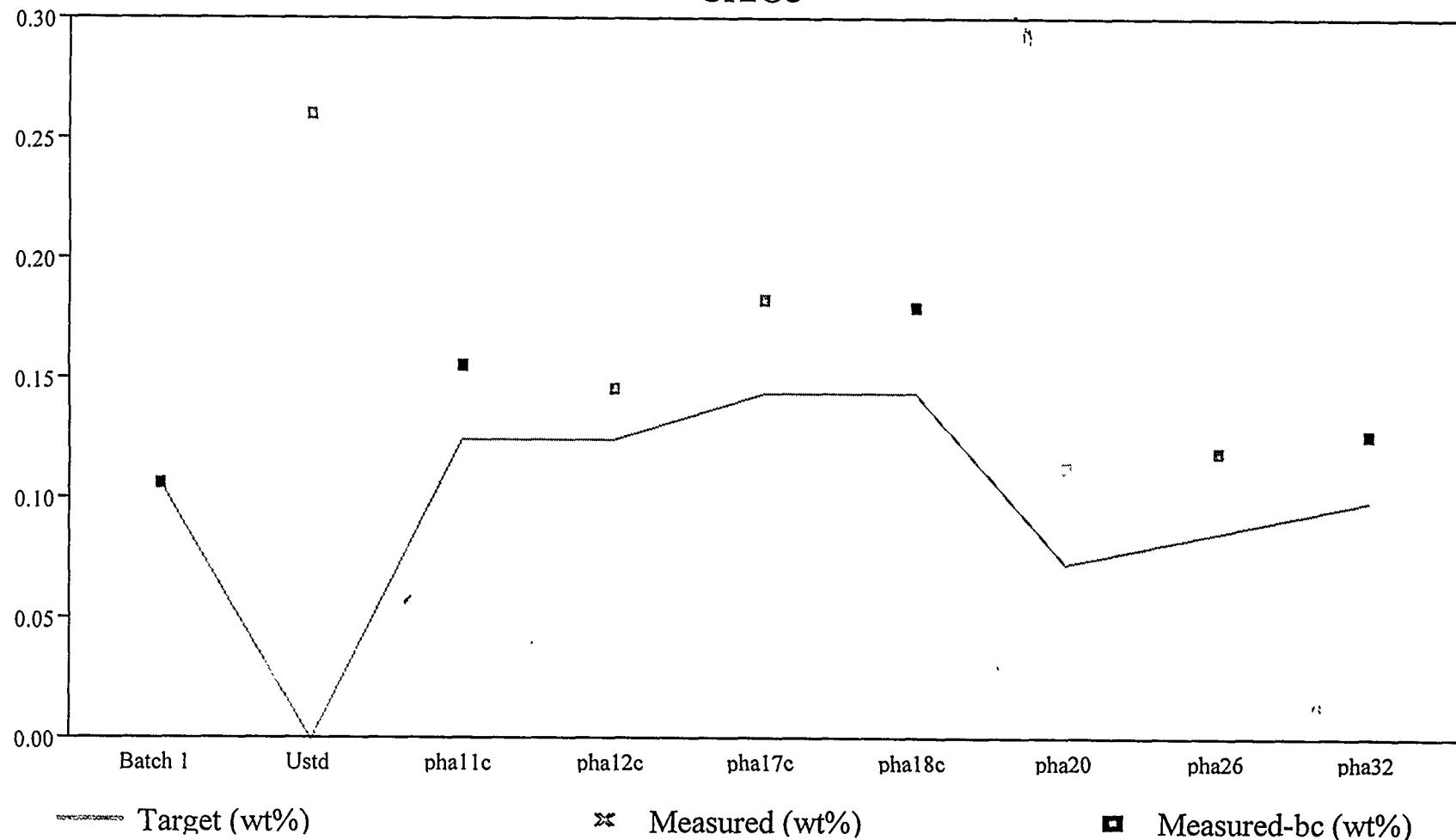


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

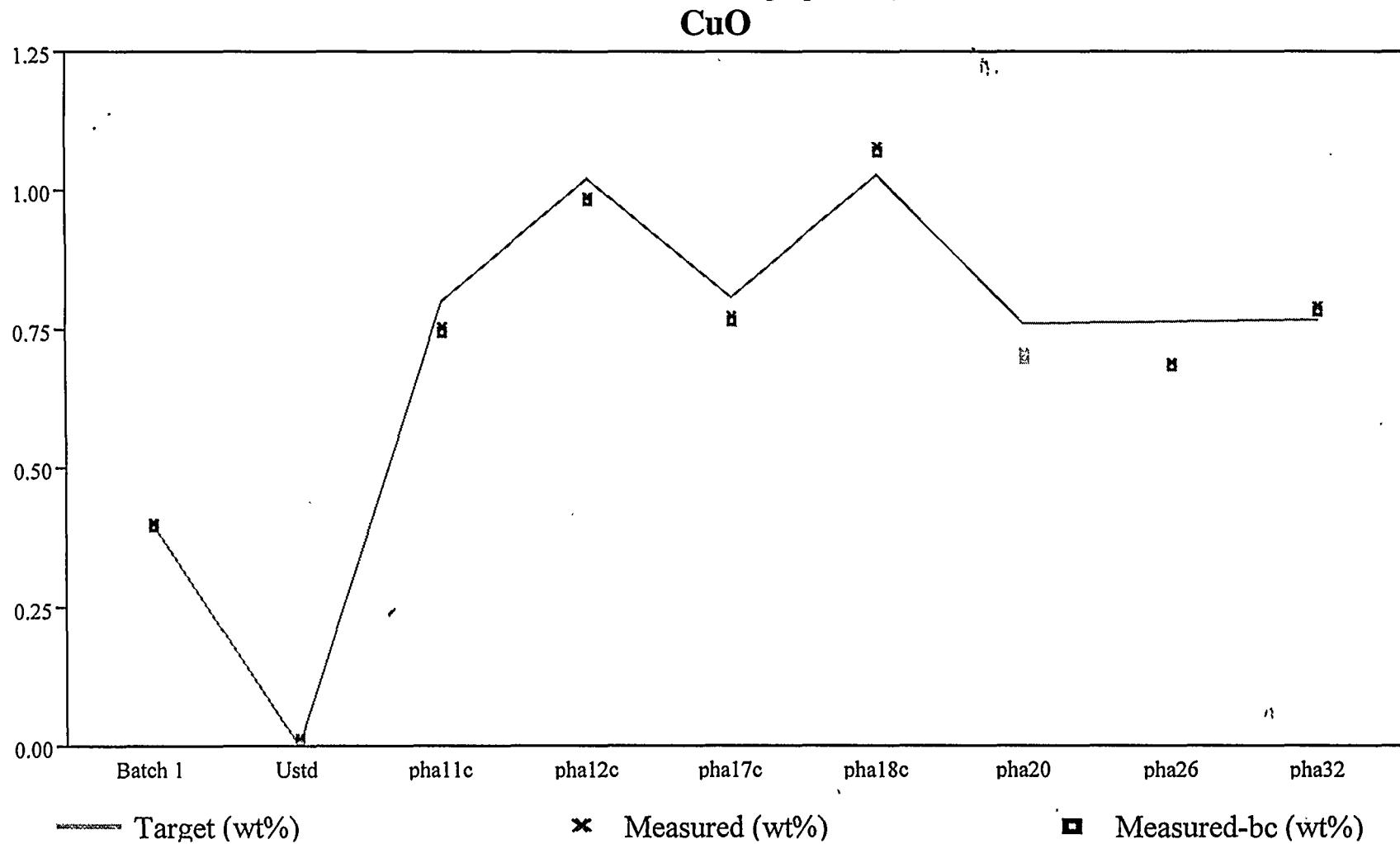


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

Fe₂O₃

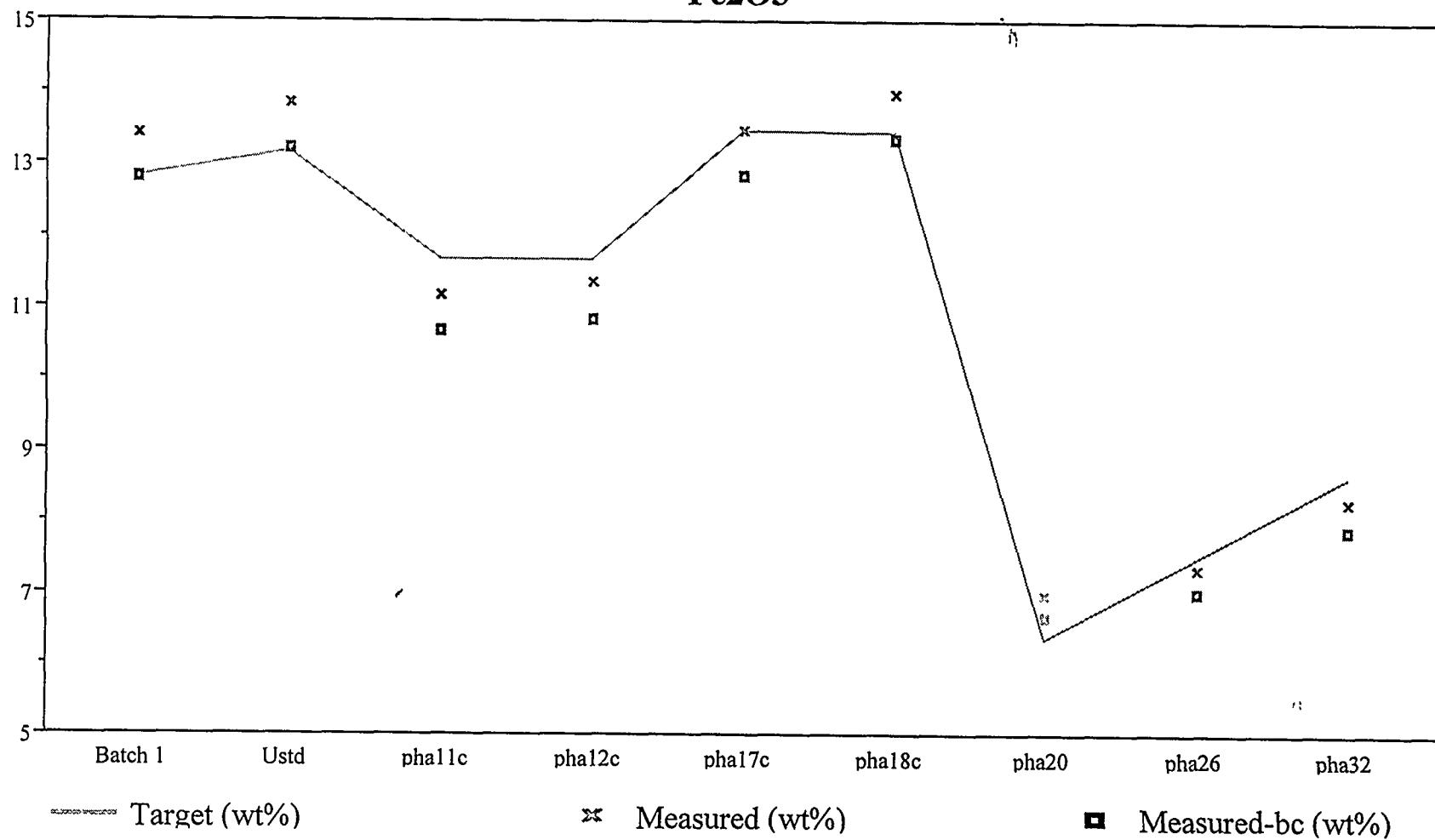


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

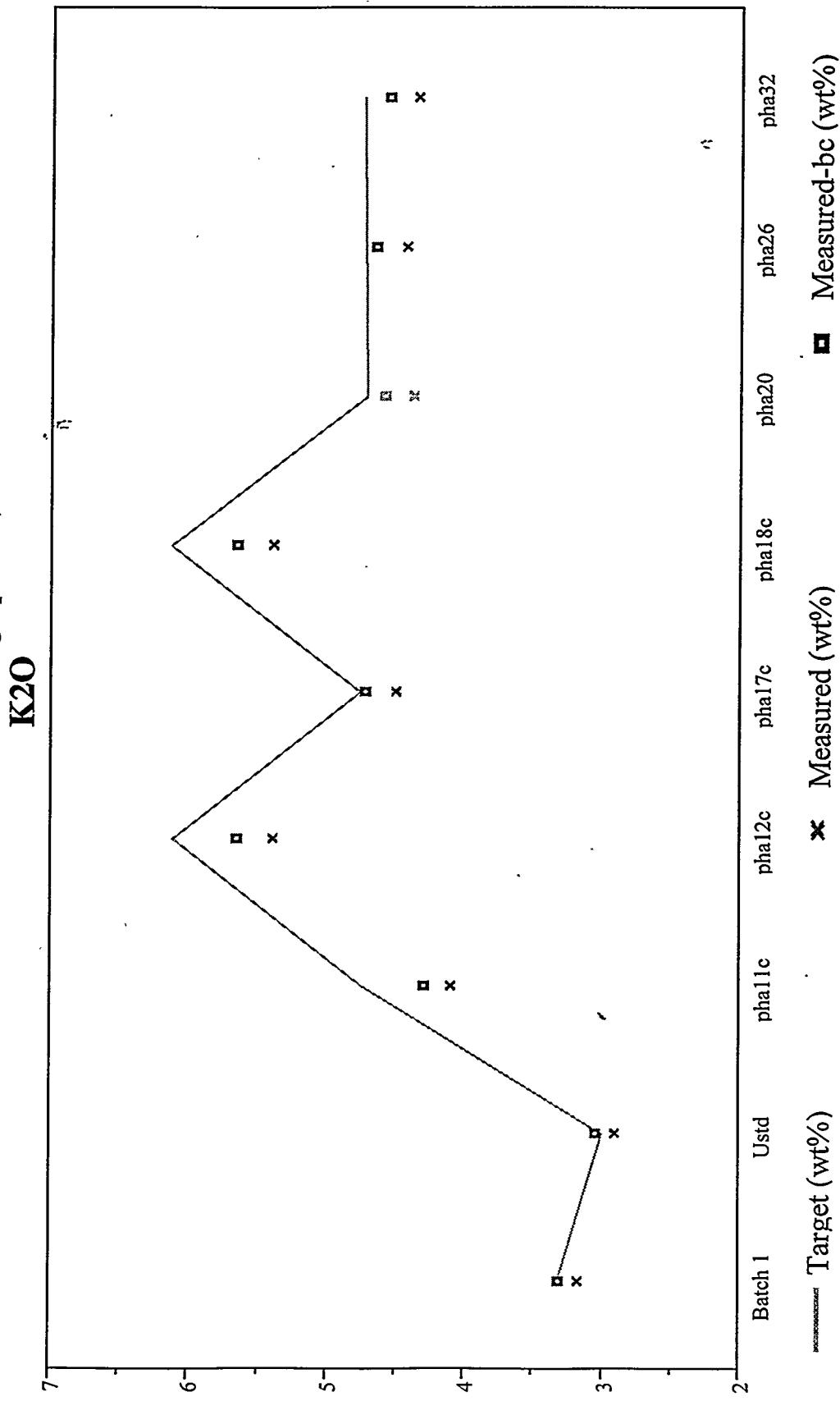


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

Li2O

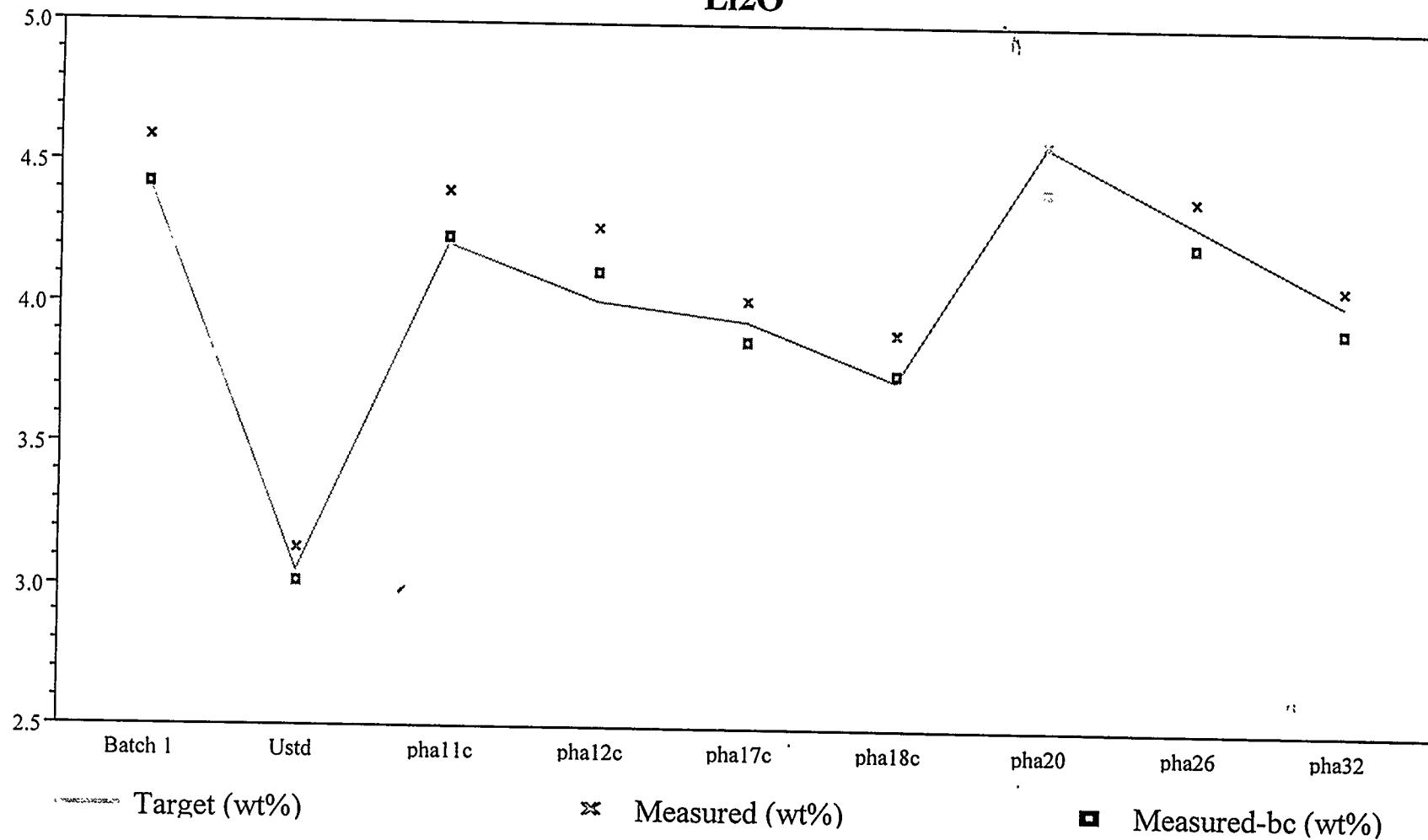


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

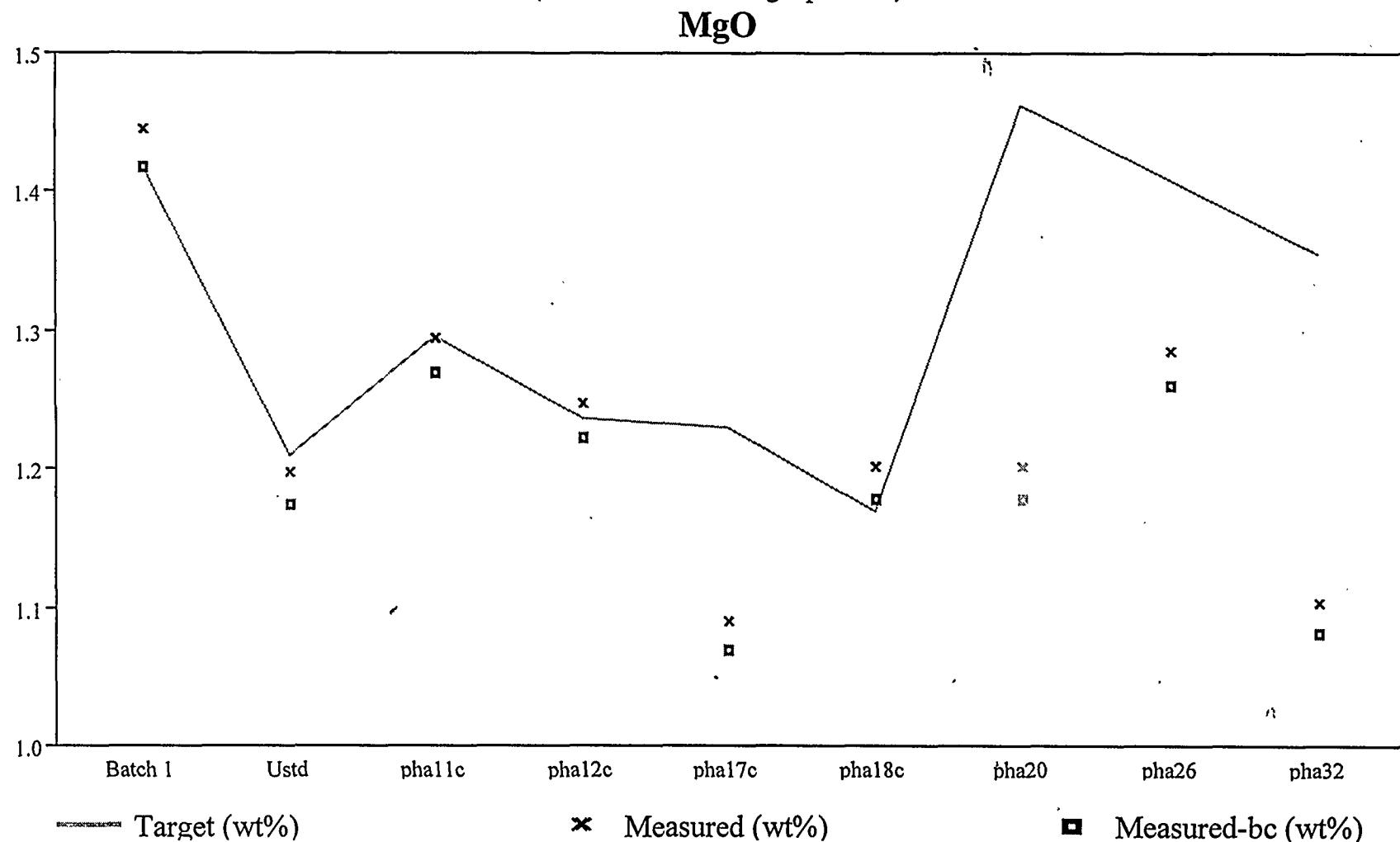


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

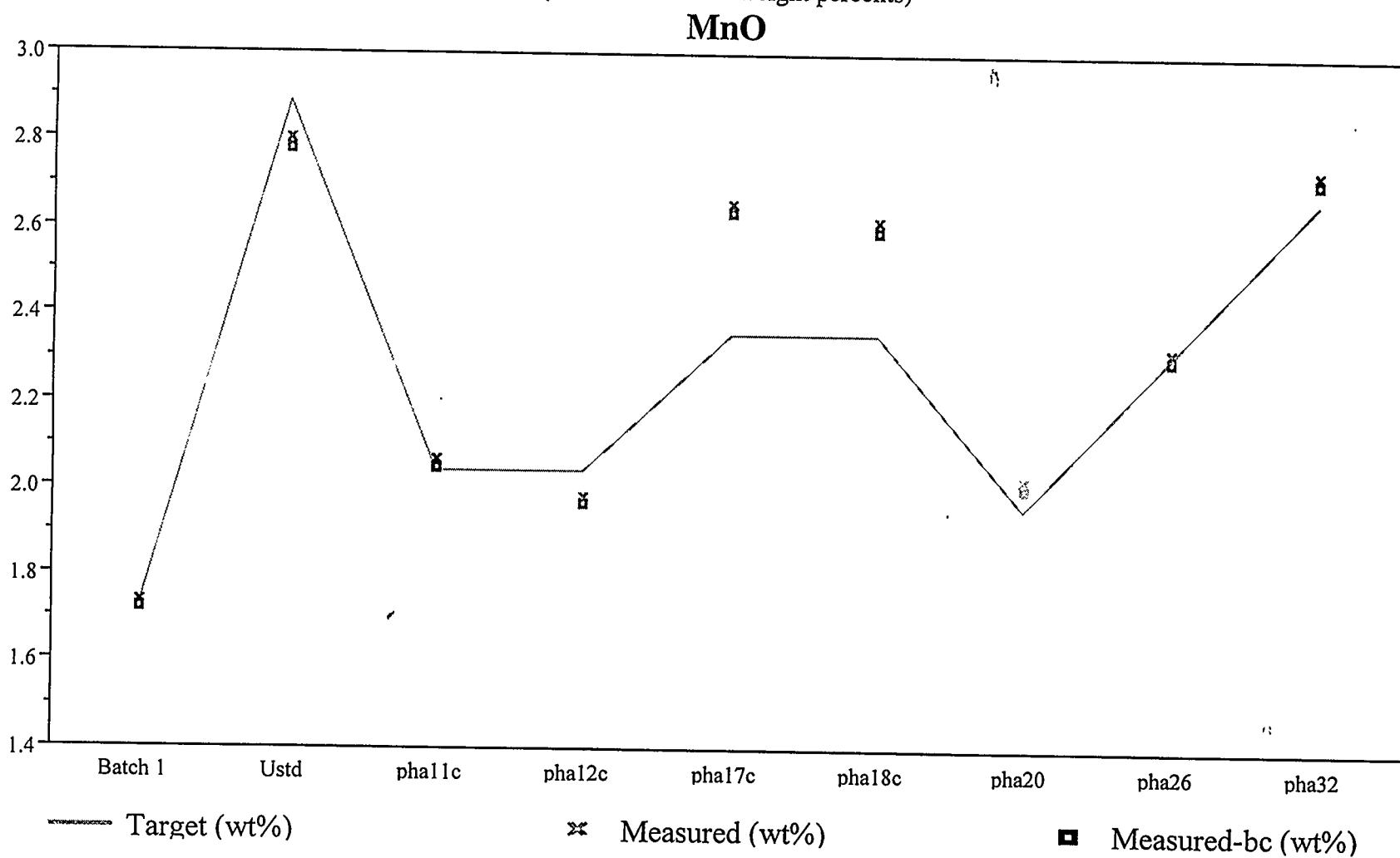


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

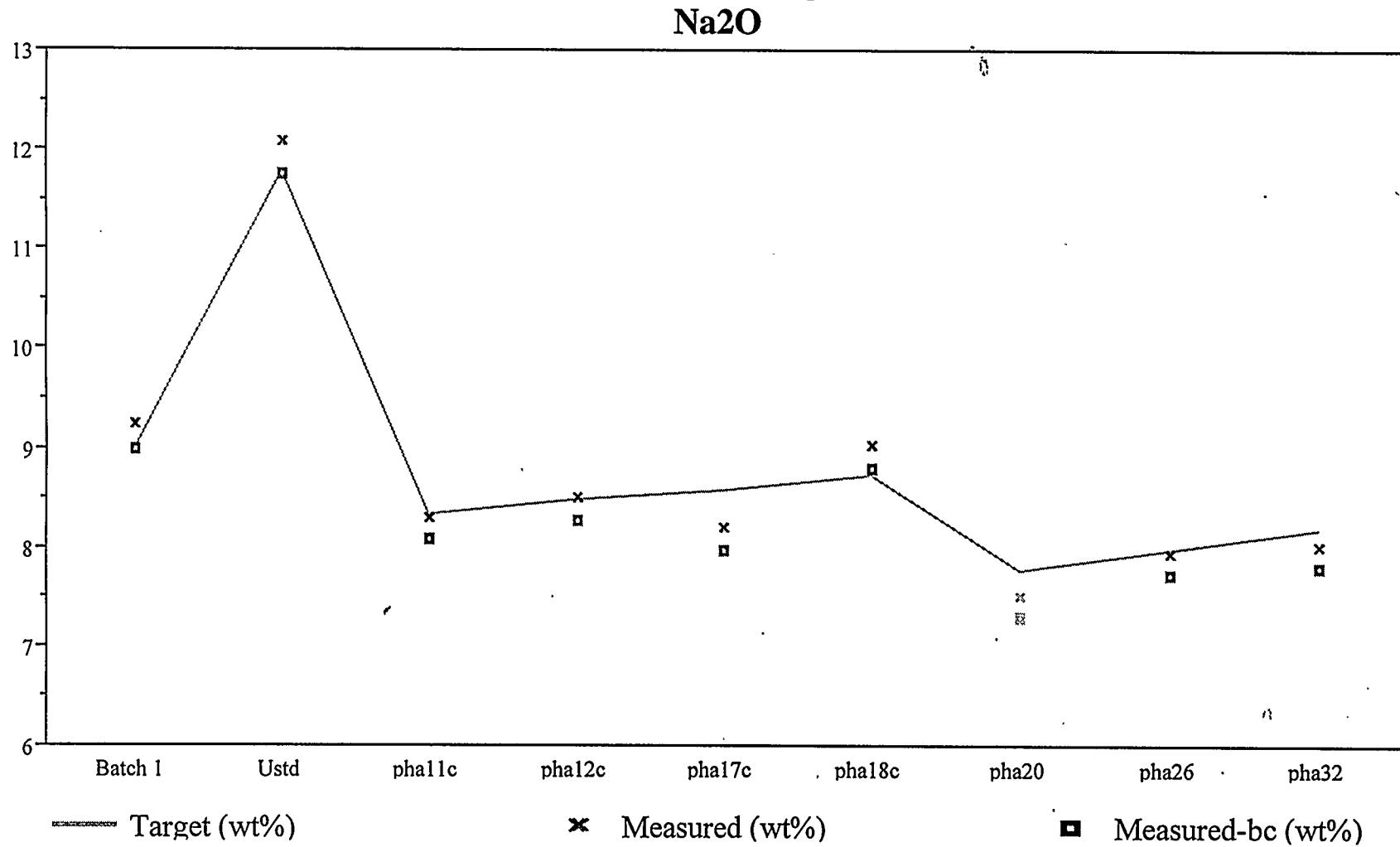


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

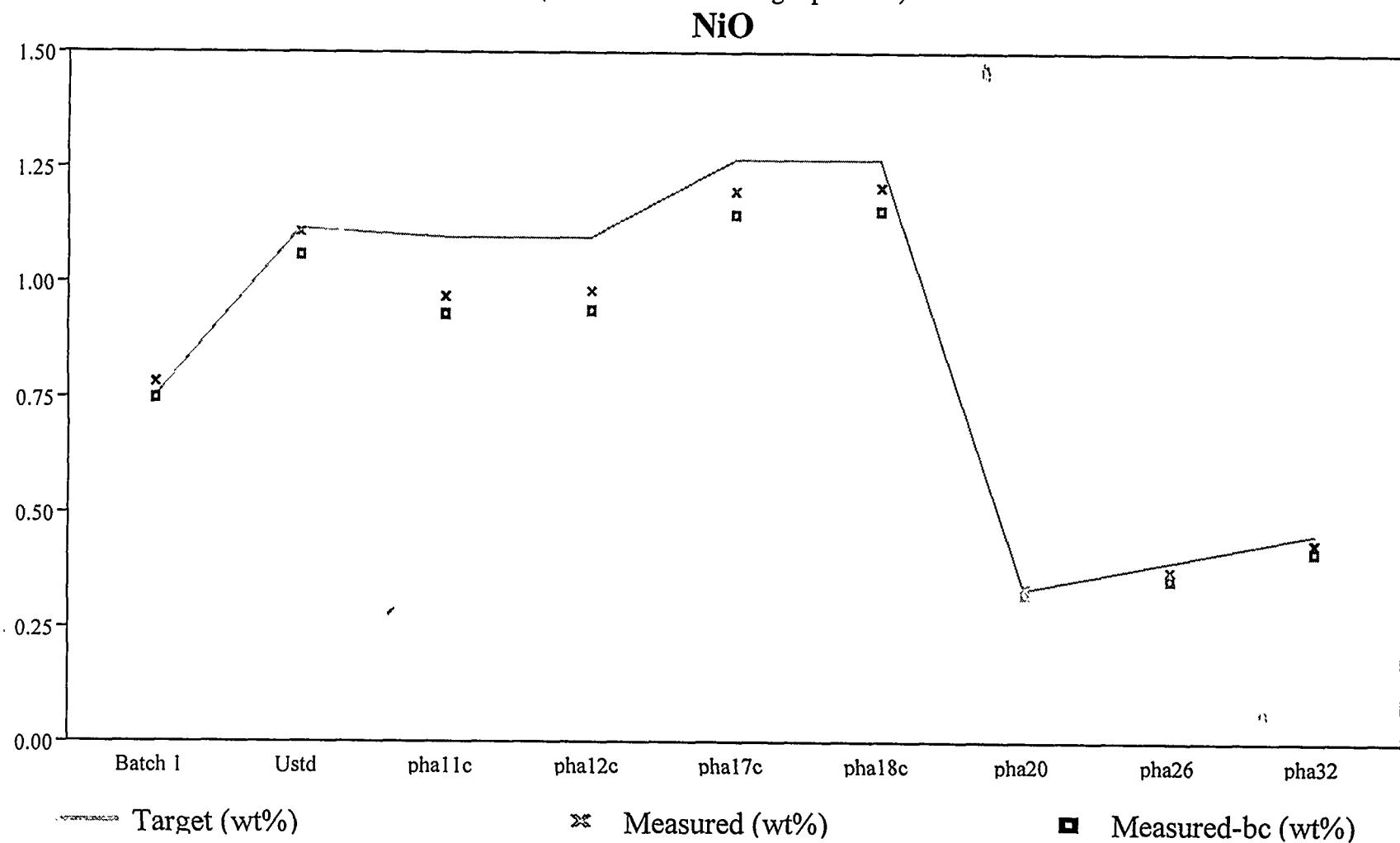


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

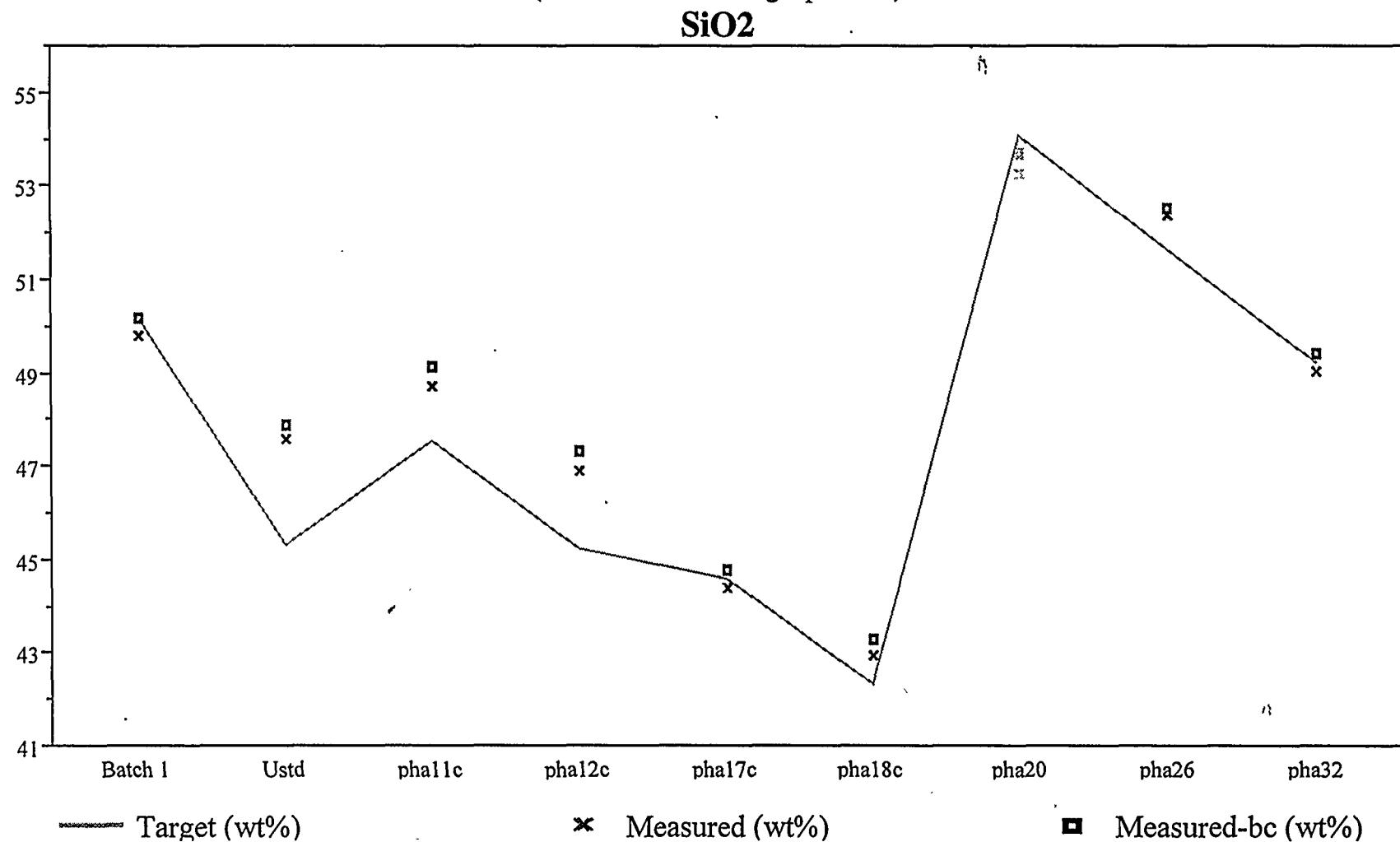


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

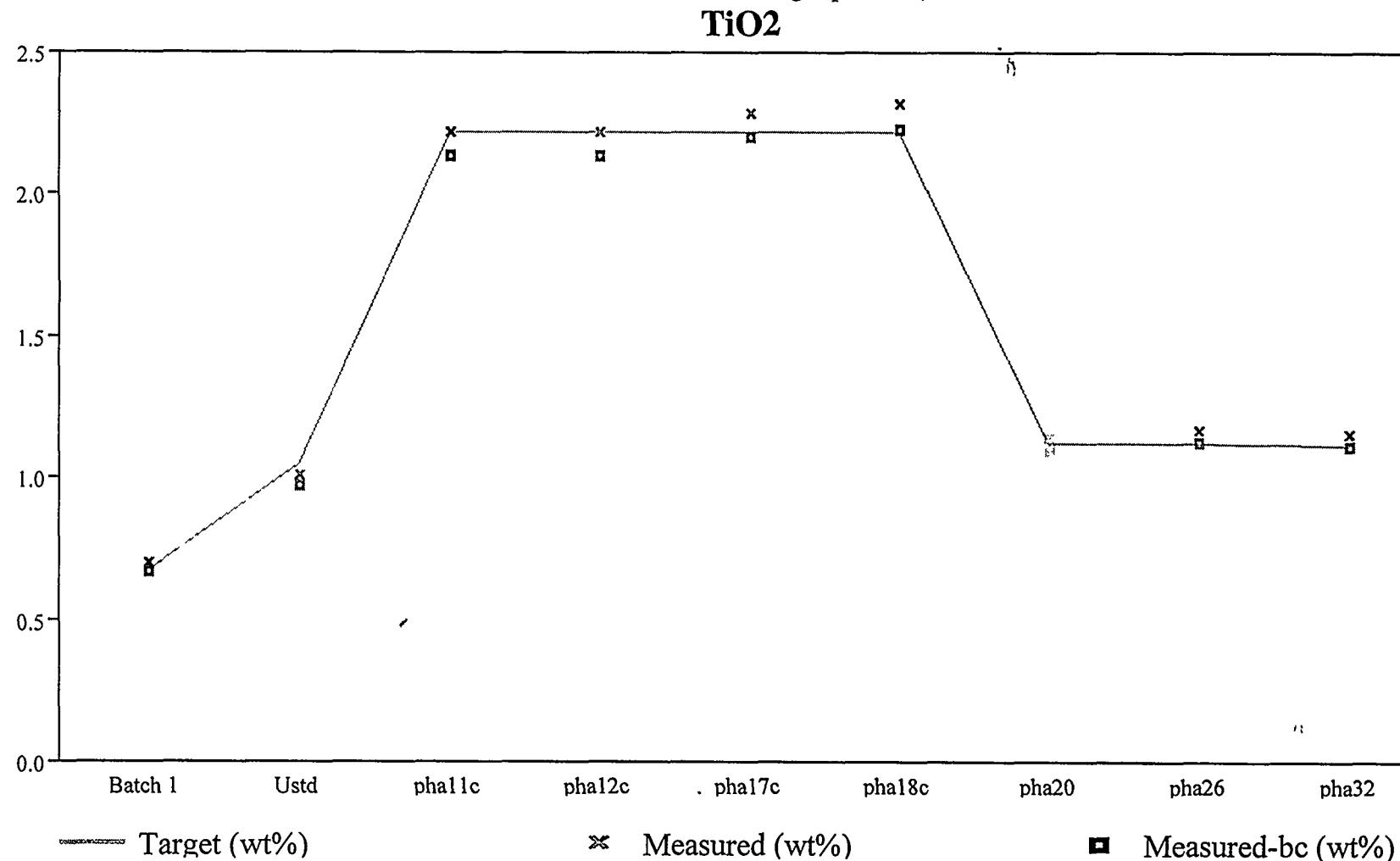


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

U3O8

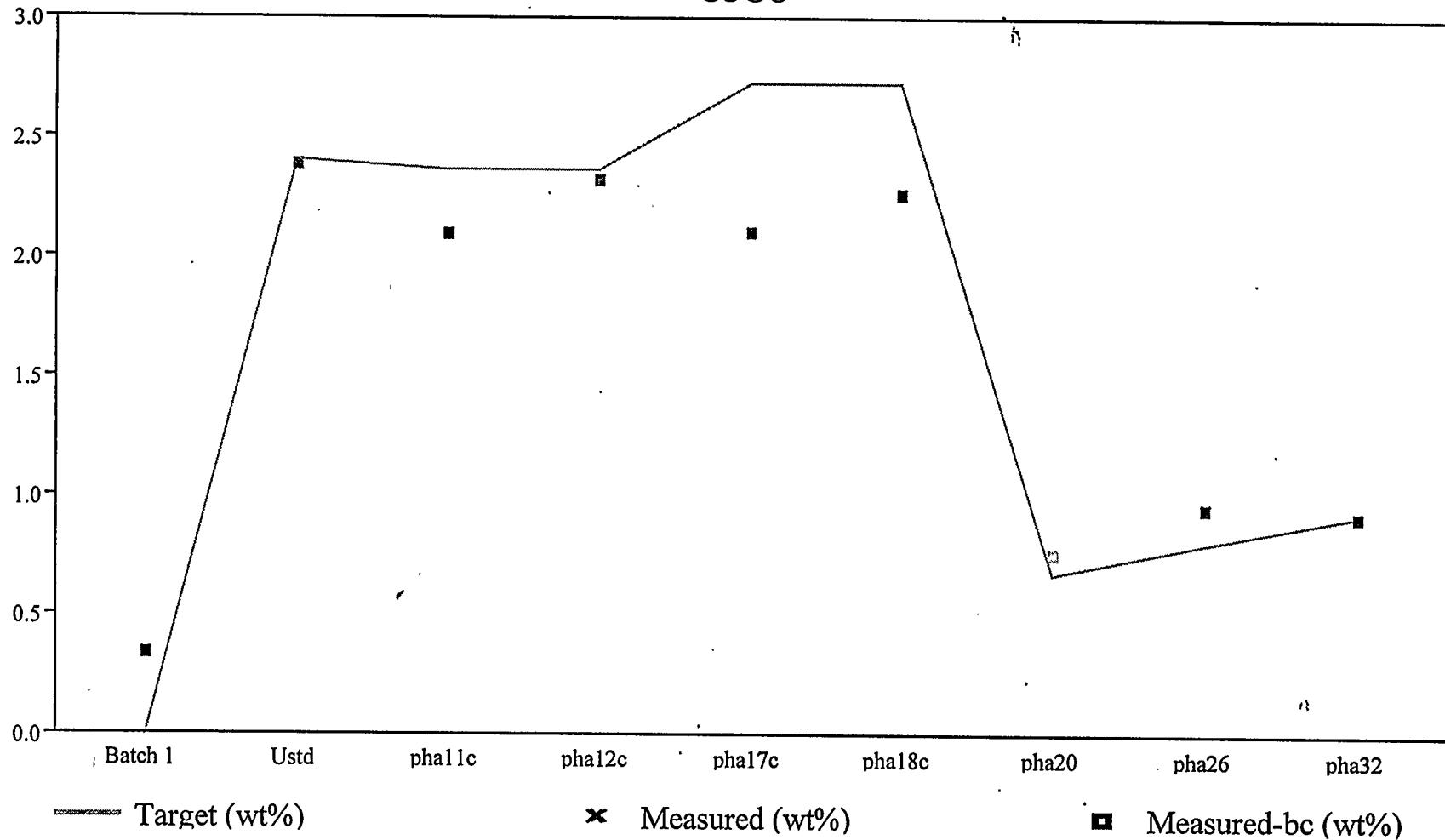


Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

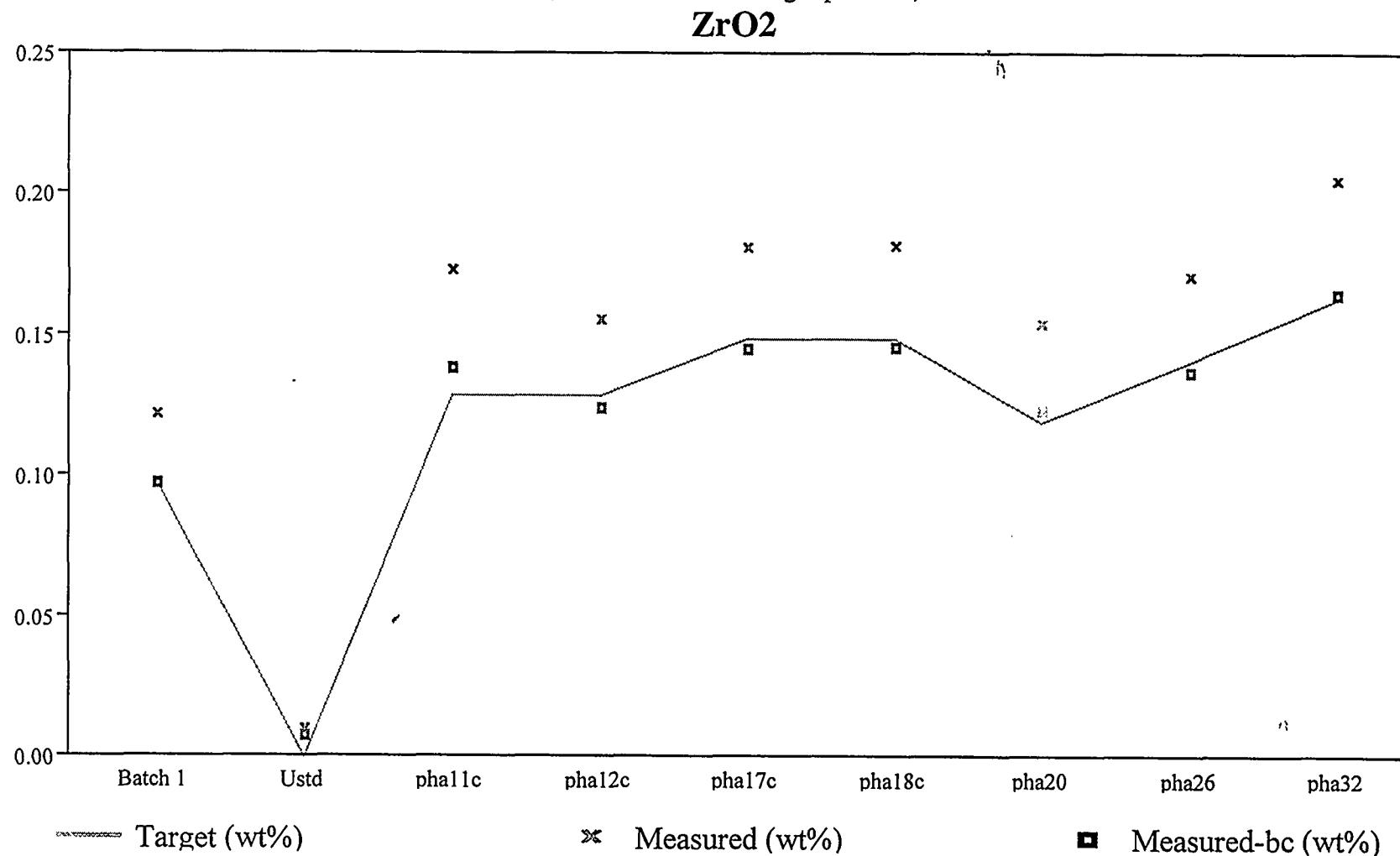
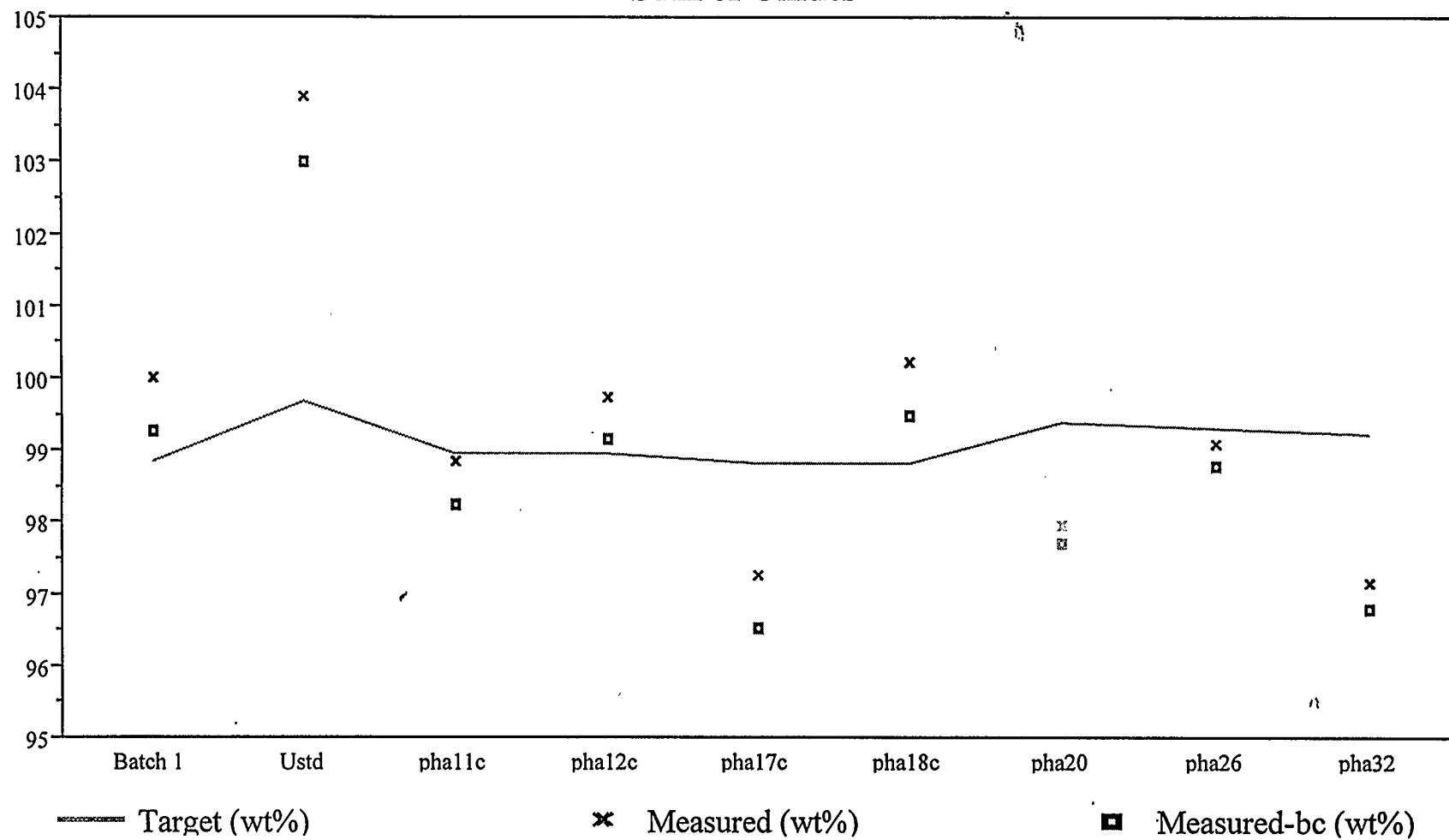


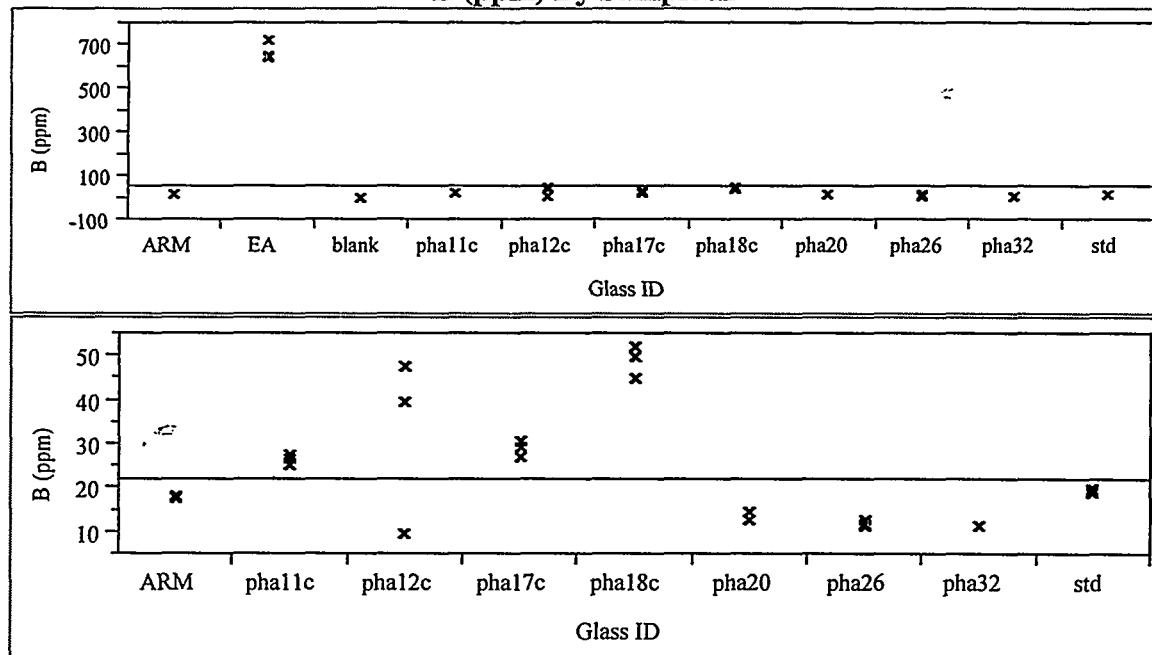
Exhibit A.4: Comparisons of Measurements versus Target Compositions
(concentrations in weight percents)

Sum of Oxides

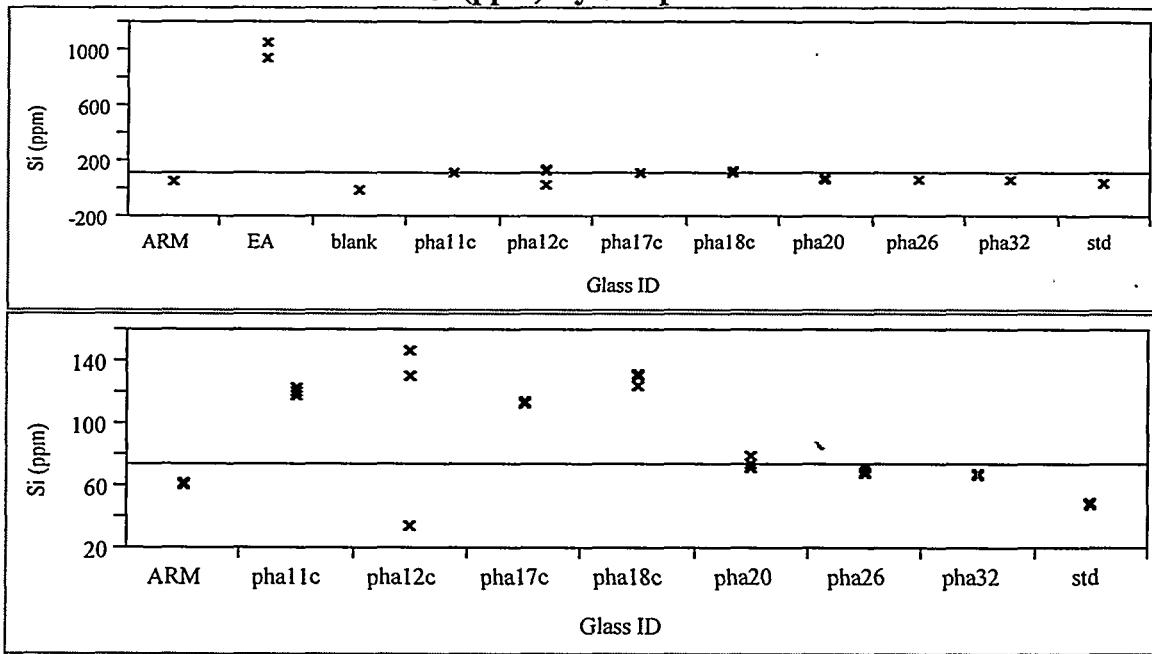


**Exhibit A.5: Plots of the Leachate Concentrations by Sample ID by Element
(with and without the EA and blank samples)**

B (ppm) By Sample ID

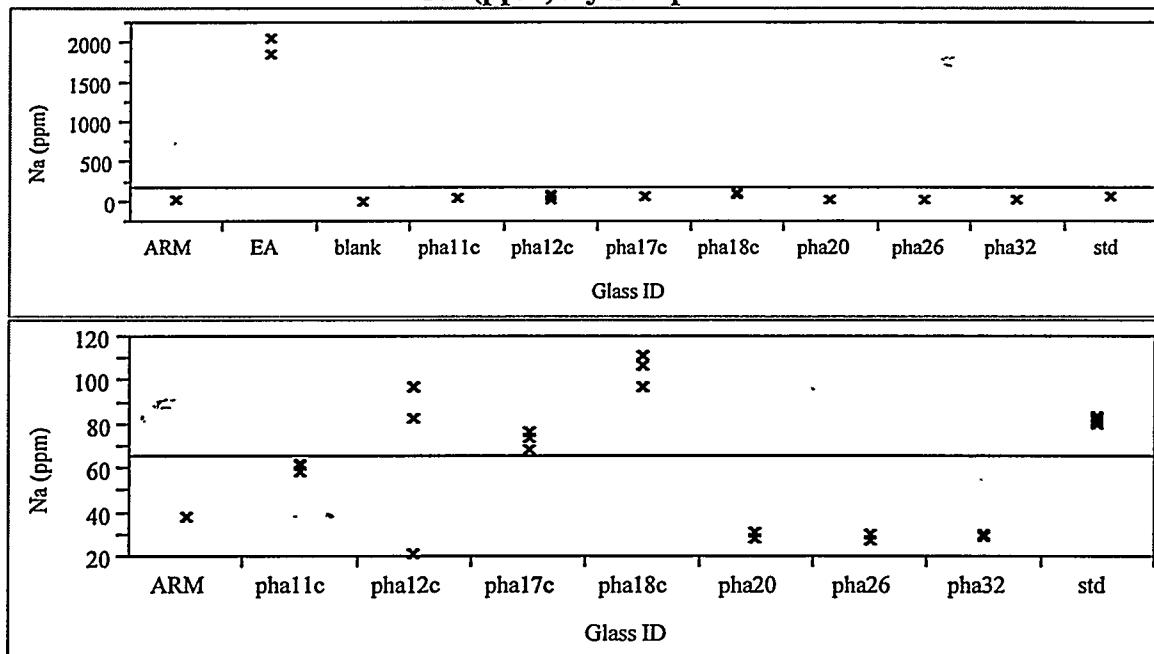


Si (ppm) By Sample ID



**Exhibit A.5: Plots of the Leachate Concentrations by Sample ID by Element
(with and without the EA and blank samples)
(continued)**

Na (ppm) By Sample ID



Li (ppm) By Sample ID

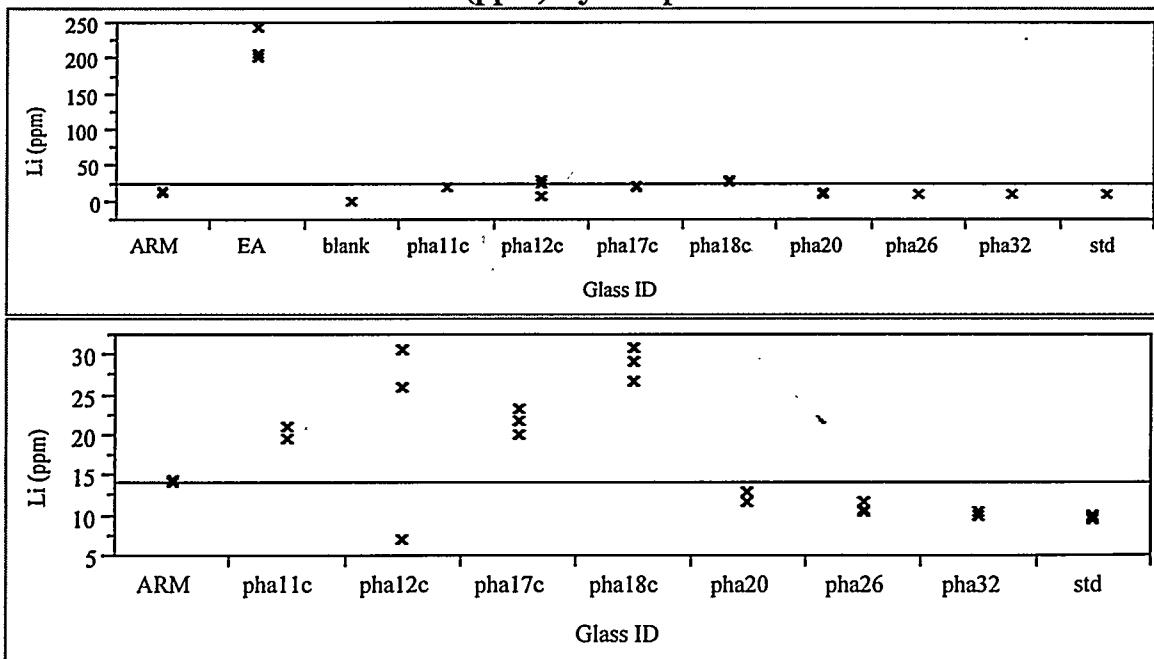


Exhibit A.6: Scatter Plots of the Normalized PCT's

Correlations Using Target Compositions

Variable	log NL[B g/L]	log NL[Si g/L]	log NL[Na g/L]	log NL[Li g/L]
log NL[B g/L]	1.0000	0.9902	0.9995	0.9994
log NL[Si g/L]	0.9902	1.0000	0.9902	0.9902
log NL[Na g/L]	0.9995	0.9902	1.0000	0.9997
log NL[Li g/L]	0.9994	0.9902	0.9997	1.0000

Correlations Using Measured Compositions

Variable	log NL[B g/L]	log NL[Si g/L]	log NL[Na g/L]	log NL[Li g/L]
log NL[B g/L]	1.0000	0.9919	0.9990	0.9997
log NL[Si g/L]	0.9919	1.0000	0.9933	0.9904
log NL[Na g/L]	0.9990	0.9933	1.0000	0.9988
log NL[Li g/L]	0.9997	0.9904	0.9988	1.0000

Correlations Using Bias-Corrected Measured Compositions

Variable	log NL[B g/L]	log NL[Si g/L]	log NL[Na g/L]	log NL[Li g/L]
log NL[B g/L]	1.0000	0.9920	0.9990	0.9997
log NL[Si g/L]	0.9920	1.0000	0.9933	0.9904
log NL[Na g/L]	0.9990	0.9933	1.0000	0.9988
log NL[Li g/L]	0.9997	0.9904	0.9988	1.0000

Scatterplot Matrix

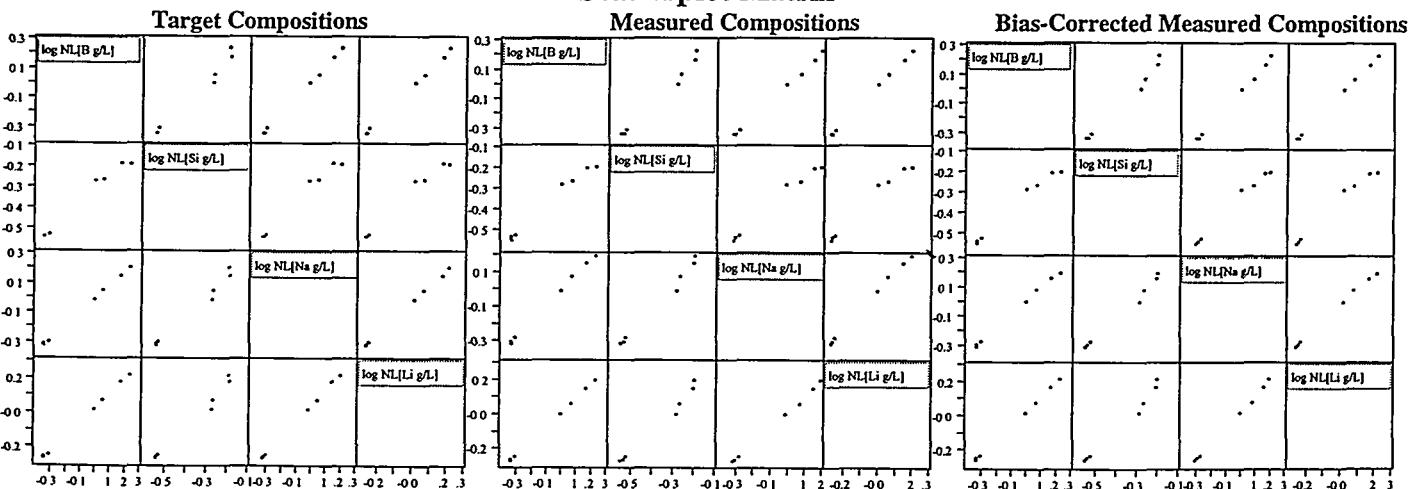
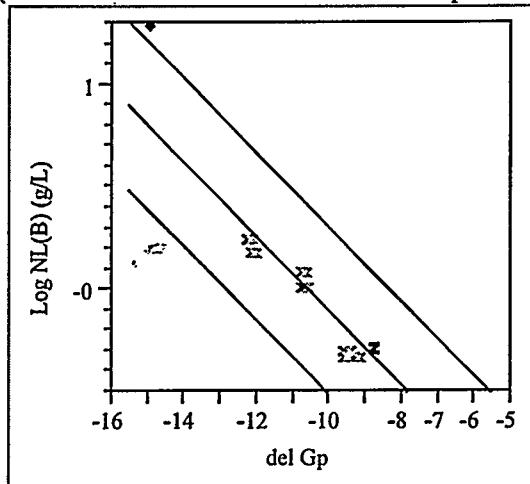


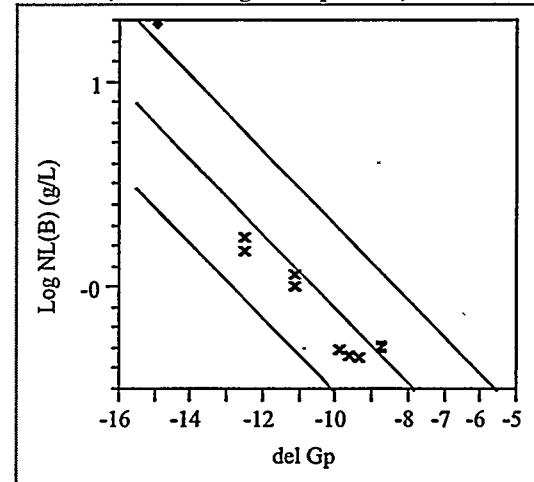
Exhibit A.7: Durability Predictions versus Measured

(Reference compositions were used to normalize the EA and ARM PCTs; their compositions were not remeasured as part of this study).

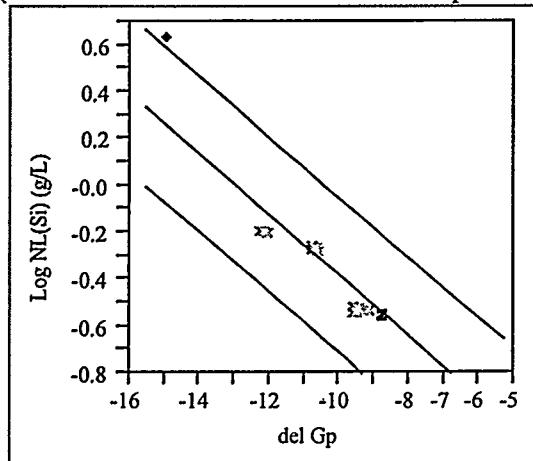
Log NL(B) (g/L) By del Gp(m)
(based on measured and bias-corrected compositions)



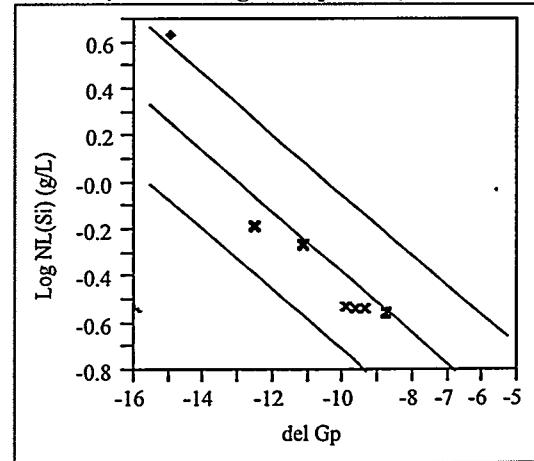
Log NL(B) (g/L) By del Gp(m)
(based on target composition)



Log NL(Si) (g/L) By del Gp(m)
(based on measured and bias-corrected compositions)



Log NL(Si) (g/L) By del Gp(m)
(based on target composition)



**Exhibit A.7: Durability Predictions versus Measured
(Continued)**

(Reference compositions were used to normalize the EA and ARM PCTs; their compositions were not remeasured as part of this study).

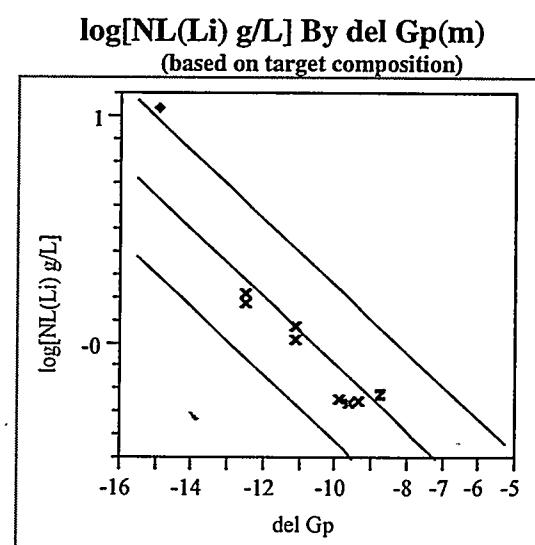
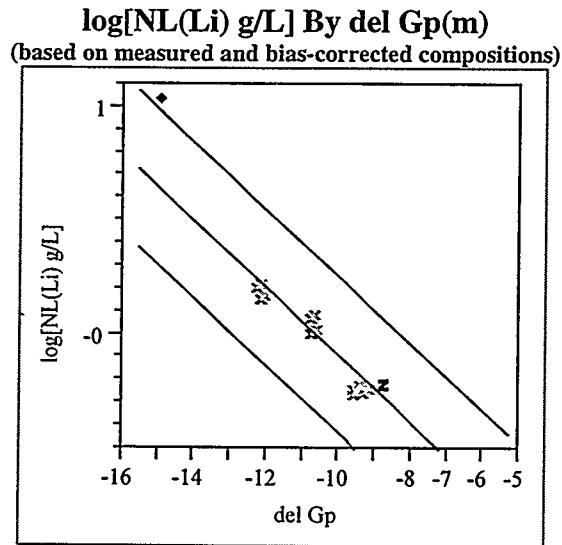
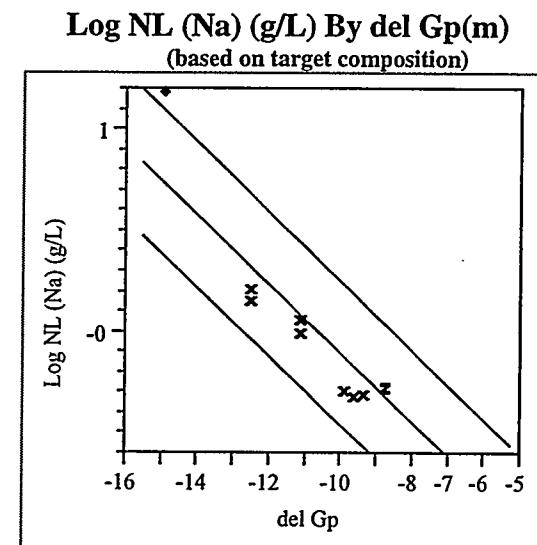
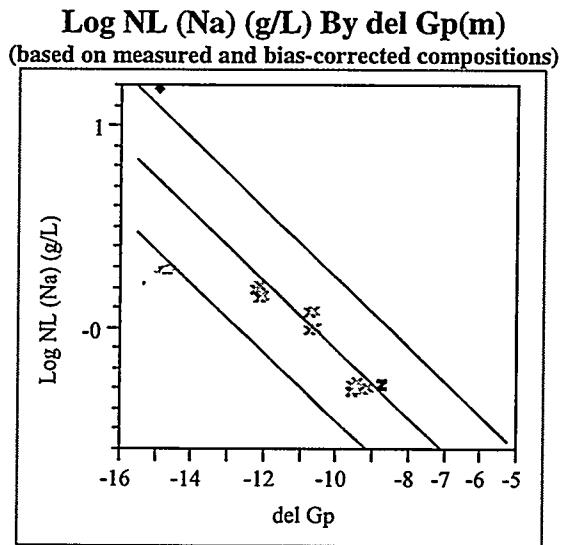
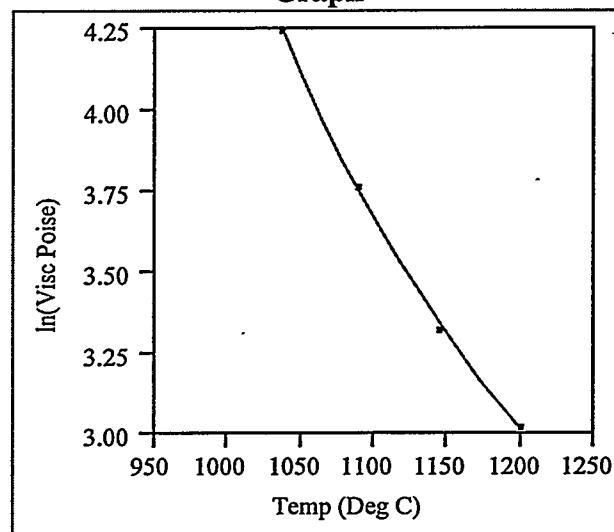


Exhibit A.8: Viscosity Measurements, Fulcher Fits, and Predictions at 1150 °C

pha12c

Parameter	Estimate	ApproxStdErr
A	-0.063709145	1.00620386
B	1756.0588712	965.597707
C	630.87637002	130.143989

Graph

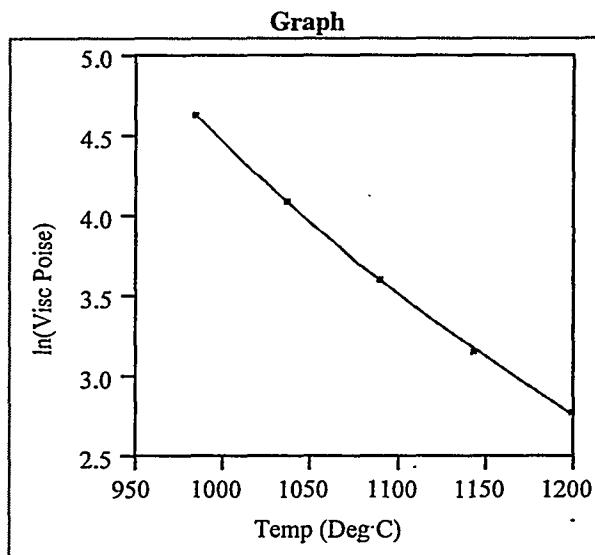


Temp (Deg C)	Visc (Poise)	ln Visc (Fulcher)	ln(Visc Poise)	Visc Pred (Poise)
1201	20.56894	3.016428	3.023782	20.42
1146	27.85945	3.345296	3.327172	28.37
1091	43.27252	3.752784	3.767518	42.64
1038	69.80163	4.249622	4.245657	70.08
1150	?	3.319028	?	27.63

Exhibit A.8: Viscosity Measurements, Fulcher Fits, and Predictions at 1150 °C
(continued)

pha15c

Parameter	Estimate	ApproxStdErr
A	-3.897646475	0.70143483
B	6567.0040005	1217.52255
C	215.12529994	79.9487732

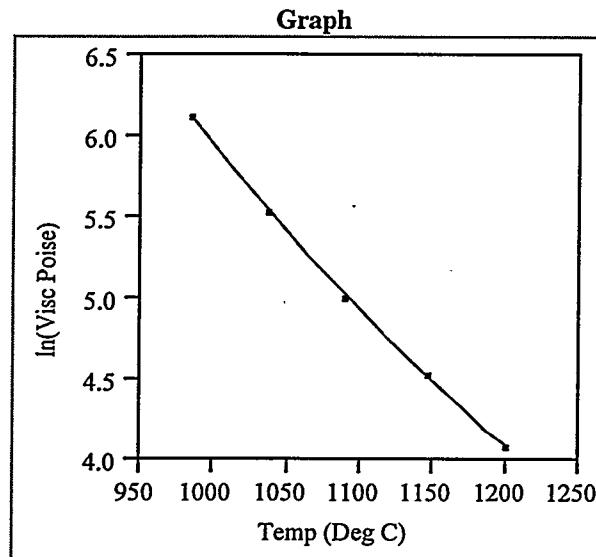


Temp (Deg C)	Visc (Poise)	ln Visc (Fulcher)	ln(Visc Poise)	Visc Pred (poise)
1199.5	16.10111	2.773598	2.778888	16.02
1144	23.58912	3.172203	3.160785	23.86
1090.5	36.93064	3.604288	3.609042	36.76
1037	60.11118	4.092627	4.096196	59.90
984	103.6705	4.643412	4.641217	103.90
1150	?	3.126829	?	22.80

Exhibit A.8: Viscosity Measurements, Fulcher Fits, and Predictions at 1150 °C
(continued)

pha32

Parameter	Estimate	ApproxStdErr
A	-4.439457996	1.33524274
B	9564.4297263	2686.15911
C	79.627339442	140.57891



Temp (Deg C)	Visc (Poise)	In Visc (Fulcher)	ln(Visc Poise)	Visc Pred (poise)
1200.5	59.48209	4.093563	4.085675	59.95
1147.5	93.03869	4.517068	4.533015	91.57
1091	150.2267	5.017422	5.012145	151.02
1038	253.1414	5.540407	5.533948	254.78
985	458.6551	6.124623	6.128299	456.97
1150	?	4.496149	?	89.67

This page intentionally left blank.

Appendix B. Two Additional Glasses: pha14c and pha15c

This appendix looks at two additional glasses (designated as pha14c and pha15c) that were batched and tested as part of PHA Phase 4. The target compositions for this pair of glasses are available in Table 2. The chemical compositions of these glasses were analyzed by ADS. The results from the analyses are provided in Table B.1 and Table B.2 at the end of this appendix. Plots of these values are provided in Exhibit B.1. No problems are seen in these results and all of the reported values are used in the calculations that follow. With the limited information by the Batch 1 results, no bias correction is conducted for these data. The average compositions for the Batch 1, pha14c, and pha15c glasses are provided in Chart B.1

Chart B.1: Target and Measured Compositions (in wt%) for Batch 1, pha14c, and pha15c

	Batch 1		pha14c		pha15c	
	Target	Measured	Target	Measured	Target	Measured
Al ₂ O ₃	4.877	4.693	3.245	3.293	3.227	3.322
B ₂ O ₃	7.777	7.501	8.174	7.729	9.003	8.515
CaO	1.220	1.364	1.234	1.393	1.230	1.351
Cr ₂ O ₃	0.107	0.243	0.144	0.154	0.144	0.157
CuO	0.399	0.417	0.808	0.813	1.031	1.022
Fe ₂ O ₃	12.839	13.695	13.470	13.760	13.467	13.621
K ₂ O	3.327	3.160	4.760	4.487	6.140	5.389
Li ₂ O	4.429	4.568	4.030	3.979	3.824	3.797
MgO	1.419	1.415	1.255	1.271	1.195	1.215
MnO	1.726	1.705	2.355	2.483	2.355	2.500
Na ₂ O	9.003	9.215	8.511	8.457	8.659	8.694
NiO	0.751	0.894	1.268	1.195	1.268	1.186
SiO ₂	50.220	48.336	45.569	44.149	43.289	41.745
TiO ₂	0.677	0.688	1.123	1.139	1.122	1.135
U ₃ O ₈	0.000	0.354	2.731	2.460	2.731	2.611
ZrO ₂	0.098	0.148	0.149	0.215	0.149	0.196
Sum of Oxides	98.869	98.469	98.826	97.052	98.834	96.530

These glasses were subjected to the Product Consistency Test (in triplicate) along with the typical standard glasses and solutions. The results were analyzed by ADS and Table B.3 provides the data from these analyses. The results are plotted in Exhibit B.2 (with and without the EA and blank samples), and no problems are seen in these data. The results from measurements of three samples of the standard multi-element solution are provided in this table and exhibit. They are summarized in Chart B.2. No indication of a problem is seen in these results.

Chart B.2: Measurements of Standard Solution

Block	Sequence	B (ppm)	Si (ppm)	Na (ppm)	Li (ppm)
1	1	19.5	48.9	82.7	9.9
1	1	19.8	49.5	84.2	10.1
1	1	19.7	49.0	83.5	10.0
Block 1	average	19.34	49.22	81.29	9.63
Reference	Value	20	50	81	10
% Difference		-3.30%	-1.55%	0.36%	-3.73%

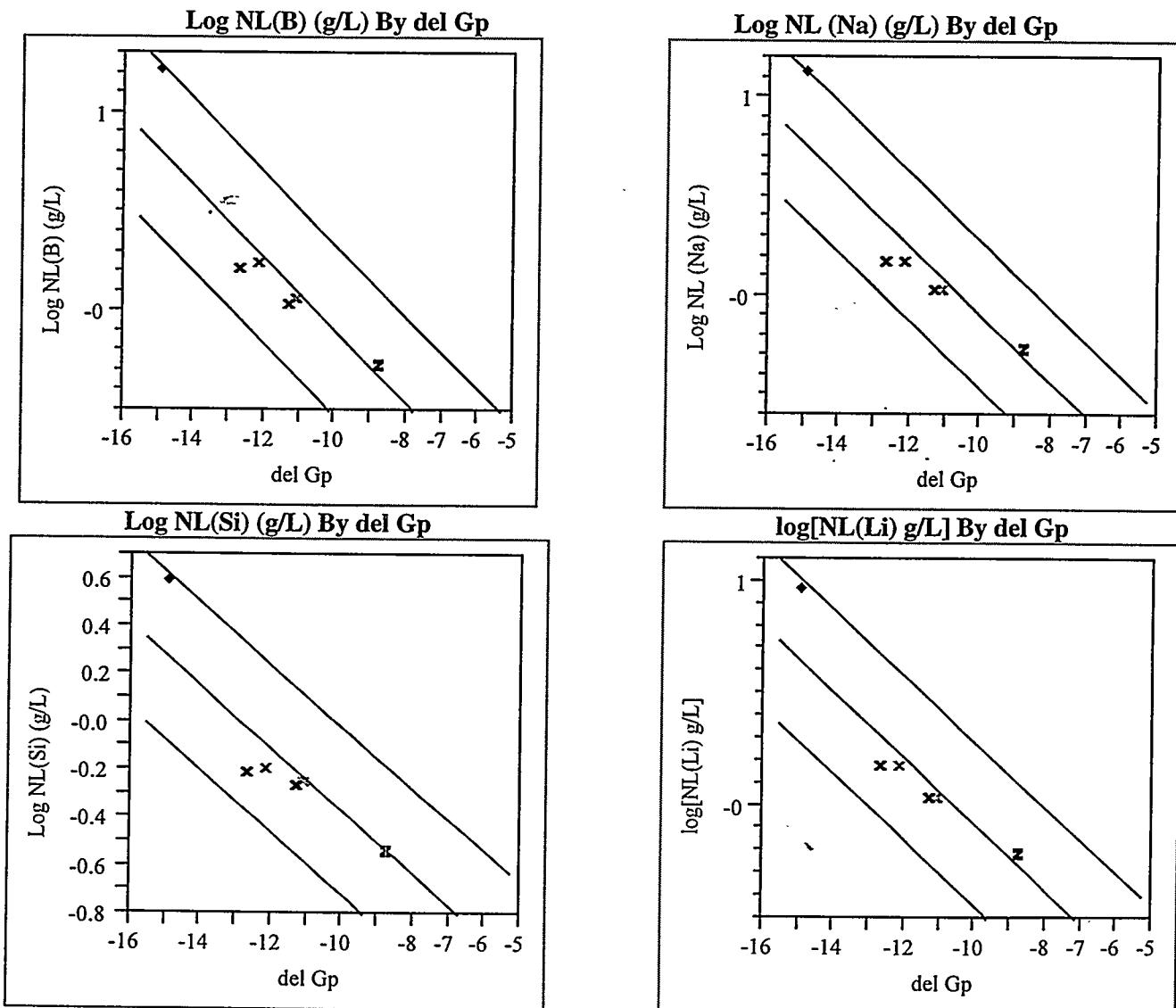
The information in Chart B.1 and Table B.3 was used to normalize the PCT results for these glasses. The normalization was conducted as described in the body of the paper. The normalized PCTs are given in Chart B.3.

Chart B.3: Normalized PCTs for Extra Phase Glasses

Glass ID	Composition	log NL	log NL	log NL	log NL	NL	NL	NL	NL
		[B (g/L)]	[Si (g/L)]	[Na (g/L)]	[Li (g/L)]	B (g/L)	Si (g/L)	Na (g/L)	Li (g/L)
ARM	reference comp. [10]	-0.26533	-0.53190	-0.25933	-0.20825	0.54	0.29	0.55	0.62
	EA reference comp. [10]	1.21765	0.59582	1.12991	0.96905	16.51	3.94	13.49	9.31
pha14c	measured	0.06317	-0.25322	0.03246	0.03802	1.16	0.56	1.08	1.09
	target	0.03885	-0.26697	0.02967	0.03252	1.09	0.54	1.07	1.08
pha15c	measured	0.24023	-0.19416	0.17513	0.18018	1.74	0.64	1.50	1.51
	target	0.21601	-0.20994	0.17687	0.17711	1.64	0.62	1.50	1.50

These PCTs reveal that pha14c and pha15c are durable when compared to EA. The DWPF models for durability and their prediction limits (at 95% confidence) are shown in Figure B.1. The PCT results for EA (shown as a diamond), ARM (shown as a "z"), pha14c, and pha15c. The two glasses, pha14c and pha15c, are each shown as an "x". The darker "x" represents the results based upon the target compositions for these two glasses and the lighter "x" represents the results based upon the measured compositions. Note that the PCT results for both pha14c and pha15c reveal acceptable PCTs that are well predicted by the current DWPF durability model.

Figure B.1: Durability Predictions versus PCT Measurements



**Table B.1: Composition Measurements Using Peroxide Fusion Dissolutions
(expressed as cation weight fractions)**

Glass ID	Block	Seq	LIMS #	Lab ID	B	Ca	Si
Batch 1	1	1	131828 10x	Na2O2 - Batch 1	0.02345	0.00932	0.23449
pha15c	1	2	131827-dup	Na2O2 - pha 15C-2	0.02669	0.00902	0.19900
pha15c	1	3	131826-dup	Na2O2 - pha 15C-1	0.02618	0.00981	0.19736
pha14c	1	4	131825-dup 10x	Na2O2 - pha 14C-2	0.02374	0.00978	0.20706
pha14c	1	5	131824-dup	Na2O2 - pha 14C-1	0.02400	0.01019-	0.20938
pha15c	1	6	131827 10x	Na2O2 - pha 15C-2	0.02680	0.00908	0.19993
pha15c	1	7	131826 10x	Na2O2 - pha 15C-1	0.02610	0.00977	0.19751
pha14c	1	8	131825 10x	Na2O2 - pha 14C-2	0.02415	0.00997	0.21106
pha14c	1	9	131824 10x	Na2O2 - pha 14C-1	0.02412	0.01032	0.21060
Batch 1	1	10	131828-dup	Na2O2 - Batch 1	0.02314	0.00927	0.23055

Values at their detection limits were set to their detection limits.

**Table B.2: Composition Measurements From Microwave Preparation
(expressed as cation weight fractions)**

Glass	ID	Block	Seq	LIMS #	Lab ID	ICP-ES														AA ⁶	
						Al	Ca	Cr	Cu	Fe	Li	Mg	Mn	Na	Nb	Ni	Si	Ti	U	Zr	
Batch 1	1	1	131739		MW10x - Batch 1	0.02538	0.01030	0.00176	0.00338	0.09840	0.02098	0.00882	0.01367	0.06815	0.00051	0.00733	0.22431	0.00424	0.00301	0.00137	0.02683
phal5c	1	2	131738-dup		MW10x - pha 15C-2	0.01783	0.01011	0.00103	0.00826	0.09520	0.01818	0.00735	0.01922	0.06661 ¹	0.00050	0.00920	0.19250	0.00681	0.02203	0.00138	0.04358
phal5c	1	3	131737-dup		MW10x - pha 15C-1	0.01733	0.00979	0.00106	0.00817	0.09460	0.01776	0.00718	0.01917	0.06401	0.00050	0.00926	0.18951	0.00676	0.02232	0.00140	0.04317
phal4c	1	4	131734-dup		MW10x - pha 14C-2	0.01740	0.00982	0.00104	0.00648	0.09542	0.01860	0.00762	0.01901	0.06332	0.00052	0.00925	0.20166	0.00678	0.02084	0.00145	0.03556
phal4c	1	5	131732-dup		MW10x - pha 14C-1	0.01731	0.00984	0.00103	0.00650	0.09602	0.01849	0.00762	0.01916	0.06233	0.00054	0.00936	0.20303	0.00682	0.02022	0.00150	0.03483
phal5c	1	6	131738		MW10x - pha 15C-2	0.01777	0.00991	0.00110	0.00814	0.09575	0.01739	0.00748	0.01953	0.06476	0.00050	0.00940	0.19401	0.00682	0.02213	0.00149	0.04635
phal5c	1	7	131737		MW10x - pha 15C-1	0.01741	0.00974	0.00110	0.00809	0.09555	0.01722	0.00731	0.01953	0.06259	0.00050	0.00944	0.19123	0.00682	0.02209	0.00154	0.04587
phal4c	1	8	131734		MW10x - pha 14C-2	0.01750	0.00985	0.00108	0.00648	0.09667	0.01836	0.00774	0.01937	0.06298	0.00052	0.00949	0.20309	0.00685	0.02138	0.00163	0.03818
phal4c	1	9	131732		MW10x - pha 14C-1	0.01751	0.00987	0.00107	0.00651	0.09687	0.01849	0.00767	0.01938	0.06230	0.00054	0.00946	0.20510	0.00687	0.02099	0.00178	0.04044
Batch 1	1	10	131739-dup		MW10x - Batch 1	0.02429	0.01010	0.00156	0.00328	0.09317	0.02146	0.00824	0.01274	0.06857	0.00051	0.00673	0.21442	0.00401	0.00301	0.00081	0.02564

Values at their detection limits were set to their detection limits.

6

The potassium values were generated via Atomic Absorption (AA).

Table B.3: Composition of PCT Leachate Solutions for Batch 1, ARM, pha14c, and pha15c

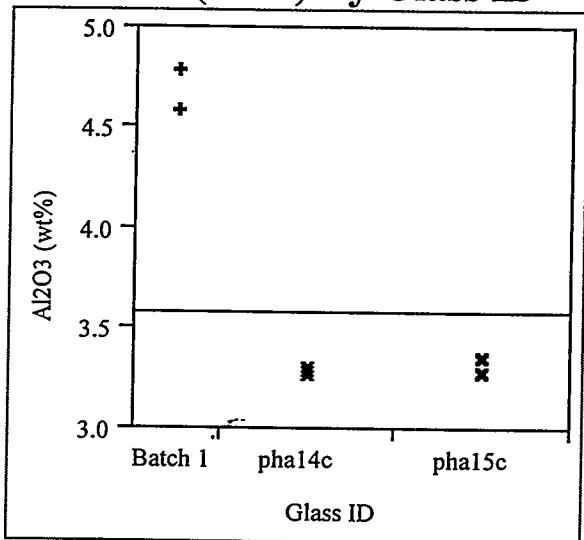
Glass	ID	Blk	Seq	LIMS #	Sample ID	Concentrations in ppm (as reported)					Concentrations in ppm (after correcting for dilution)					Common Logarithm of ppm Concentrations			
						B	Li	Na	Pb	Si	B	Si	Na	Li	Pb	log[B]	log[Si]	log[Na]	log[Li]
						std	1	1	132114	PCT leachate - Std-1	19.45	9.66	81.52	0.03	49.44	19.45	49.44	81.52	9.66
EA	1	2	132129	PCT leachate - EA-3	361.98	115.01	1049.48	0.29	555.43	603.31	925.73	1749.17	191.69	0.48	2.78054	2.96648	3.24283	2.28259	
EA	1	3	132128	PCT leachate - EA-2	370.15	117.56	1061.62	0.29	560.26	616.93	933.78	1769.40	195.94	0.48	2.79024	2.97024	3.24783	2.29212	
EA	1	4	132127	PCT leachate - EA-1	313.35	99.94	920.58	0.29	502.87	522.26	838.14	1534.33	166.57	0.48	2.71788	2.92332	3.18592	2.22160	
pha15c	1	5	132126	PCT leach- Pha 15C-3	27.46	15.96	57.56	0.03	75.75	45.77	126.26	95.94	26.59	0.05	1.66061	2.10125	1.98199	1.42478	
pha15c	1	6	132125	PCT leach- Pha 15C-2	27.72	16.15	58.37	0.03	74.58	46.21	124.30	97.29	26.92	0.05	1.64471	2.09446	1.98808	1.43003	
pha15c	1	7	132124	PCT leach- Pha 15C-1	27.57	15.96	57.81	0.03	74.29	45.96	123.82	96.35	26.61	0.05	1.66236	2.09279	1.98387	1.42500	
std	1	8	132123	PCT leachate - Std-2	19.31	9.59	81.09	0.03	49.35	19.31	49.35	81.09	9.59	0.03	1.28585	1.69327	1.90895	0.98195	
pha14c	1	9	132122	PCT leach- Pha 14C-3	17.08	12.34	41.58	0.03	70.53	28.47	117.55	69.29	20.57	0.05	1.45445	2.07021	1.84070	1.31321	
pha14c	1	10	132121	PCT leach- Pha 14C-2	16.32	11.93	39.63	0.03	67.80	27.20	113.00	66.05	19.88	0.05	1.43458	2.05307	1.81986	1.29850	
pha14c	1	11	132120	PCT leach- Pha 14C-1	16.58	12.05	40.50	0.03	69.05	27.63	115.08	67.50	20.08	0.05	1.44131	2.06101	1.82932	1.30270	
ARM	1	12	132119	PCT leachate - ARM-3	10.90	8.54	23.03	0.03	37.82	18.16	63.03	38.39	14.24	0.05	1.25920	1.79957	1.58417	1.15347	
ARM	1	13	132118	PCT leachate - ARM-2	10.55	8.40	22.46	0.03	37.04	17.58	61.73	37.43	13.99	0.05	1.24503	1.79052	1.57317	1.14593	
ARM	1	14	132117	PCT leachate - ARM-1	12.99	9.39	25.71	0.03	40.17	21.65	66.95	42.84	15.65	0.05	1.33550	1.82576	1.63189	1.19438	
blank	1	15	132116	PCT leachate - Blk-2	0.04	0.00	0.12	0.03	0.06	0.07	0.10	0.20	0.01	0.05	-1.17608	-1.00729	-0.69536	-2.30102	
blank	1	16	132115	PCT leachate - Blk-1	0.04	0.00	0.12	0.03	0.07	0.07	0.11	0.20	0.01	0.05	-1.17608	-0.96523	-0.70626	-2.30102	
std	1	17	132130	PCT leachate - Std-3	19.26	9.63	81.26	0.03	48.88	19.26	48.88	81.26	9.63	0.03	1.28470	1.68916	1.90989	0.98345	

Notes:

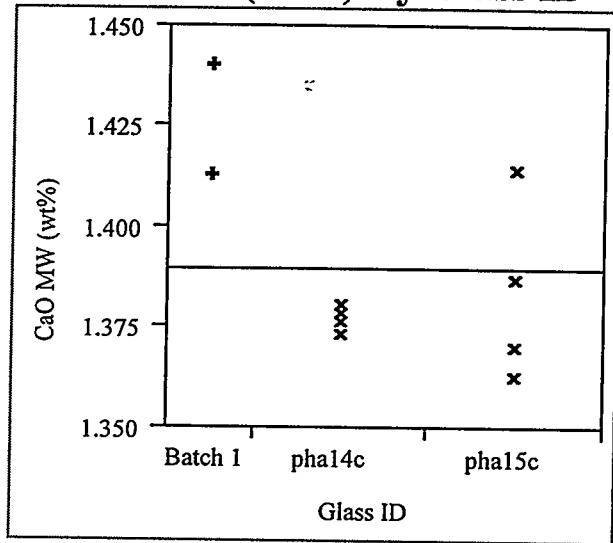
(1). Values that are below detection (indicated by a "<") were converted to their detection limit.

Exhibit B.1: Measurements by Glass Sample ID by Oxide for pha14c and pha15c

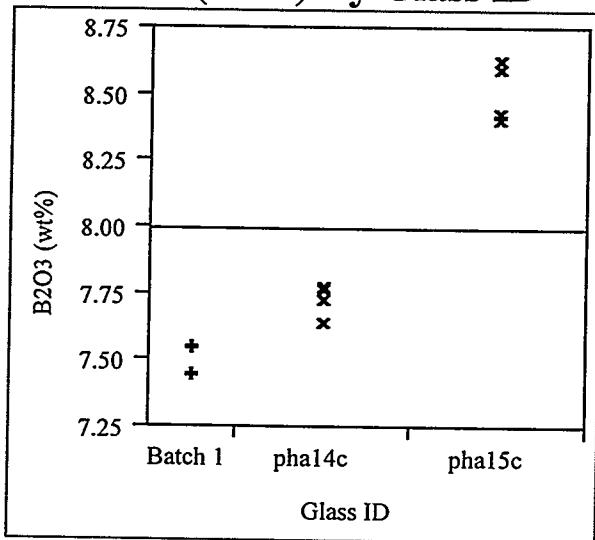
Al₂O₃ (wt%) By Glass ID



CaO MW (wt%) By Glass ID



B₂O₃ (wt%) By Glass ID



Ca pf (wt%) By Glass ID

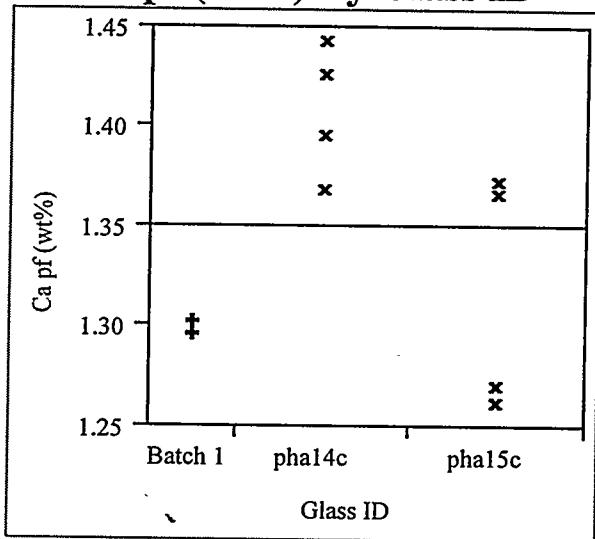


Exhibit B.1: Measurements by Glass Sample ID by Oxide for pha14c and pha15c

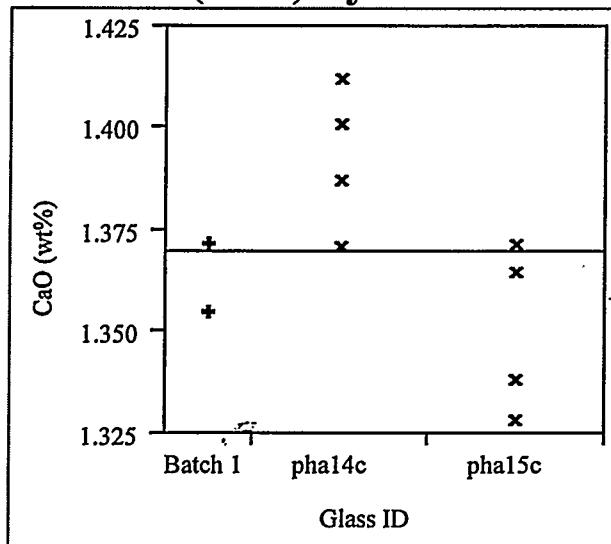
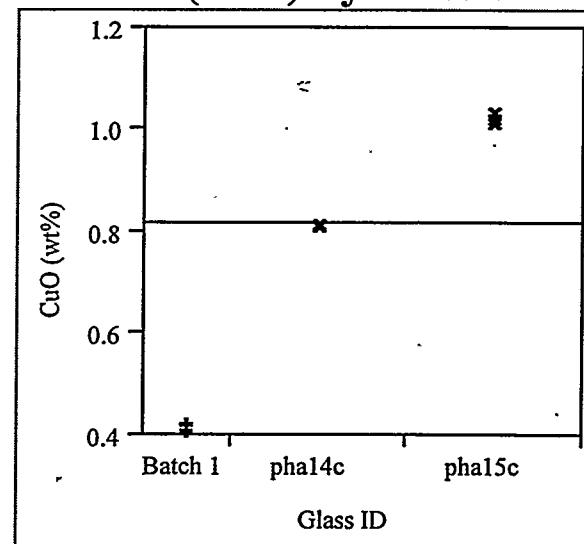
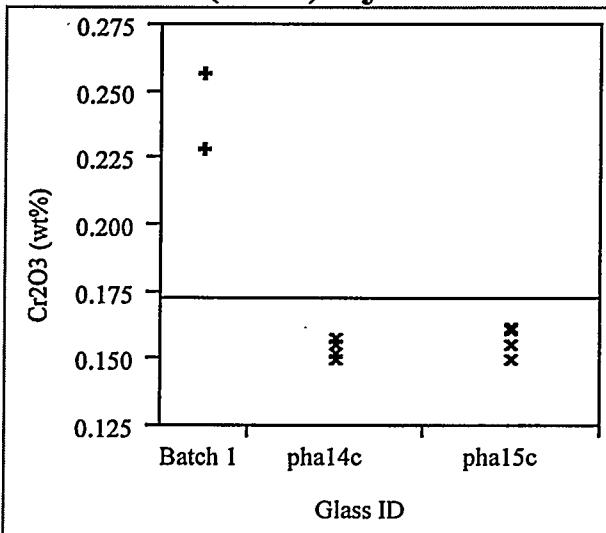
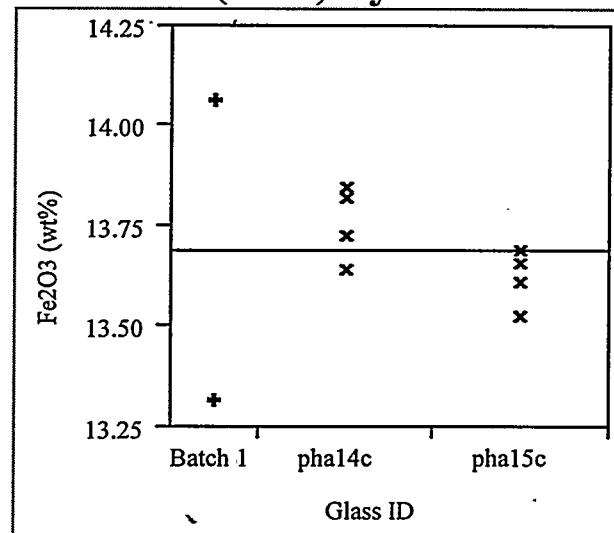
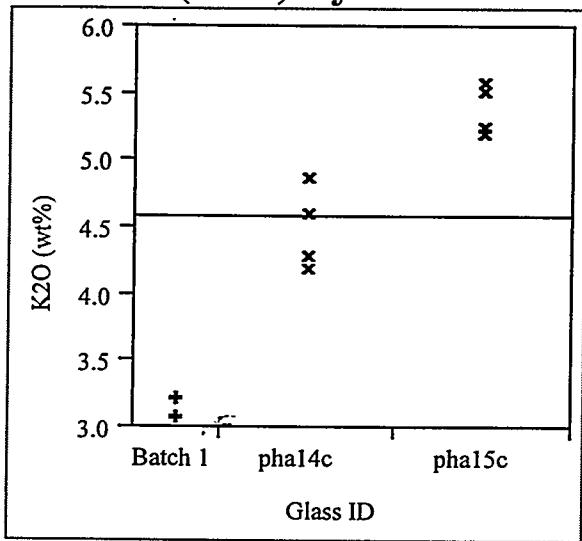
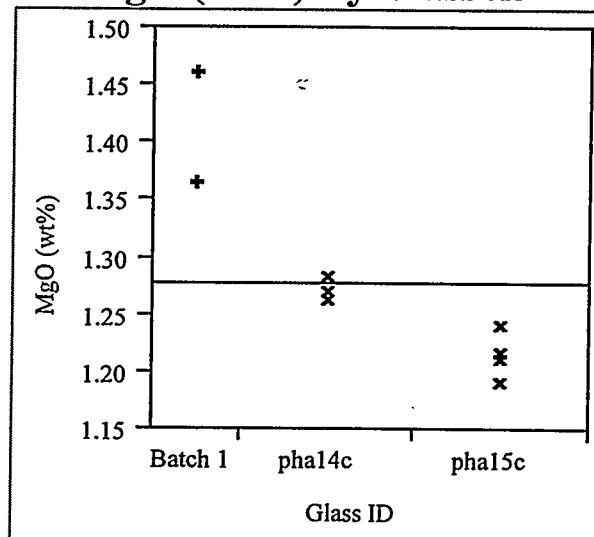
CaO (wt%) By Glass ID**CuO (wt%) By Glass ID****Cr₂O₃ (wt%) By Glass ID****Fe₂O₃ (wt%) By Glass ID**

Exhibit B.1: Measurements by Glass Sample ID by Oxide for pha14c and pha15c

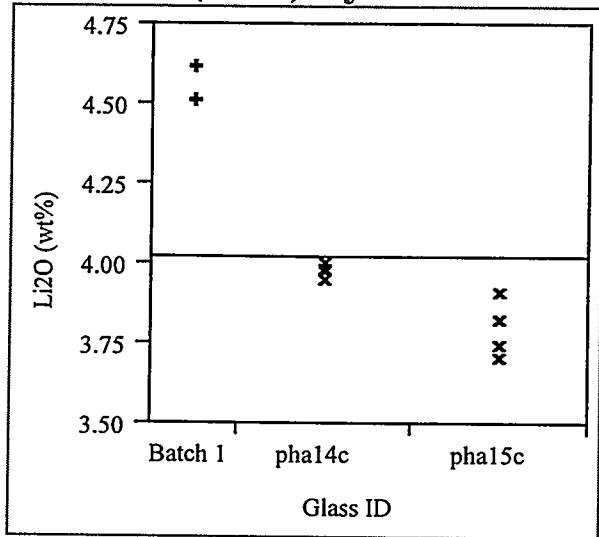
K2O (wt%) By Glass ID



MgO (wt%) By Glass ID



Li2O (wt%) By Glass ID



MnO (wt%) By Glass ID

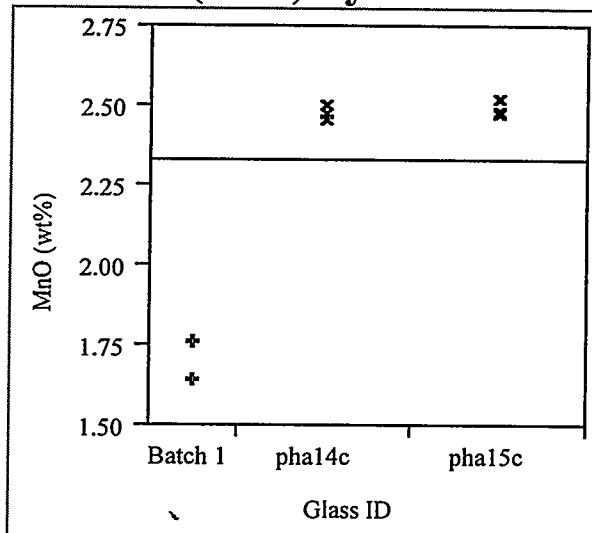
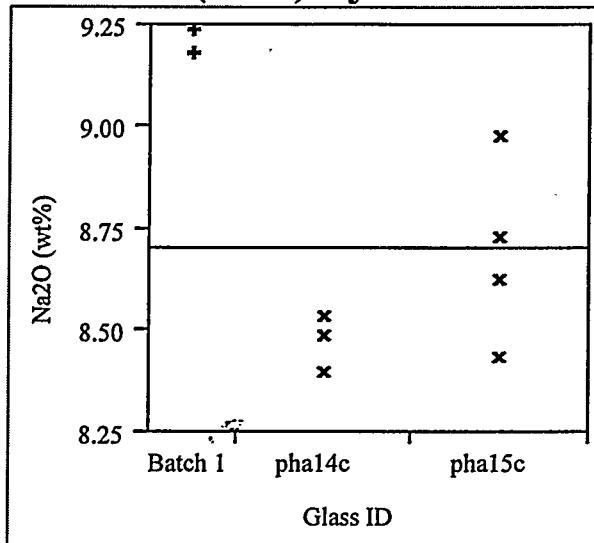
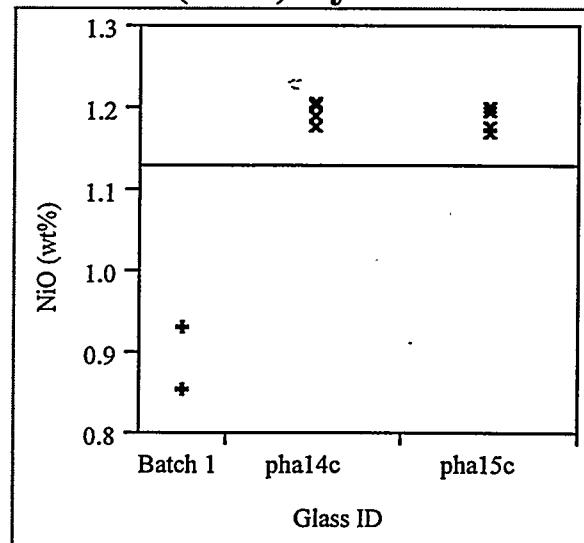


Exhibit B.1: Measurements by Glass Sample ID by Oxide for pha14c and pha15c

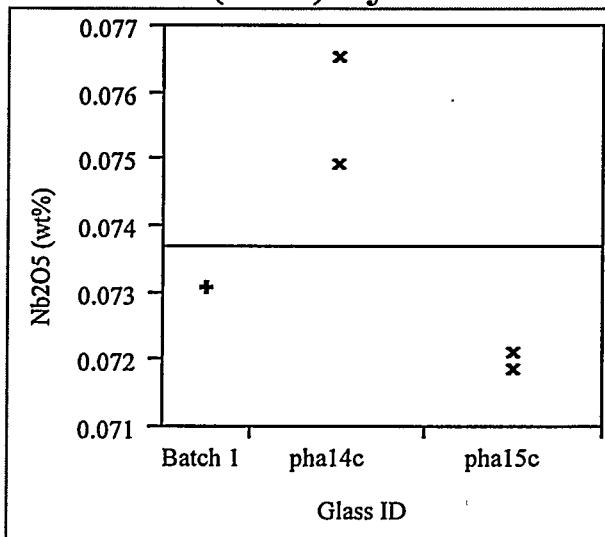
Na₂O (wt%) By Glass ID



NiO (wt%) By Glass ID



Nb₂O₅ (wt%) By Glass ID



SiO₂ MW (wt%) By Glass ID

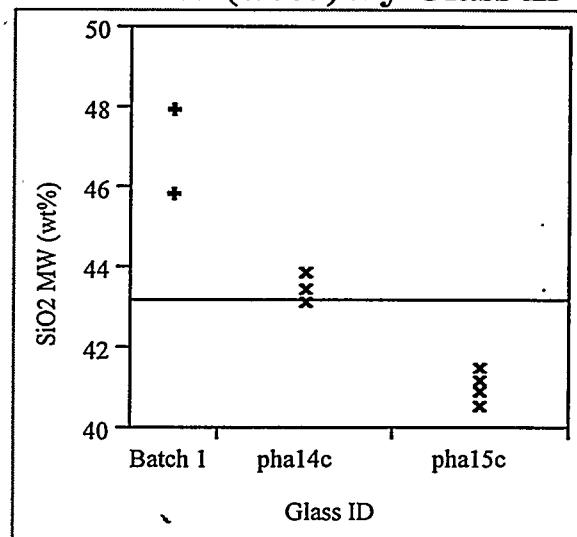
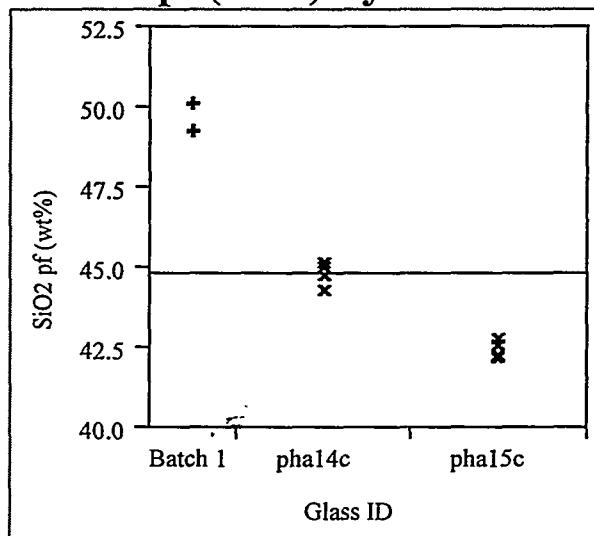
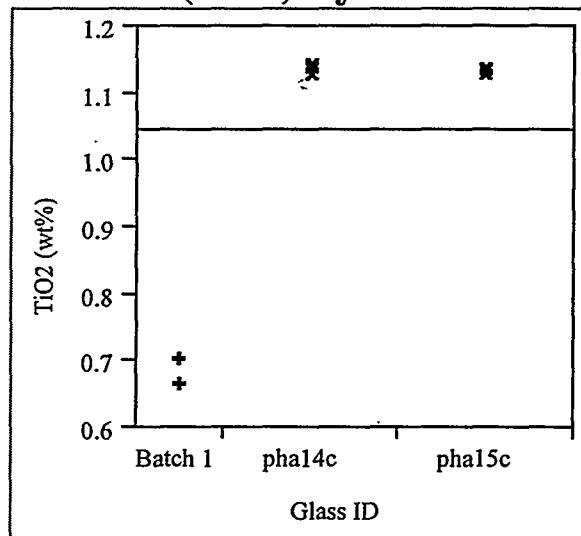


Exhibit B.1: Measurements by Glass Sample ID by Oxide for pha14c and pha15c

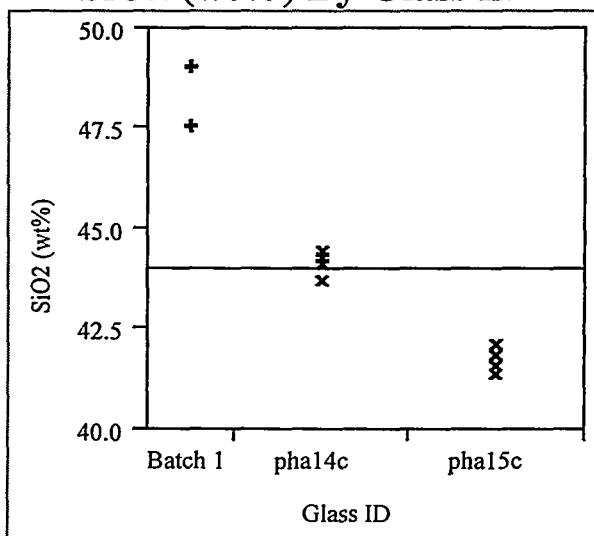
SiO₂ pf (wt%) By Glass ID



TiO₂ (wt%) By Glass ID



SiO₂ (wt%) By Glass ID



U₃O₈ (wt%) By Glass ID

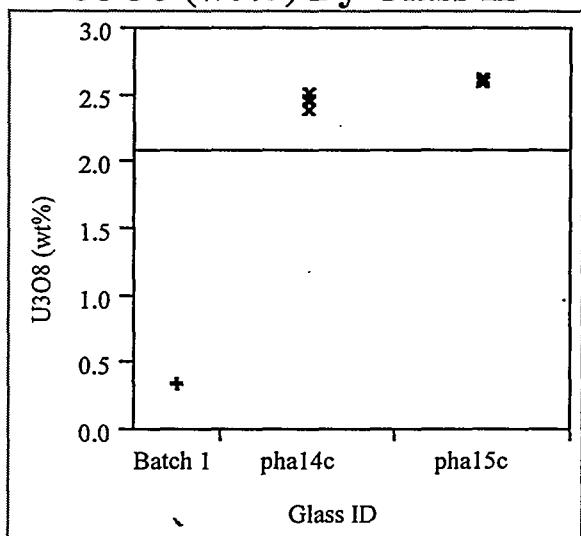
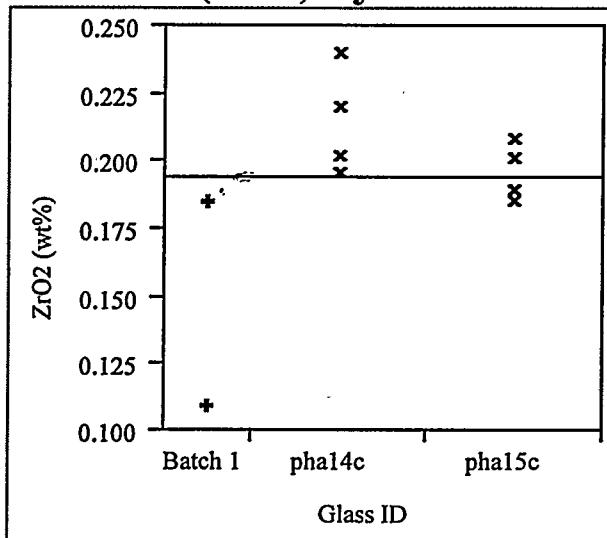
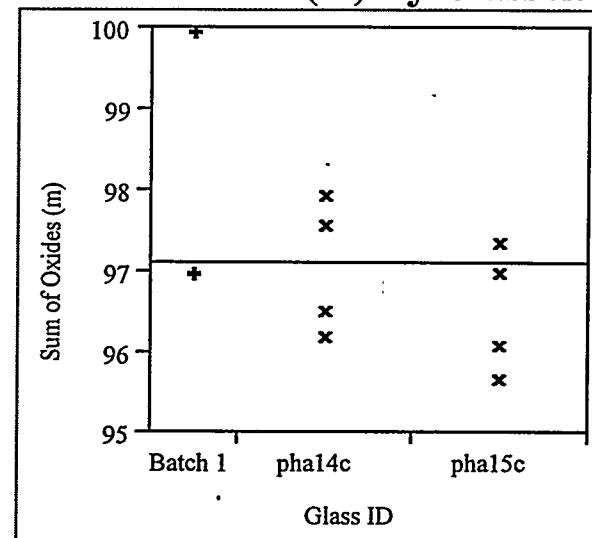


Exhibit B.1: Measurements by Glass Sample ID by Oxide for pha14c and pha15c

ZrO₂ (wt%) By Glass ID



Sum of Oxides (m) By Glass ID



**Exhibit B.2: Plots of the Leachate Concentrations by Sample ID by Element
(with and without the EA and Blank samples)**

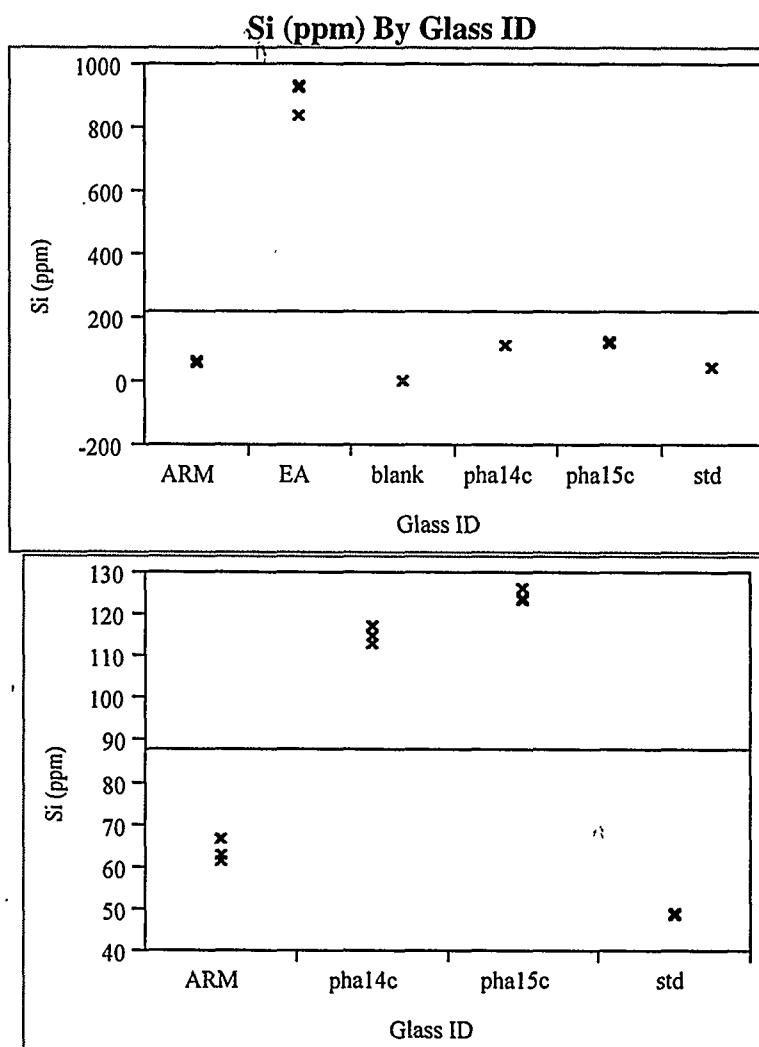
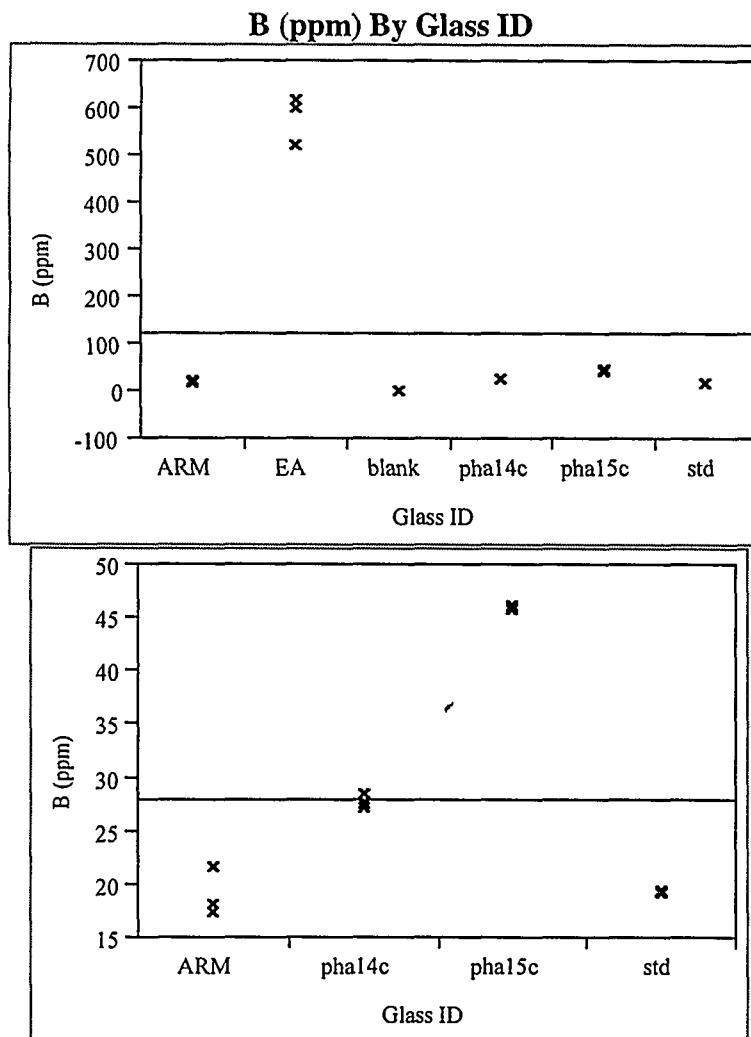
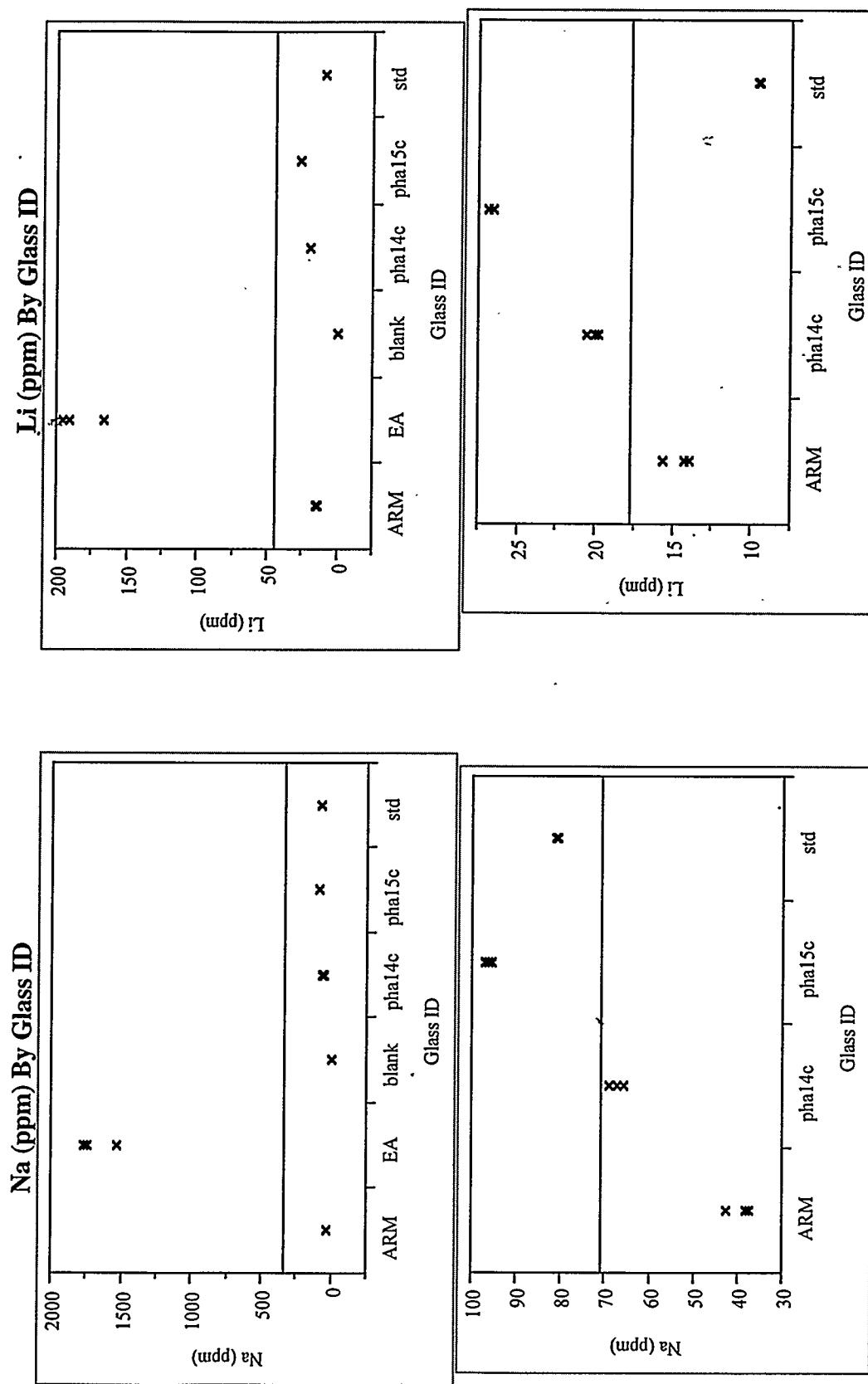


Exhibit B.2: Plots of the Leachate Concentrations by Sample ID by Element
(with and without the EA and Blank samples)



Distribution

J. L. Barnes, 704-3N
N. E. Bibler, 773-A
D. F. Bickford, 773-43A
K. G. Brown, 704-1T
J. T. Carter, 704-3N
J. J. Connelly, 773-41A
A. D. Cozzi, 77-43A
D. A. Crowley, 773-43A
T. B. Edwards, 773-42A
H. H. Elder, 704-S
S. D. Fink, 773-A
J. R. Harbour, 773-43A
E. W. Holtzscheiter, 773-A
R. A. Jacobs, 704-3N
C. M. Jantzen, 773-A
R. T. Jones, 704-3N
D. P. Lambert, 704-1T
L. F. Landon, 704-1T
S. L. Marra, 704-25S
D. B. Moore-Shedrow, 773-A
L. M. Papouchado, 773-A
D. K. Peeler, 773-43A
J. A. Pike, 704-3N
K. J. Rueter, 704-3N
R. F. Schumacher, 773-43A
M. E. Smith, 773-43A
T. K. Snyder, 704-1T
P. C. Suggs, 704-196N
W. L. Tamosaitis, 773-A
R. C. Tuckfield, 773-43A
R. J. Workman, 773-A
TIM (4 copies), 703-43A