WSRC-STI-2006-00009
Revision 0

DURABILITY AND NEPHELINE CRYSTALLIZATION STUDY FOR HIGH LEVEL WASTE (HLW) SLUDGE BATCH 4 (SB4) GLASSES FORMULATED WITH FRIT 503

K.M. Fox
T.B. Edwards
D.K. Peeler
D.R. Best
I.A. Reamer
R.J. Workman

June 2006

Process Science and Engineering Section
Savannah River National Laboratory
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under
Contract Number DEAC09-96SR18500

DISCLAIMER

Printed in the United States of America

Prepared For
U.S. Department of Energy

The Savannah River National Laboratory is operated for the U.S. Department of Energy by Washington Savannah River Company.

WSRC-STI-2006-00009
Revision 0

Keywords: *high level waste, glass, durability, crystallization, nepheline*

Retention: *permanent*

DURABILITY AND NEPHELINE CRYSTALLIZATION STUDY FOR HIGH LEVEL WASTE (HLW) SLUDGE BATCH 4 (SB4) GLASSES FORMULATED WITH FRIT 503

K.M. Fox
T.B. Edwards
D.K. Peeler
D.R. Best
I.A. Reamer
R.J. Workman

June 2006

Process Science and Engineering Section
Savannah River National Laboratory
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under
Contract Number DEAC09-96SR18500

REVIEWS AND APPROVALS

AUTHORS:

K.M. Fox, Materials Science and Technology Section	Date
T.B. Edwards, Statistical Consulting Section	Date
D.K. Peeler, Process Science and Engineering Section	Date
D.R. Best, Process Science and Engineering Section	Date
I.A. Reamer, Process Science and Engineering Section	Date
R.J. Workman, Process Science and Engineering Section	Date

TECHNICAL REVIEWER:

C.M. Jantzen, Process Science and Engineering Section	Date

APPROVERS:

R.E. Edwards, Manager, Process Science and Engineering Section	Date
C.C. Herman, Manager, Process Engineering Technology Group	Date
J.E. Occhipinti, Manager, DWPF Process Engineering	Date

EXECUTIVE SUMMARY

The Defense Waste Processing Facility (DWPF) is about to process High Level Waste (HLW) Sludge Batch 4 (SB4). This sludge batch is high in alumina and nepheline can crystallize readily depending on the glass composition. Large concentrations of crystallized nepheline can have an adverse effect on HLW glass durability. Several studies have been performed to study the potential for nepheline formation in SB4. The Phase 3 Nepheline Formation study of SB4 glasses examined sixteen different glasses made with four different frits. Melt rate experiments were performed by the Process Science and Engineering Section (PS&E) of the Savannah River National Laboratory (SRNL) using the four frits from the Phase 3 work, plus additional high B_2O_3/high Fe_2O_3 frits. Preliminary results from these tests showed the potential for significant improvements in melt rate for SB4 glasses using a higher B_2O_3-containing frit, particularly Frit 503. The main objective of this study was to investigate the durability of SB4 glasses produced with a high B_2O_3 frit likely to be recommended for SB4 processing. In addition, a range of waste loadings (WLs) was selected to continue to assess the effectiveness of a nepheline discriminator in predicting concentrations of nepheline crystallization that would be sufficient to influence the durability response of the glass. Five glasses were selected for this study, covering a WL range of 30 to 50 wt% in 5 wt% increments.

The Frit 503 glasses were batched and melted. Specimens of each glass were heat-treated to simulate cooling along the centerline of a DWPF-type canister (ccc) to gauge the effects of thermal history on product performance. Visual observations on both quenched and ccc glasses were documented. A representative sample from each glass was submitted to the SRNL Process Science Analytical Laboratory (PSAL) for chemical analysis to confirm that the as-fabricated glasses corresponded to the defined target compositions. The Product Consistency Test (PCT, ASTM C1285) was performed in triplicate on each Frit 503 quenched and ccc glass to assess chemical durability. The experimental test matrix also included the Environmental Assessment (EA) glass and the Approved Reference Material (ARM-1) glass. Representative samples of all the ccc glasses were examined for homogeneity visually and by X-ray diffraction (XRD) analysis.

Chemical composition measurements indicated that the experimental glasses were close to their target compositions. PCT results showed that all of the Fit 503 quenched glasses had an acceptable durability compared to the EA benchmark glass. The durability of one of the ccc glasses, NEPHB-04, was statistically greater than its quenched counterpart. However, this was shown to be of little practical significance, as the durability of the NEPHB-04 ccc glass was acceptable when compared to the durability of the EA benchmark glass.

Visual observations and PCT results indicated that all of the Frit 503 quenched glasses were free of any crystallization that impacts durability. For the ccc glasses, XRD results indicated that the lower WL glasses (30 to 40 wt%) were amorphous, which was consistent with visual observations and PCT responses. The higher WL glasses (45 and 50 wt%) were shown by XRD to contain spinel (trevorite, $NiFe_2O_4$). It is possible that some of the other high WL glasses also contained some nepheline, but that the amount of nepheline crystallization was below the detection limit (0.5 vol%) associated with XRD.

The results indicate that Frit 503 is a good candidate for SB4 processing, based on chemical durability of homogeneous and devitrified glasses over a WL range of 30 – 50%. It should be noted that the higher WL glasses would not be fit for processing in DWPF as they exceed other process related criteria (such as liquidus temperature). However, this is only one of many factors influencing the frit selection. Melt rate and the final SB4 composition are also important factors in frit selection. Additional melt rate studies are currently underway, and the final composition projection for SB4 is expected shortly.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ACRONYMS

AD	Analytical Development
ANOVA	ANalysis Of VAriance
ARM	Approved Reference Material
ARP	Actinide Removal Process
ASTM	American Society for Testing and Materials
bc	bias-corrected
CBU	Closure Business Unit
ccc	centerline canister cooled
DWPF	Defense Waste Processing Facility
EA	Environmental Assessment
HLW	High Level Waste
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
LM	Lithium-Metaborate
LWO	Liquid Waste Operations
MAR	Measurement Acceptability Region
PCCS	Product Composition Control System
PCT	Product Consistency Test
PF	Peroxide Fusion
PSAL	Process Science Analytical Laboratory
SB4 / SB5	Sludge Batch 4 / Sludge Batch 5
SME	Slurry Mix Evaporator
SRL	Savannah River Laboratory
SRNL	Savannah River National Laboratory
T_L	liquidus temperature
WL	Waste Loading (weight percent)
XRD	X-Ray Diffraction

1.0 Introduction

Crystallization (or devitrification) is an important factor in the processing and performance of nuclear waste glass. In terms of processing, the Defense Waste Processing Facility (DWPF) uses a liquidus temperature (T_L) model[1-3] and an imposed T_L limit for feed acceptability to avoid bulk devitrification within the melter. In terms of performance of the glass waste form, the impact of devitrification on durability depends on the type and extent of crystallization.

Numerous studies[4-11] have assessed the potential for devitrification in various high level waste (HLW) glasses and its impact on durability. These studies generally agree that the impact of devitrification on durability is dependent upon the type and extent of crystallization. For example, a strong increase in the rate of glass dissolution (or decrease in durability) was observed in studies[6, 11-13] of glasses that formed aluminum-containing crystals, such as $NaAlSiO_4$ (nepheline), $LiAlSi_2O_6$, or crystalline SiO_2. The report by Jantzen and Bickford[11] also indicated that the formation of spinel had little or no effect on the durability of Savannah River Laboratory (SRL) 165- or SRL 131-based glasses, while the formation of acmite produced a small but noticeable increase in the rate of dissolution of the matrix glass. The impact of devitrification on durability is complex and depends on several interrelated factors including the change in residual glass composition, the development of internal stress or microcracks, and preferential attack at the glass – crystal interface.

The next sludge batch to be processed by DWPF, Sludge Batch 4 (SB4), is projected to contain a relatively large concentration of Al_2O_3.[14] While the addition of Al_2O_3 to borosilicate glasses generally enhances the durability of the waste form (through creation of network-forming tetrahedral Na^+-$[AlO_{4/2}]^-$ pairs), nepheline formation, which depends in part on the Al_2O_3 content, can result in severe deterioration of the chemical durability of the glass through residual glass compositional changes and microcracking. Three moles of glass forming oxides (Al_2O_3 and $2SiO_2$) are removed from the continuous glass phase per each mole of Na_2O as nepheline crystallizes. Therefore, nepheline formation produces an Al_2O_3 and SiO_2 deficient continuous glass matrix (relative to the same composition without crystallization) which reduces the durability of the final product. The magnitude of the reduction ultimately depends on the extent (volume fraction) of crystallization.

Li *et al.*[9, 15] indicated that sodium alumino-borosilicate glasses are prone to nepheline crystallization if their compositions projected on the Na_2O-Al_2O_3-SiO_2 ternary fall within or close to the nepheline primary phase field. In particular, glasses with $SiO_2/(SiO_2+Na_2O+Al_2O_3) > 0.62$, where the chemical formulae stand for the mass fractions in the glass, do not tend to precipitate nepheline as a primary crystalline phase.

Initial composition projections of SB4[14] indicated that the sludge will be enriched in Al_2O_3 relative to the Al_2O_3 concentrations of previous sludge batches processed through the DWPF. Candidate frits were identified which ranged in Na_2O concentration from 8-13% by mass for the initial SB4 composition projections.[16] The combination of high Al_2O_3 and Na_2O concentrations, coupled with lower SiO_2 concentrations as waste loadings are increased (given the primary source of SiO_2 is from the frit), shifts the overall glass compositions toward the nepheline phase field, raising the potential for nepheline crystallization. Therefore, strategic frit development efforts[17] have been made to suppress the development of nepheline formation by lowering the Na_2O content while increasing B_2O_3, Fe_2O_3, and/or Li_2O concentration in the frit.

Peeler *et al.*[18, 19] provided insight into the potential impact of nepheline formation on SB4 glasses based on the Lilliston[14] SB4 composition projections. In that study (referred to as Phase 1), twelve SB4-based glasses were fabricated (only two of which were prone to nepheline formation using the 0.62 value of Li *et al.*[15] as a guide) and the durability of each was measured. The results indicated that all the glasses in the Phase 1 study (both quenched and centerline canister cooled (ccc)) had a durability as defined by the Product Consistency Test (PCT)[20] that was acceptable (lower than the EA benchmark glass). The two glasses prone to nepheline formation (NEPH-01 and NEPH-02) had a statistically significant difference in PCT response between the quenched and ccc versions, but the durability of the ccc glasses, while decreased, was still considerably better than that of the EA glass.[21] When the PCT responses were coupled with the X-ray diffraction (XRD) results and/or visual observations, it was concluded that the formation of nepheline in these glasses did have a negative impact on durability, though in this case the impact was not of practical concern. The results of the Phase 1 study suggested that the 0.62 value, as proposed by Li *et al.*,[15] appeared to be a reasonable guide to monitor the potential for nepheline formation in the alumino-borosilicate based SB4 glass system.

After issuance of the Phase 1 report, revised composition projections from the Closure Business Unit (CBU) for SB4 were issued.[22, 23] In response to these revised projections, candidate frits whose operating windows (i.e., waste loading intervals that meet Product Composition Control System (PCCS) Measurement Acceptability Region (MAR) criteria) are robust to and/or selectively optimal for these sludge options were identified via a paper study.[24] The results of the paper study indicated that candidate frits were available for the various SB4 options presented and relatively large operating windows were provided. In addition, the 0.62 value for the nepheline discriminator was used as a screening tool to evaluate the potential impact of nepheline formation on the projected operating windows. The results of applying the nepheline discriminator[24] indicated that access to higher WLs for almost all SB4 frit – sludge options was restricted. That is, a relatively large WL interval was available in which all PCCS MAR criteria were satisfied except when the nepheline discriminator value was invoked. This suggested possible composition regions associated with crystallization and its potentially adverse impact on durability. Therefore, the value of the nepheline discriminator was challenged to determine if access to those higher WLs could be regained without compromising durability.

Phase 2 of the nepheline crystallization study[25] was then undertaken to complement the Phase 1 work[19] by selecting glasses to cover WLs over which nepheline was the only criterion restricting acceptability. The primary difference between the Phase 1 and Phase 2 nepheline studies was that Phase 2 challenged the nepheline discriminator for all glasses tested – not just a few select glasses as in Phase 1. In order to meet this objective, WLs of ~ 40% or higher were targeted for the Phase 2 glasses,[26] whereas 40% was the maximum WL used during Phase 1. Twenty eight glasses, encompassing five different frit compositions (Frit 320, Frit 417, Frit 425, Frit 426, and Frit 418), were fabricated and tested following the experimental methods used in Phase 1.

All of the Phase 2 quenched glasses had normalized boron releases of less than 1.19 g/L, which is approximately an order of magnitude better than the EA benchmark glass.[21] However, the potential for crystallization is suppressed kinetically in quenched glasses. That is, the glasses may have been prone to nepheline formation but the rapid cooling limited the formation of nepheline (or other crystalline phases). For the Phase 2 ccc glasses, visual observations suggested that as the targeted WL within a specific frit – sludge system was increased, the degree of crystallization became more extensive. This is expected, as the slower cooling rate provides the kinetic path for a glass with a composition that is thermodynamically favorable for nepheline formation (i.e., a composition that falls within the nepheline primary phase field) to devitrify. XRD results indicated the presence of nepheline, trevorite ($NiFe_2O_4$), and/or lithium silicate (Li_2SiO_3) in select Phase 2 ccc glasses. Also, the difference between the quenched and ccc PCT response for each specific frit system increased as WL increased. Coupling this trend with the XRD crystallization results, the durability responses as a function of WL were easily explained. As WL increased within a specific frit – sludge system, the durability of the ccc based glasses decreased due to the formation of nepheline and/or lithium silicate. These trends are in agreement with previous observations that the impact on durability is dependent upon the type and extent of crystallization and the resulting change in the residual glass composition.

For Phase 3 of the nepheline study, 16 glasses were selected[27] to complement the earlier work[19, 25] by continuing the investigation into the ability of the nepheline discriminator to predict the occurrence of nepheline crystallization in SB4 glasses and into the impact of such phases on the durability of the SB4 glasses. Four frits (Frit 418, Frit 425, Frit 501, and Frit 502) were used, combined with the most recent composition projection for SB4.[28] A primary objective of the Phase 3 study was to continue to demonstrate the ability of the nepheline discriminator value to adequately predict the nepheline formation potential for specific glass systems of interest. Glasses were selected to cover WLs that tightly bound the nepheline discriminator value of 0.62, with the intent of refining this value to a level of confidence where it could be incorporated into offline administrative controls and/or the PCCS to support Slurry Mix Evaporator (SME) acceptability decisions. In addition, lower WLs (30 – 40%) were targeted which provided consistency with the Phase 2 work.

The results of the Phase 3 study[29] concurred with the earlier phases in that a nepheline discriminator of 0.62 was shown to be the appropriate value for screening out glasses with the potential for nepheline crystallization upon slow cooling (and therefore reduced chemical durability). The results also showed that the nepheline discriminator was successful in screening out the one glass in the study that contained nepheline and would be unpredictable by the ΔG_P model.[30]

Following the Phase 3 study, melt rate experiments were performed using the four frits from the Phase 3 work, and additional high B_2O_3/high Fe_2O_3 frits. Preliminary results from these tests showed the potential for significant improvements in melt rate for SB4 glasses using a higher B_2O_3-containing frit, particularly Frit 503.[31] As Frit 503 / SB4 glasses had not yet been examined experimentally for durability performance, a small study was undertaken, which is the focus of this report. The main objective of this study was to investigate the durability of SB4 glasses produced with a high B_2O_3 frit likely to be recommended (based on the information in hand) for SB4 processing. In addition, a range of WLs were selected to continue to assess the effectiveness of a nepheline discriminator value of 0.62 in predicting nepheline crystallization sufficient to influence the durability response of the glass.

The results of this study will provide valuable input for the frit development efforts and subsequent feedback to Liquid Waste Operations (LWO) regarding the viability of a high B_2O_3 frit option (Frit 503) for SB4 vitrification. Additional data provided through other studies, such as the continuing melt rate experiments, will also influence the frit recommendation decision for SB4. This work was initiated by a Technical Task Request[32] and is covered by a Technical Task and Quality Assurance Plan.[33]

This page intentionally left blank.

2.0 Experimental Procedure

2.1 Glass selection

Five glass compositions were selected for this study. Only one sludge option, Case 15C Blend 1 (~96 inch SB3 heel, SB4 washed to 1.4 M Na^+ before blending)[28] was used as this option was seen as providing the most likely representation of SB4 at the time this task was initiated. A high B_2O_3 frit (Frit 503) was chosen for this study based on preliminary melt rate experiments[a] and the assessments associated with projected operating windows.[34] The melt rate experiments showed a significant improvement in melt rate for Frit 503 / SB4 glasses compared with earlier, lower B_2O_3 frits. Frit 503, whose composition is given in Table 2-1, was used in the current study. The model-based MAR assessment indicated that the high B_2O_3-based frits suppressed nepheline formation to higher WLs which ultimately resulted in an another property (besides nepheline crystallization) defining the maximum WL attainable via PCCS.[34]

Table 2-1. Composition (as mass fractions) of Frit 503.

B_2O_3	Li_2O	Na_2O	SiO_2
0.14	0.08	0.04	0.74

The frit and sludge were combined at five WL levels to examine the durability and potential for nepheline crystallization of Frit 503 / SB4 glasses over a range of WLs. The WLs utilized covered a range likely to be processed at DWPF (i.e., 30 – 40% WL), as well as higher WLs to challenge nepheline formation (i.e., 45 and 50% WL). It should be noted that at these higher WLs, other properties (in particular T_L) may not have been acceptable, but in order to meet study objectives (challenge nepheline formation and/or refine the discriminator value), other processing criteria were ignored. More specifically, given the higher B_2O_3 and lower Na_2O concentrations of Frit 503, T_L predictions limit WLs with Case 15C Blend 1 to 43% or lower. At 44% WL, T_L predictions exceed the control limits in PCCS. Therefore, DWPF would be restricted from processing the 45 and 50% WL glasses – although these WLs will be targeted in this study.

The target compositions of the five Frit 503 / SB4 glasses (NEPHB-01 through NEPHB-05) are listed in Table 2-2. Values of the nepheline discriminator, calculated using the target compositions, are also given.

---

[a] See WSRC-NB-2006-00017, page 40, for details and results of the melt rate experiments.

Table 2-2. Target Compositions of the Frit 503 Glasses (in wt%).

Glass ID	NEPHB-01	NEPHB-02	NEPHB-03	NEPHB-04	NEPHB-05
%WL	30	35	40	45	50
nepheline discriminator	0.759	0.723	0.685	0.646	0.606
Al_2O_3	7.4417	8.6820	9.9223	11.1626	12.4029
B_2O_3	9.8000	9.1000	8.4000	7.7000	7.0000
BaO	0.0378	0.0441	0.0504	0.0567	0.0630
CaO	0.7162	0.8356	0.9550	1.0743	1.1937
Ce_2O_3	0.0449	0.0524	0.0599	0.0674	0.0749
Cr_2O_3	0.0636	0.0742	0.0848	0.0954	0.1061
CuO	0.0179	0.0209	0.0239	0.0269	0.0299
Fe_2O_3	7.9697	9.2979	10.6262	11.9545	13.2827
K_2O	0.1032	0.1204	0.1376	0.1548	0.1721
La_2O_3	0.0325	0.0379	0.0434	0.0488	0.0542
Li_2O	5.6000	5.2000	4.8000	4.4000	4.0000
MgO	0.7486	0.8734	0.9982	1.1229	1.2477
MnO	1.6440	1.9180	2.1920	2.4660	2.7401
Na_2O	9.4239	10.3279	11.2318	12.1358	13.0398
NiO	0.4731	0.5520	0.6308	0.7097	0.7886
PbO	0.0271	0.0317	0.0362	0.0407	0.0452
SO_4^{2-}	0.4014	0.4683	0.5352	0.6021	0.6690
SiO_2	53.0340	49.5396	46.0453	42.5509	39.0566
ThO_2	0.0197	0.0230	0.0263	0.0296	0.0329
TiO_2	0.0080	0.0093	0.0107	0.0120	0.0133
U_3O_8	2.2921	2.6741	3.0561	3.4381	3.8201
ZnO	0.0293	0.0342	0.0390	0.0439	0.0488
ZrO_2	0.0711	0.0830	0.0948	0.1067	0.1185
Sum	100.0000	100.0000	100.0000	100.0000	100.0000

2.2 Glass Fabrication

Each Frit 503 glass was prepared from the proper proportions of reagent-grade metal oxides, carbonates, boric acid, and salts in 150 g batches.[35] The raw materials were thoroughly mixed and placed into a 95% Platinum/5% Gold 250 mL crucible. The batch was placed into a high-temperature furnace at the target melt temperature of 1150°C.[36] After an isothermal hold at 1150°C for 1.0 h, the crucible was removed from the furnace. The glass was poured onto a clean stainless steel plate and allowed to air cool (quench). The glass pour patty was used as a sampling stock for the various property measurements (i.e., chemical composition and durability).

Approximately 25 g of each glass was heat-treated to simulate cooling along the centerline of a DWPF-type canister[8] to gauge the effects of thermal history on product performance. This cooling schedule is referred to as the ccc curve. Visual observations on both quenched and ccc glasses were documented.[a]

---

[a] WSRC-NB-2006-00016 contains the visual observations of the quenched and ccc glasses as well as the results of the XRD and PCT analyses for the Frit 503 glasses.

2.3 Measurement of the Properties and Performance of the Glasses

This section provides a general discussion of the chemical composition analyses, the PCTs, and the XRD analyses of the Frit 503 glasses.

2.3.1 *Compositional Analysis*

To confirm that the as-fabricated glasses corresponded to the defined target compositions, a representative sample from each glass was submitted to the SRNL Process Science Analytical Laboratory (PSAL) for chemical analysis under the auspices of an analytical plan. The plan (see Appendix A) identified the cations to be analyzed and the dissolution techniques (i.e., sodium peroxide fusion [PF] and lithium-metaborate [LM]) to be used. The samples prepared by LM were used to measure barium (Ba), calcium (Ca), cerium (Ce), chromium (Cr), copper (Cu), potassium (K), lanthanum (La), magnesium (Mg), manganese (Mn), sodium (Na), lead (Pb), sulfur (S), thorium (Th), titanium (Ti), zinc (Zn), and zirconium (Zr) concentrations. Samples prepared by PF were used to measure aluminum (Al), boron (B), iron (Fe), lithium (Li), nickel (Ni), silicon (Si), and uranium (U) concentrations. Each glass was prepared in duplicate for each cation dissolution technique (PF and LM). All of the prepared samples were analyzed (twice for each element of interest) by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) with the instrumentation being re-calibrated between the duplicate analyses. The analytical plan was developed in such a way as to provide the opportunity to evaluate potential sources of error. Glass standards were also intermittently measured to assess the performance of the ICP-AES instrument over the course of these analyses.

2.3.2 *SO_4^{2-} Solubility*

Although not a primary focus of this study, SO_4^{2-} solubility is of concern for SB4 glass systems. The applicability of the current 0.6 wt% SO_4^{2-} limit (established for the Frit 418 – SB3 system[37]) to SB4 was investigated. From Table 2-2, the targeted SO_4^{2-} concentrations in the Frit 503 glasses range from 0.401 to 0.669 wt%. Previous tests have suggested that the use of reagent grade raw materials is conservative with respect to SO_4^{2-} retention and/or volatility.[a] Since the Frit 503 glasses have both high SO_4^{2-} concentrations and are batched from reagent grade raw materials, the ability of the glasses to retain the targeted SO_4^{2-} concentrations will provide valuable insight into the applicability of the current SO_4^{2-} limit to SB4. Both visual observations (i.e., formation of a salt layer on the surface of the glass indicating that SO_4^{2-} limit has been exceeded) and a comparison of measured versus targeted SO_4^{2-} concentrations were used to support this assessment.

2.3.3 *Product Consistency Test (PCT)*

The PCT[20] was performed in triplicate on each Frit 503 quenched and ccc glass to assess chemical durability. Also included in the experimental test matrix was the EA glass,[21] the Approved Reference Material (ARM-1) glass, and blanks from the sample cleaning batch. Samples were ground, washed, and prepared according to the standard procedure.[20] Approximately fifteen milliliters of Type I American Society for Testing and Materials (ASTM) water were added to approximately 1.5 g of glass in stainless steel vessels. The vessels were closed, sealed, and placed in an oven at 90 ± 2°C where the samples were maintained for 7 days. Once cooled, the resulting solutions were sampled (filtered and acidified), then labeled and analyzed by PSAL under the auspices of an analytical plan (see Appendix B). The aim of the plan was to provide an opportunity to assess the consistency (repeatability) of the PCT and analytical procedures to evaluate the chemical durability of the Frit 503 glasses. Normalized release rates were calculated based on targeted, measured, and bias-corrected (bc) compositions using the average of the logs of the leachate concentrations.

---

[a] Previous results have indicated that the use of raw materials (reagent grade chemicals) to produce the glasses minimizes SO_4^{2-} volatilization during the fabrication process. Since volatilization is anticipated in slurry-fed melters, this approach will provide a conservative measure of SO_4^{2-} retention in the glass.

As will be discussed in Section 3.0, the PCT results indicated a possible sample labeling error for two of the ccc glasses, NEPHB-04 and NEPHB-05. The PCT was run a second time for all of the ccc glasses to determine whether an error had indeed been made. A second analytical plan was written for these experiments and is included as Appendix C.

2.3.4 *XRD Analyses*

Although visual observations for crystallization were performed and documented, representative samples for all ccc Frit 503 glasses were submitted to Analytical Development (AD) for XRD analyses. The quenched glasses were not submitted for XRD analyses based on visual observations and the PCT responses. Samples were run under conditions providing a detection limit of approximately 0.5 vol%. That is, if crystals (or undissolved solids) were present at 0.5 vol% or greater, the diffractometer would not only be capable of detecting the crystals but would also allow a qualitative determination of the type of crystal(s) present. Otherwise, a characteristically high background devoid of crystalline peaks indicated that the glass product was amorphous, suggesting either a completely amorphous product or that the degree of crystallization was below the detection limit.

3.0 Results and Discussion

3.1 A Statistical Review of the Chemical Composition Measurements for the Frit 503 Glasses

In this section, the measured versus targeted compositions of the five SB4/Frit 503 study glasses (NEPHB-01 through NEPHB-05) are presented and compared. The targeted compositions for these glasses are provided in Table 2-2, as well as Table D1 of Appendix D. A sum of oxides column is provided in these tables as well. Chemical composition measurements for these glasses were conducted by PSAL following the analytical plan provided in Appendix A.

Table D2 in Appendix D provides the elemental concentration measurements derived from the samples prepared using LM digestions, and Table D3 in Appendix D provides the measurements derived from the samples prepared using PF digestions. Measurements for standards (Batch 1 and a uranium standard, U_{std}) that were included in the PSAL analytical plan along with the study glasses are also provided in these two tables.

The elemental concentrations were converted to oxide concentrations by multiplying the values for each element by the gravimetric factor for the corresponding oxide. During this process, an elemental concentration that was determined to be below the detection limit of the analytical procedures used by PSAL was reduced to half of that detection limit as the oxide concentration was determined.

In the sections that follow, the analytical sequences of the measurements are explored, the measurements of the standards are investigated and used for bias-correction, the measurements for each glass are reviewed, the average chemical compositions (measured and bias-corrected) for each glass are determined, and comparisons are made between the measurements and the targeted compositions for the glasses.

3.1.1 *Measurements in Analytical Sequence*

Exhibit D1 in Appendix D provides plots of the measurements generated by the PSAL for samples prepared using the LM method. The plots are in analytical sequence with different symbols and colors being used to represent each of the study and standard glasses. Similar plots for the samples prepared using the PF method are provided in Exhibit D2 in Appendix D. These plots include all of the measurement data from Tables D2 and D3. A review of these plots indicates no significant patterns or trends in the analytical process over the course of these measurements, and there appear to be no obvious outliers in these chemical composition measurements.

3.1.2 *Batch 1 and Uranium Standard Results*

In this section, the PSAL measurements of the chemical compositions of the Batch 1 and uranium standard (U_{std}) glasses are reviewed. These measurements are investigated across the ICP-AES analytical blocks, and the results are used to bias-correct the measurements for the study glasses.

Exhibit D3 in Appendix D provides statistical analyses of the Batch 1 and U_{std} results generated by the LM prep method by block for each oxide of interest. The results include analysis of variance (ANOVA) investigations looking for statistically significant differences between the block means for each of the oxides for each of the standards. The reference values for the oxide concentrations of the standard are given in the header for each set of measurements in the exhibit. The results from the statistical tests for the Batch 1 standard may be summarized as follows: Na_2O and ZnO (a detection limit effect) had measurements that indicate a significant ICP calibration effect on the block averages at the 5% significance level. For the U_{std}, no oxides exhibited a significant ICP-AES calibration effect on the block averages at the 5% significance level.

Exhibit D4 in Appendix D provides a similar set of analyses for the measurements derived from samples prepared via the PF method. Once again, the reference values for the oxide concentrations of the standard are given in the headers for each set of measurements in the exhibit. The results from the statistical tests for the Batch 1 standard may be summarized as follows: only Al_2O_3 had measurements that indicate a significant ICP-AES calibration effect on the block averages at the 5% significance level. For the U_{std}, only U_3O_8 had measurements that indicate a significant ICP-AES calibration effect on the block averages at the 5% significance level.

Thus, some of these results provide incentive for adjusting the measurements by the effect of the ICP-AES calibration. Therefore, the oxide measurements of the study glasses were bias-corrected for the effect of the ICP-AES calibration on each of the analytical blocks. The basis for this bias-correction is presented as part of Exhibits D3 and D4 – the average measurement for Batch 1 for each ICP-AES block for Al_2O_3, B_2O_3, BaO, CaO, Cr_2O_3, CuO, Fe_2O_3, Li_2O, MgO, MnO, Na_2O, NiO, SiO_2, and TiO_2 and the average measurement for U_{std} for each ICP-AES block for U_3O_8. The Batch 1 results served as the basis for bias-correcting all of the oxides (that were bias-corrected) except uranium. The U_{std} results were used to bias-correct for uranium. For the other oxides, the Batch 1 results were used to conduct the bias-correction as long as the reference value for the oxide concentration in the Batch 1 glass was greater than or equal to 0.1 wt%. No bias-correction was conducted for Ce_2O_3, La_2O_3, PbO, SO_4, ThO_2, ZnO, or ZrO_2.

The bias-correction was conducted as follows. For each oxide, let $\overline{a}_{ij}$ be the average measurement for the i^{th} oxide at analytical block j for Batch 1 (or U_{std} for uranium), and let t_i be the reference value for the i^{th} oxide for Batch 1 (or for U_{std} if uranium). (The averages and reference values are provided in Exhibits D3 and D4.) Let $\overline{c}_{ijk}$ be the average measurement for the i^{th} oxide at analytical block j for the k^{th} glass. The bias-adjustment was conducted as follows:

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

Bias-corrected measurements are indicated by a "bc" suffix, and such adjustments were performed for all of the oxides of this study except for Ce_2O_3, La_2O_3, PbO, SO_4, ThO_2, ZnO, and ZrO_2. Both measured and measured "bc" values are included in the discussion that follows. In these discussions bias-corrected values for Ce_2O_3, La_2O_3, PbO, SO_4, ThO_2, ZnO, and ZrO_2 are duplicated as the measured-bc values for completeness (i.e., to allow a sum of oxides to be computed for the bias-corrected results).

3.1.3 *Composition Measurements by Glass Number*

Exhibits D5 and D6 in Appendix D provide plots of the oxide concentration measurements by Glass ID # (including both Batch 1, labeled as glass number 100 and U_{std}, labeled as glass number 200) for the measured and bias-corrected (bc) values for the LM and PF preparation methods, respectively. Different symbols and colors are used to represent the different glasses. These plots show the individual measurements across the duplicates of each preparation method and the two ICP-AES calibrations. A review of the plots presented in these exhibits reveals the repeatability of the four individual oxide values for each glass. Some scatter exists in the B_2O_3, Fe_2O_3, Na_2O, and SiO_2 values, though this scatter should not have a significant impact on the results presented here. No other problems are evident in these plots. More detailed discussions of the average, measured chemical compositions of the study glasses are provided in the sections that follow.

3.1.4 *Measured versus Targeted Compositions*

The four measurements for each oxide for each glass (over both preparation methods) were averaged to determine a representative chemical composition for each glass. These determinations were conducted both for the measured and for the bias-corrected data. A sum of oxides was also computed for each glass based upon both the measured and bias-corrected values. Exhibit D7 in Appendix D provides plots showing results for each glass for each oxide to help highlight the comparisons among the measured, bias-corrected, and targeted values.

Some observations from the plots of Exhibit D7 are offered: For nearly every Frit 503/SB4 study glass, the measured CaO, NiO and ZrO_2 values are slightly less than their respective targeted and bias-corrected concentrations, and the measured ThO_2 and ZnO concentrations are higher than their targets. For NEPHB-01, the measured PbO value is somewhat higher than the target. The measured Fe_2O_3 values are close to the targets for the study glasses, while the bias-corrected values are slightly below the targets. Notice that the targeted sums of oxides for the standard glasses do not sum to 100% due to an incomplete coverage of the oxides in the Batch 1 (glass # 100) and U_{std} (glass # 200) glasses. All of the sums of oxides (both measured and bias-corrected) for the study glasses fall within the interval of 95 to 105 wt%.

Table D4 in Appendix D provides a summary of the average and targeted compositions for the study glasses and standards. Entries in Table D4 show the relative differences between the measured or bias-corrected values and the targeted values. These differences are shaded when they are greater than or equal to 5%. Overall, these comparisons between the measured and targeted compositions suggest only minor difficulties in hitting the targeted compositions for some of the oxides (including NiO and ThO_2) for some of the glasses. These should have no impact on the conclusions drawn to support the objectives of this report.

3.1.5 SO_4^{2-} *retention*

Although not the primary focus of the Frit 503 study, a secondary concern is the potential need to redefine the SO_4^{2-} solubility limit for SB4. The compositional analysis, coupled with the visual observations of the as-fabricated glasses (see Section 3.3.1), will serve as primary indicators to determine whether the current 0.6 wt% SO_4^{2-} limit (established for the Frit 418 – SB3 system[37]) is still applicable for SB4. From Table 2-2, the targeted SO_4^{2-} concentrations in the Frit 503 glasses range from 0.401 wt% (NEPHB-01) to 0.669 wt% (NEPHB-05).

Figure 3-1 summarizes the targeted versus measured SO_4^{2-} concentrations in each glass. The solid line represents the targeted concentrations as noted in Table 2-2. The x's represent the measured SO_4^{2-} concentrations in the glass, while the squares are the measured, bias-corrected values. The data suggest a possible reduction in SO_4^{2-} retention as WL increased. For example, at 30% WL the targeted SO_4^{2-} content was ~0.40 wt% with the measured concentration being ~0.38 wt% (a 0.02 wt% difference), which is within analytical uncertainties as determined by Peeler *et al.*[37] during the Frit 418 – SB3 assessment. At 50% WL, the targeted SO_4^{2-} content was ~0.67 wt% with the measured concentration being ~0.59 wt% (a 0.08 wt% difference). Although there does appear to be a slight reduction in the retention of SO_4^{2-} at the higher WLs, the ability of the NEPHB-05 glass to retain ~0.59 wt% in glass (which when compared to the 0.6 wt% PCCS value is within the ±0.02 wt% measurements uncertainty previously defined) suggests that the 0.6 wt% PCCS value is still applicable. The measured values for the standard glasses are shown to be above the target (zero) due to the detection limit of the ICP-AES instrument.

In addition to the measured SO_4^{2-} concentrations, no signs of a salt layer were evident on any of the Phase 3 glasses upon fabrication (visual observations are discussed in more detail in Section 3.3.1). If the SO_4^{2-} concentration in the SB4 feed to DWPF contains the projected levels, then no issues with SO_4^{2-} solubility are anticipated.

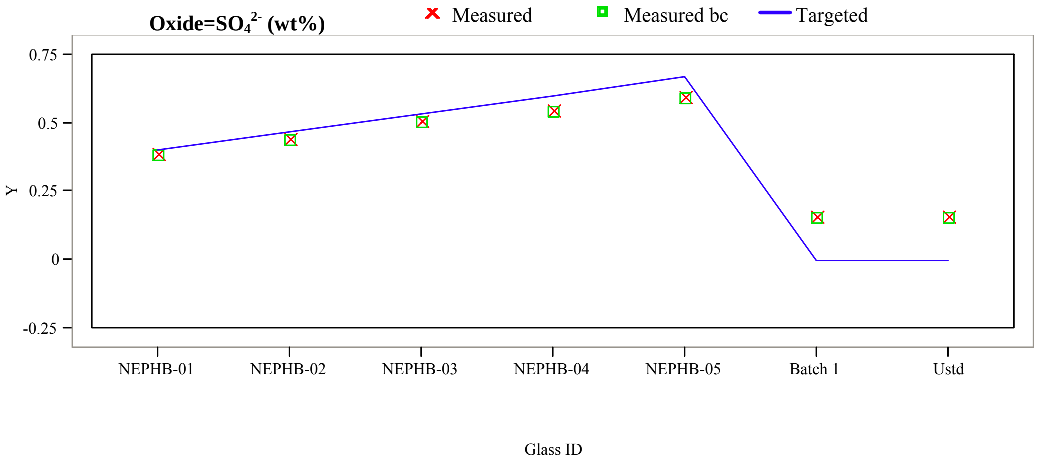


Figure 3-1. Average Measured and Bias-Corrected versus Targeted SO_4^{2-} Values

3.2 A Statistical Review of the PCT Measurements

The Frit 503 / SB4 study glasses, after being batched and fabricated, were subjected to the 7-day PCT[20] to assess their durability. Durability is the critical product quality metric for DWPF glass studies. The PCT was performed in triplicate on the quenched and ccc study glasses, the EA glass, and the ARM glass.

An analytical plan, presented in Appendix B, was provided to the PSAL to support the measurement of the compositions of the solutions resulting from the PCTs. Samples of a multi-element, standard solution were also included in the analytical plan as a check of the accuracy of the ICP-AES instrument used for these measurements. In this and the following sections, the measurements generated by the PSAL for these PCTs are presented and reviewed.

Table E1 in Appendix E provides the elemental leachate concentration measurements determined by the PSAL for the solution samples generated by the PCTs. One of the quality control checkpoints for the PCT procedure is solution-weight loss over the course of the 7-day test. None of these PCT results indicated a solution-weight loss problem. Any measurement in Table E1 below the detection limit of the analytical procedure (indicated by a "<") was replaced by ½ of the detection limit in subsequent analyses. In addition to adjustments for detection limits, the values were adjusted for dilution. The values for the study glasses, the blanks, and the ARM glass in Table E3 were multiplied by a dilution factor of 1.6667. The values for EA were multiplied by a dilution factor of 16.6667. Table E2 in Appendix E provides the resulting dilution corrected measurements.

One of the important objectives of this study is the investigation of the effects of the heat treatment on the glass durability. In the sections that follow, the analytical sequence of the measurements is explored, the measurements of the standards are investigated and used to assess the overall accuracy of the ICP-AES measurement process, the measurements for each glass are reviewed, plots are provided that explore the effects of heat treatment on the PCTs for these glasses, the PCTs are normalized using the compositions (targeted, measured, and bias-corrected) presented in Table D4, and the normalized PCTs are compared to durability predictions for these compositions generated from the current DWPF models.[30]

3.2.1 *Measurements in Analytical Sequence*

Exhibits E1 and E2 in Appendix E provide plots of the leachate (ppm) concentrations in analytical sequence as generated by the PSAL for all of the data and for the data from only the study glasses, respectively. A different color and symbol are used for each study glass or standard. No problems are seen in these plots.

3.2.2 *Results for the Samples of the Multi-Element Solution Standard*

Exhibit E3 in Appendix E provides analyses of the PSAL measurements of the samples of the multi-element solution standard by ICP-AES analytical (or calibration) block. An ANOVA investigating for statistically significant differences among the block averages for these samples for each element of interest is included in these exhibits. These results indicate a statistically significant (at approximately a 5% level) difference among only the Si average measurements over these blocks. However, no bias-correction of the PCT results for the study glasses was conducted. This approach was taken since the triplicate PCTs for a single study glass were placed in different ICP-AES blocks. Averaging the log ppm's for each set of triplicates across the blocks helps to minimize the impact of the ICP-AES calibration effects.

Table 3-1 summarizes the average measurements and the reference values for the 4 primary elements of interest. The results indicate consistent and accurate measurements from the PSAL processes used to conduct these analyses.

Table 3-1. Results from Samples of the Multi-Element Solution Standard

Analytical Block	Avg B (ppm)	Avg Li (ppm)	Avg Na (ppm)	Avg Si (ppm)
1	19.77	9.44	82.00	48.33
2	19.73	9.60	83.13	48.80
3	19.93	9.62	81.07	50.40
Grand Average	19.81	9.55	82.07	49.18
Reference Value	20.0	10.0	81.0	50.0
% difference	-0.9%	-4.5%	1.3%	-1.6%

3.2.3 *Measurements by Glass Number*

Exhibit E4 in Appendix E provides plots of the leachate concentrations for each type of submitted sample: the study glasses and the standards (EA (101), ARM (102), the multi-element solution standard (100), and blanks (103)). Exhibit E5 in Appendix E provides plots of the leachate concentrations for the PCT results of the study glasses. These plots allow for the assessment of the repeatability of the measurements, which suggests some scatter in the triplicate values for some analytes for some of the glasses. Also, note that the results from the two heat treatments are shown for each study glass and that the biggest differences between the two sets of values are evident for NEPHB-04.

3.2.4 *Normalized PCT Results*

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 targeted cation and the bias-corrected cation compositions were also 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 was determined and used for comparison. To accomplish this computation, one must

1. Determine the common logarithm of the elemental parts per million (ppm) leachate concentration for each of the triplicates and each of the elements of interest (these values are provided in Table E2 of Appendix E),

2. Average the common logarithms over the triplicates for each element of interest, and then

Normalizing Using Measured Composition (preferred method)

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

Or Normalizing Using Target Composition

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

Or Normalizing Using Measured Bias-Corrected Composition

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

Exhibit E6 in Appendix E provides scatter plots for these results and offers an opportunity to investigate the consistency in the leaching across the elements for the glasses of this study. All combinations of the normalizations of the PCTs (i.e., those generated using the targeted, measured, and bias-corrected compositional views) and both heat treatments are represented in the series of scatter plots. Consistency in the leaching across the elements is typically demonstrated by a high degree of linear correlation among the values for pairs of these elements. For the study glasses, the ccc results demonstrate a higher degree of correlation (smallest value is 0.9221 for B and Na responses based on the targeted compositions) than do the quenched results (smallest value is 0.6810 for Na and Si responses based on the measured and measured bias-corrected compositions). This may be due to the limited range of PCT responses for the quenched glasses as opposed to the ccc glasses as revealed by the scale of the axes of the two sets of PCT measurements in the scatter plots of Exhibit E6.

Table 3-2 summarizes the normalized PCTs for the glasses of this study. The glasses are listed by glass identifier. It should be noted that the EA elemental releases are slightly lower than those reported by Jantzen *et al.*[21] This has been observed in previous studies and should not raise questions regarding the PCT results obtained in this study. In fact, the ARM glass is used to demonstrate control and a comparison of the ppm values obtained from the ARM (see Table E2 in Appendix E) during this study are within the control chart limits shown by Jantzen *et al.*[30]

Table 3-2. Normalized PCTs by Glass ID/Compositional View

Glass ID	Heat Treatment	Composition	log NL [B(g/L)]	log NL [Li(g/L)]	log NL [Na(g/L)]	log NL [Si(g/L)]	NL B(g/L)	NL Li(g/L)	NL Na(g/L)	NL Si(g/L)
ARM	N/A	reference	-0.2693	-0.2235	-0.2767	-0.5452	0.538	0.598	0.529	0.285
EA	N/A	reference	1.1282	0.8542	1.0266	0.4982	13.435	7.148	10.633	3.149
NEPHB-01	quenched	measured	-0.1940	-0.1239	-0.3418	-0.3525	0.640	0.752	0.455	0.444
NEPHB-01	quenched	measured bc	-0.1779	-0.1272	-0.3225	-0.3561	0.664	0.746	0.476	0.440
NEPHB-01	quenched	targeted	-0.1959	-0.1415	-0.3298	-0.3581	0.637	0.722	0.468	0.438
NEPHB-01	ccc	measured	-0.2002	-0.1504	-0.3256	-0.3666	0.631	0.707	0.473	0.430
NEPHB-01	ccc	measured bc	-0.1841	-0.1537	-0.3062	-0.3703	0.655	0.702	0.494	0.426
NEPHB-01	ccc	targeted	-0.2021	-0.1680	-0.3135	-0.3722	0.628	0.679	0.486	0.424
NEPHB-02	quenched	measured	-0.1936	-0.1261	-0.2827	-0.3637	0.640	0.748	0.522	0.433
NEPHB-02	quenched	measured bc	-0.1774	-0.1294	-0.2634	-0.3673	0.665	0.742	0.545	0.429
NEPHB-02	quenched	targeted	-0.1854	-0.1441	-0.2830	-0.3671	0.653	0.718	0.521	0.429
NEPHB-02	ccc	measured	-0.2001	-0.1495	-0.2709	-0.3652	0.631	0.709	0.536	0.431
NEPHB-02	ccc	measured bc	-0.1840	-0.1528	-0.2515	-0.3688	0.655	0.703	0.560	0.428
NEPHB-02	ccc	targeted	-0.1919	-0.1674	-0.2711	-0.3686	0.643	0.680	0.536	0.428
NEPHB-03	quenched	measured	-0.1519	-0.1186	-0.2160	-0.3592	0.705	0.761	0.608	0.437
NEPHB-03	quenched	measured bc	-0.1358	-0.1219	-0.1967	-0.3628	0.731	0.755	0.636	0.434
NEPHB-03	quenched	targeted	-0.1467	-0.1349	-0.2124	-0.3602	0.713	0.733	0.613	0.436
NEPHB-03	ccc	measured	-0.1637	-0.1265	-0.2240	-0.3615	0.686	0.747	0.597	0.435
NEPHB-03	ccc	measured bc	-0.1476	-0.1298	-0.2047	-0.3651	0.712	0.742	0.624	0.431
NEPHB-03	ccc	targeted	-0.1585	-0.1427	-0.2204	-0.3625	0.694	0.720	0.602	0.434
NEPHB-04	quenched	measured	-0.1376	-0.1188	-0.1415	-0.3555	0.728	0.761	0.722	0.441
NEPHB-04	quenched	measured bc	-0.1215	-0.1221	-0.1221	-0.3591	0.756	0.755	0.755	0.437
NEPHB-04	quenched	targeted	-0.1076	-0.1148	-0.1323	-0.3515	0.781	0.768	0.737	0.445
NEPHB-04	ccc	measured	0.0772	0.0597	-0.0466	-0.2984	1.195	1.147	0.898	0.503
NEPHB-04	ccc	measured bc	0.0934	0.0564	-0.0272	-0.3021	1.240	1.139	0.939	0.499
NEPHB-04	ccc	targeted	0.1072	0.0637	-0.0374	-0.2944	1.280	1.158	0.918	0.508
NEPHB-05	quenched	measured	-0.0548	-0.0731	-0.0724	-0.3233	0.882	0.845	0.846	0.475
NEPHB-05	quenched	measured bc	-0.0387	-0.0764	-0.0530	-0.3269	0.915	0.839	0.885	0.471
NEPHB-05	quenched	targeted	-0.0536	-0.0893	-0.0603	-0.3258	0.884	0.814	0.870	0.472
NEPHB-05	ccc	measured	-0.0949	-0.0636	-0.1203	-0.3373	0.804	0.864	0.758	0.460
NEPHB-05	ccc	measured bc	-0.0788	-0.0669	-0.1009	-0.3409	0.834	0.857	0.793	0.456
NEPHB-05	ccc	targeted	-0.0937	-0.0797	-0.1082	-0.3399	0.806	0.832	0.780	0.457

3.2.5 *Effects of Heat Treatment on PCTs*

Exhibit E7 in Appendix E provides a series of plots and statistical comparisons that show the effects of heat treatment on the common logarithm ppm-responses of interest on the triplicate PCTs for each element for each study glass. The ccc version of a given glass yielded measurements indicating a significantly (at the 5% significance level) larger mean log(ppm) response than the quenched version of the glass for a given element if the **Prob>t** value in the exhibit is 0.05 or smaller. This was the outcome for all 4 elements (B, Li, Na, and Si) for NEPHB-04. No other glass had any element for which the ccc version had a statistically greater mean than the quenched version.

Exhibit E8 in Appendix E provides a series of plots that show the effects of heat treatment on the PCT response based on the three different compositional views: measured, measured bias-corrected, and targeted. These plots allow for an assessment of the differences in PCT responses from a practical perspective and show, once again, that the PCT responses for the ccc version of NEPHB-04 were greater than their quenched counterparts. The normalized releases for boron, based on the measured compositions, are shown graphically in Figure 3-2 below. The PCT responses are indicated by the symbol (□) for the quenched glasses and the symbol (●) for the ccc glasses.

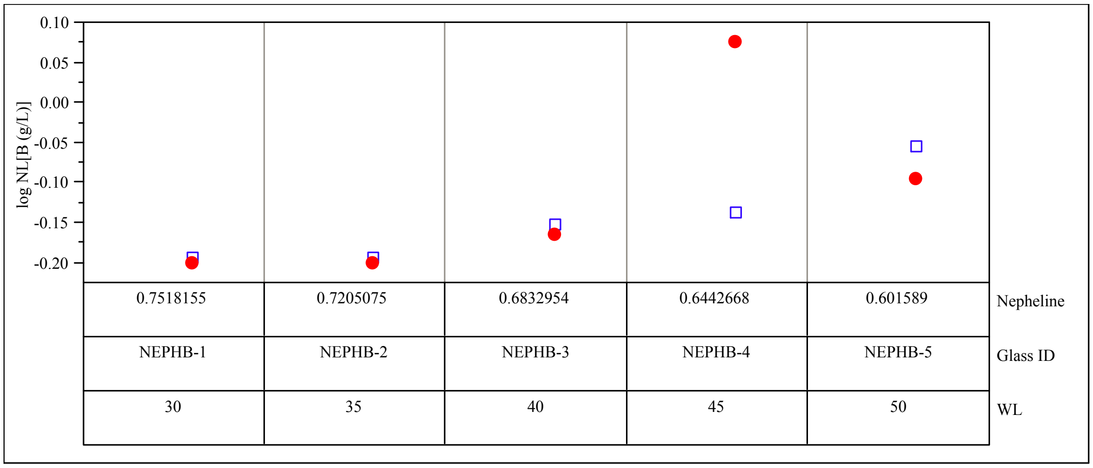


Figure 3-2. Normalized releases for boron, based on the measured compositions, for the Frit 503 glasses.

It was expected that the ccc version of NEPHB-05 would show the highest NL [B] due to its relatively high WL (50%) and its nepheline discriminator value of 0.60, which is below the critical value of 0.62. However, the NEPHB-05 glass, along with NEPHB-01, NEPHB-02 and NEPHB-03, showed little or no difference in NL [B] between the quenched and ccc versions of the glass. The highest NL [B] for the ccc version of NEPHB-04 (1.280 g/L based on targeted composition) is more than an order of magnitude below that of the EA glass (16.695 g/L),[21] so the difference in PCT response between the quenched and ccc versions of this glass, while curious from an experimental standpoint, presents no practical impact to processing at DWPF.

The results shown in Figure 3-2 may also indicate that the test solutions were reversed in the laboratory for NEPHB-04ccc and NEPHB-05ccc. A comparison of the target and measured compositions indicated that the glasses were not switched during the batching process. To test the potential that the PCT solutions may have been switched or mislabeled, the PCT was performed again for the ccc versions of the five Frit 503 glasses (i.e., archival specimens of each quenched glass from the original batching and melting process were re-heat treated, ground, and the PCT performed according to procedure). A second analytical plan was issued for this task, and is included as Appendix C. The results for the "re-tested" ccc glasses are included in Appendix E.

Table E3 in Appendix E provides the elemental leachate concentration measurements determined by the PSAL for the solution samples generated by the re-tested PCTs. None of these PCT results indicated a solution-weight loss problem. Consistent with earlier data, any measurement below the detection limit of the analytical procedure (indicated by a "<") was replaced by ½ of the detection limit in subsequent analyses. In addition to adjustments for detection limits, the values were adjusted for dilution. The values for the study glasses, the blanks, and the ARM glass in Table E3 were multiplied by a dilution factor of 1.6667 to determine the values in parts per million (ppm). The values for EA were multiplied by a dilution factor of 16.6667. Table E4 in Appendix E provides the resulting measurements.

Exhibit E9 in Appendix E indicates that the ccc version of NEPHB-04 was again shown to have higher releases than the other Frit 503 glasses. The re-tested PCTs indicate that no mishandling of samples occurred during the original PCTs. Therefore, the original PCT results were considered valid. Again, the NL [B] for the ccc version of NEPHB-04 is well below that of EA and does not present a concern for DWPF with respect to durability.

3.2.6 *Predicted versus Measured PCTs*

Exhibit E10 in Appendix E provides plots of the DWPF models that relate the logarithm of the normalized PCT (for each element of interest) to a linear function of a free energy of hydration term (ΔG_P, kcal/100g glass) derived from all of the glass compositional views and heat treatments.[30] Prediction limits (at a 95% confidence) for an individual PCT result are also plotted along with the linear fit. The EA and ARM-1 results are also indicated on these plots. Exhibit E11 in Appendix E provides a version of these plots for the quenched glasses only, while Exhibit E12 in Appendix E provides a version for ccc glasses only.

All of the quenched glasses and all but one of the ccc glasses show acceptable and predictable PCT responses. For NEPHB-04ccc, the B response shows an acceptable (NL [B] of 1.28 g/L) yet not predictable PCT value for the targeted and measured bias-corrected compositional views. Note that the ΔG_P value for this glass is more toward the positive-end of the range of values typically covered for ΔG_P. The unpredictability of this particular glass is not a concern, since glasses in the more positive ΔG_P range have been observed to fall outside the upper 95% confidence band while still having an acceptable durability.[38]

3.2.7 *Values of the Nepheline Constraint and Predictability*

Li *et al.* proposed 0.62 as the critical value for the nepheline discriminator.[15] Glass compositions with a nepheline discriminator value of less than 0.62 are prone to nepheline crystallization.[15] Table 3-3 provides the nepheline constraint values for each study glass for each compositional view. Note that only NEPHB-05 fails to satisfy the constraint. However, as will be shown below, nepheline crystallization did not occur (to an extent detectible by XRD) in this glass. Also, the PCT responses do not indicate significant nepheline crystallization for NEPHB-05. These results indicate that it would be useful to assess the measurement uncertainty associated with the nepheline discriminator. The uncertainty of the critical nepheline discriminator value will be important if the nepheline discriminator is to be included in DWPF process controls.

Table 3-3. Nepheline Constraint Values by Composition View

WL	Glass ID	measured	measured bc	targeted
30	NEPHB-01	0.752	0.756	0.759
35	NEPHB-02	0.721	0.725	0.723
40	NEPHB-03	0.683	0.688	0.685
45	NEPHB-04	0.644	0.649	0.646
50	NEPHB-05	0.602	0.606	0.606

3.3 Homogeneity

In this section, the primary interest is the possible formation of nepheline (and/or other crystalline phases) in the Frit 503 ccc glasses, which could be responsible for the measurable differences in PCT responses as compared to their quenched counterparts. Table 3-4 summarizes the visual and XRD results for the quenched and ccc Frit 503 glasses. It should be noted that only the ccc versions of the glasses were submitted for XRD analysis given that the visual observations and durability responses suggested no significant crystallization in the quenched glasses. That is, with normalized boron releases ranging from 0.64 g/L to 0.92 g/L, there is no evidence of nepheline formation in the quenched glasses – even if present, the impact is of no practical concern.

Prior to discussing the results, a few words regarding the terminology used in the tables are warranted. The use of "homogeneous" for visual observations indicates that the sample was classified as a single-phase system (i.e., no evidence of crystallization). The term "surface crystals" (used as a descriptor for visual observations) implies that the surface of the glass was characterized by the presence of crystallization while the cross-section of bulk glass appeared homogeneous (i.e., single-phase, black and shiny). Surface crystallization in the Frit 503 glasses was apparent through the presence of a "textured" surface that ranged in appearance from a "dull or matte" surface to a "highly metallic-like" surface.

The XRD results are more qualitative in nature. As previously mentioned, only the ccc glasses were submitted for XRD analysis based on both the PCT responses as well as visual observations of the quenched glasses. The PCT responses of the quenched glasses were "acceptable and predictable" and visual observations suggested only the presence of surface devitrification on the higher WL glasses. Historically, surface devitrification occurs as WLs increase, and this is typically the result of spinel formation for DWPF type glasses. The Frit 503 PCT responses suggested that for those quenched glasses that were classified as having "metallic swirls on the surface", spinel formation was highly probable, which is consistent with historical and recent observations and the inert effect on the PCT response. For the ccc glasses, the XRD results suggested that the glass was either amorphous or contained some degree of crystallization. The presence of a characteristically high background devoid of crystalline spectral lines indicates that the glass product is amorphous (suggesting either a completely amorphous product or that the degree of crystallization is below the detection limit – approximately 0.5 vol% in glass). In terms of crystallization, the XRD results indicated the presence of spinel (Trevorite, $NiFe_2O_4$). Nepheline ($NaAlSiO_4$) was not present at a detectible level in these glasses. For a more detailed description of the visual observations and XRD results of both the quenched and ccc glasses, see WSRC-NB-2006-00016 (pages 77 – 78).

3.3.1 *Visual Observations*

Visual observations of the quenched Frit 503 glasses indicate that four of the glasses were homogeneous, while NEPHB-05 was characterized by metallic swirls on the surface with the bulk (cross-section) being homogeneous. The noted surface crystallization on the quenched, high WL glass (NEPHB-05) is consistent with historical, visual observations of DWPF-based glasses, especially those targeting higher waste loadings. More specifically, use of descriptions such as a dull or matte texture and/or metallic-like surface is common for DWPF-type glasses targeting higher WLs and/or having undergone a slow cooling schedule. Previous XRD analyses have indicated that the textured or metallic-like surfaces are typically a result of spinels that precipitate during the cooling process. This is in-line with glass theory which suggests that as WL increases, the concentrations of sludge components such as Fe_2O_3, NiO, Cr_2O_3, and/or MnO also increase, enhancing the likelihood of spinel devitrification. Based on the PCT responses for the quenched glasses, spinel formation resulting in the metallic haze is reasonable as spinels have been shown to have no impact on the durability response.[4]

A metallic haze, either somewhat shiny or dull, characterized the surface of four of the ccc glasses, with NEPHB-01 (at the lowest WL) being the exception. The primary difference among the ccc glasses is the degree of devitrification visually observed within the bulk glass. That is, when examining the cross-sections of the heat treated samples, visual observations ranged from "clean, black and shiny" (indicating a homogeneous glass) to "crystals throughout". The transition from homogeneous to partially devitrified and completely devitrified resulted as WL increased. In general, visual observations indicate that devitrification was more prevalent in the ccc glasses than in the quenched glasses, as expected, given kinetics are more favorable for devitrification during the slower cooling cycle.

Table 3-4. Visual observations and XRD results for the Frit 503 glasses.

Glass	Target WL	Heat treatment	Visual Observations	XRD Results
NEPHB-01	30	quenched	Patty - clean, black and shiny, homogeneous; Crucible – clean with bubbles	-
NEPHB-01	30	ccc	Surface – clean, black and shiny; Bulk – clean, black and shiny	amorphous
NEPHB-02	35	quenched	Patty – clean, black and shiny, homogeneous; Crucible – clean with bubbles	-
NEPHB-02	35	ccc	Surface – shiny, metallic haze; Bulk – clean	amorphous
NEPHB-03	40	quenched	Patty – clean, black and shiny, homogeneous; Crucible – clean with bubbles	-
NEPHB-03	40	ccc	Surface – shiny metallic haze with spots of crystals; Bulk - clean	amorphous
NEPHB-04	45	quenched	Patty – one spot of undissolved material in bulk, otherwise clean; Crucible – one spot of undissolved material, otherwise clean	-
NEPHB-04	45	ccc	Surface – duller metallic haze with spots of crystals; Bulk – shiny, some crystals	$NiFe_2O_4$
NEPHB-05	50	quenched	Patty – small amount of metallic swirls on surface, bulk clean; Crucible – one spot of undissolved material, otherwise clean	-
NEPHB-05	50	ccc	Surface – dull, matte, crystals across most of surface; Bulk – crystals throughout	$NiFe_2O_4$

3.3.2 *XRD Results*

XRD results indicated that the low WL glasses (i.e., 30 to 40 wt%) were amorphous. This agrees well with the PCT data, in that no statistical or practical difference in NL [B] response was seen between the quenched and ccc versions of NEPHB-01, NEPHB-02 and NEPHB-03. The higher WL ccc glasses (45% and 50% WL) were shown by XRD to contain spinel (trevorite, $NiFe_2O_4$). Previous work has shown that spinels do not have a negative impact on durability,[11] so this is likely not the cause of the higher NL [B] measured for the ccc version of NEPHB-04. While the data collected in this study are not sufficient to elucidate the cause of this higher PCT response, it is again of little practical concern as the NL [B] for NEPHB-04 (1.280 g/L based on targeted composition) is more than an order of magnitude below that of the EA glass (16.695 g/L).[21]

4.0 Conclusions

The results of this study concur with the earlier nepheline crystallization studies (Phases 1 through 3) in that a nepheline discriminator of 0.62 appears to be the appropriate value for screening out glasses with the potential for nepheline crystallization upon slow cooling. Further discussion of the nepheline discriminator and a recommendation for its inclusion in DWPF process controls will be addressed in a forthcoming report.

Chemical composition measurements indicated that the experimental glasses were close to their target compositions. PCT results showed that all of the Fit 503 quenched glasses were acceptable as compared with the EA reference glass. The durability of one of the ccc glasses, NEPHB-04, was statistically greater than its quenched counterpart. However, this was not driven by nepheline crystallization and was shown to be of little practical significance, as the durability of the NEPHB-04 ccc glass was also well below that of the EA reference glass. The PCT response of this glass was also unpredictable by the model but historically, glasses in the more positive ΔG_P range falling outside the upper 95% confidence band have been observed.[38]

Visual observations and PCT results indicated that all of the Frit 503 quenched glasses were amorphous. For the ccc glasses, XRD results indicated that the lower WL glasses (30 to 40 wt%) were amorphous, which was consistent with visual observations and the similarity in PCT responses. The higher WL glasses (40 and 50 wt%) were shown by XRD to contain spinel (trevorite, $NiFe_2O_4$). It is possible that some of the other high WL glasses also contained some nepheline, but that the amount of nepheline crystallization was below the detection limit associated with XRD.

With respect to frit selection for SB4, the results indicate that Frit 503 is a good candidate for SB4 processing, based on PCT responses for both quenched and ccc glasses over a WL range of 30 – 50%. It should be noted that the higher WL glasses would not be processable in DWPF as they exceed other process related criteria (such as T_L). However, melt rate and the final SB4 composition projection are also important factors in frit selection. Additional melt rate studies are currently underway, and the final composition projection for SB4 is expected shortly.

This page intentionally left blank.

5.0 Recommendations

The path forward for evaluating the impact of nepheline formation on SB4-based glasses should include an assessment of the impact of implementing a nepheline discriminator value of 0.62 as part of PCCS at DWPF, based on the results of the Phase 1 – 3 studies and the Frit 503 study. A determination should be made as to whether the nepheline discriminator would be effective in screening out glasses that are either unacceptable (based on PCT responses) and/or unpredictable (using the ΔG_P models).

In addition, the impact of measurement uncertainty (MAR) on the projected operating windows for the frit-SB4 systems of interest must be made. The nepheline discriminator value of 0.62 does not yet have a measurement uncertainty associated with it. An assessment must be made to determine whether the inclusion of measurement uncertainty in the nepheline discriminator will restrict the range of WLs available to DWPF.

The impact of applying a nepheline discriminator to process controls must be evaluated for glasses that have already been fabricated at DWPF. Future work should identify what impact, if any, implementation of the nepheline discriminator would have on acceptability of historical glass compositions.

Finally, Li *et al*[15] suggest that B_2O_3 suppresses nepheline formation based on a structural role or competition with Al_2O_3 for Na_2O in the borosilicate glass network. More specifically, B_2O_3 tends to lower the activity of Na_2O in the melt which restricts or reduces the amount of Na_2O available to form nepheline. That being said, it is interesting to note that B_2O_3 is not associated with the nepheline discriminator proposed by Li *et al*.[15] Although the experimental results from the nepheline studies associated with SB4 have shown that the 0.62 nepheline value is effective, perhaps the effect of B_2O_3 may improve this predictive tool.

This page intentionally left blank.

6.0 References

1. Brown, K. G., R. L. Postles and T. B. Edwards, **SME Acceptability Determination for DWPF Process Control**, WSRC-TR-95-00364, Revision 4, Westinghouse Savannah River Company, Aiken, South Carolina (2002).

2. Jantzen, C. M. and K. G. Brown, "Predicting the Spinel-Nepheline Liquidus for Application to Nuclear Waste Glass Processing: Part I. Primary Phase Analysis, Liquidus Measurement, and Quasicrystaline Approach," *J. Am. Ceram. Soc.,* **in press** (2006).

3. Jantzen, C. M. and K. G. Brown, "Predicting the Spinel-Nepheline Liquidus for Application to Nuclear Waste Glass Processing: Part II. Quasicrystalline Freezing Point Depression Model," *J. Am. Ceram. Soc.,* **in press** (2006).

4. Bickford, D. F. and C. M. Jantzen, "Devitrification of SRL Defense Waste Glass," *Sci. Basis for Nuclear Waste Management VII, edited by G. L. McVay.* Elsevier, New York, pp. 557-565 (1984).

5. Bickford, D. F. and C. M. Jantzen, "Devitrification of Defense Nuclear Waste Glasses: Role of Melt Insolubles," *J. Non-Crystalline Solids,* **84** [1-3] 299-307 (1986).

6. Jantzen, C. M., D. F. Bickford, D. G. Karraker and G. G. Wicks, "Time-Temperature-Transformation Kinetics in SRL Waste Glass," pp. 30-38 in *Advances in Ceramics, Vol. 8,* American Ceramic Society, Westerville, OH (1984).

7. Spilman, D. B., L. L. Hench and D. E. Clark, "Devitrification and Subsequent Effects on the Leach Behavior of a Simulated Borosilicate Nuclear Waste Glass," *Nuclear and Chemical Waste Management,* **6** 107-119 (1986).

8. Marra, S. L. and C. M. Jantzen, **Characterization of Projected DWPF Glass Heat Treated to Simulate Canister Centerline Cooling**, WSRC-TR-92-142, Revision 1, Westinghouse Savannah River Company, Aiken, South Carolina (1993).

9. Li, H., J. D. Vienna, P. Hrma, D. E. Smith and M. J. Schwieger, "Nepheline Precipitation in High-Level Waste Glasses - Compositional Effects and Impact on the Waste Form Acceptability," *Mat. Res. Soc. Proc., Vol. 465,* pp. 261-268 (1997).

10. Riley, B. J., J. A. Rosario and P. Hrma, **Impact of HLW Glass Crystallinity on the PCT Response**, PNNL-13491, Pacific Northwest National Laboratory, Richland, Washington (2001).

11. Jantzen, C. M. and D. F. Bickford, "Leaching of Devitrified Glass Containing Simulated SRP Nuclear Waste," pp. 135-146 in *Sci. Basis for Nuclear Waste Management, Vol. 8,* J. A. Stone and R. C. Ewing, eds. Materials Research Society, Pittsburgh, PA (1985).

12. Cicero, C. A., S. L. Marra and M. K. Andrews, **Phase Stability Determinations of DWPF Waste Glasses (U)**, WSRC-TR-93-00227, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (1993).

13. Kim, D. S., D. K. Peeler and P. Hrma, "Effect of Crystallization on the Chemical Durability of Simulated Nuclear Waste Glasses," *Environmental Issues and Waste Management Technologies in the Ceramic and Nuclear Industries, Vol. 61,* pp. 177-185 (1995).

14. Lilliston, G. R., **Development of Elemental Sludge Compositions for Variations of Sludge Batch 4 (SB4)**, CBU-PIT-2004-00011, Revision 1, Westinghouse Savannah River Company, Aiken, South Carolina (2005).

15. Li, H., P. Hrma, J. D. Vienna, M. Qian, Y. Su and D. E. Smith, "Effects of Al_2O_3, B_2O_3, Na_2O, and SiO_2 on Nepheline Formation in Borosilicate Glasses: Chemical and Physical Correlations," *J. Non-Crystalline Solids,* **331** 202-216 (2003).

16. Peeler, D. K. and T. B. Edwards, **Frit Development Efforts for Sludge Batch 4: Model-Based Assessments**, WSRC-TR-2005-00103, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2005).

17. Peeler, D. K. and T. B. Edwards, **Model Based Assessments for the Baseline Sludge Batch 4 (Case 15C) Flowsheet**, WSRC-TR-2006-00049, Revision 0, Washington Savannah River Company, Aiken, South Carolina (2006).

18. Peeler, D. K., T. B. Edwards and T. H. Lorier, **Nepheline Formation Potential in Sludge Batch (SB4) Glasses**, WSRC-TR-2005-00153, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2005).

19. Peeler, D. K., T. B. Edwards, I. A. Reamer and R. J. Workman, **Nepheline Formation Study for Sludge Batch 4 (SB4): Phase 1 Experimental Results**, WSRC-TR-2005-00371, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2005).

20. ASTM, **Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT)**, ASTM C-1285-2002, (2002).

21. Jantzen, C. M., N. E. Bibler, D. C. Beam, C. L. Crawford and M. A. Pickett, **Characterization of the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) Glass Standard Reference Material**, WSRC-TR-92-346, Revision 1, Westinghouse Savannah River Company, Aiken, South Carolina (1993).

22. Elder, H. H., **Estimate of Sludge Batch 4 Calcine Composition**, CBU-PIT-2005-00134, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2005).

23. Elder, H. H., **Estimate of Sludge Batch 4 Calcine Composition Additional Cases**, CBU-PIT-2005-00176, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2005).

24. Peeler, D. K. and T. B. Edwards, **Frit Development Effort for SB4: Nominal and Variation Stage Assessments**, WSRC-TR-2005-00372, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2005).

25. Peeler, D. K., T. B. Edwards, D. R. Best, I. A. Reamer and R. J. Workman, **Nepheline Formation Study for Sludge Batch 4 (SB4): Phase 2 Experimental Results**, WSRC-TR-2006-00006, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2006).

26. Edwards, T. B. and D. K. Peeler, **Nepheline Formation Potential in Sludge Batch 4 (SB4) and Its Impact on Durability: Selecting Glasses for a Phase 2 Study**, WSRC-TR-2005-00370, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2005).

27. Fox, K. M., T. B. Edwards and D. K. Peeler, **Nepheline Formation Potential in Sludge Batch 4 (SB4) and Its Impact on Durability: Selecting Glases for a Phase 3 Study**, WSRC-TR-2006-00053, Revision 0, Washington Savannah River Company, Aiken, South Carolina (2006).

28. Shah, H. B., **Estimate of Sludge Batch 4 Calcine Composition: Additional Cases for Final Recommendation**, CBU-PIT-2006-0001, Westinghouse Savannah River Company, Aiken, South Carolina (2006).

29. Fox, K. M., D. K. Peeler, T. B. Edwards, D. R. Best, I. A. Reamer and R. J. Workman, **Nepheline Formation Study for Sludge Batch 4 (SB4): Phase 3 Experimental Results**, WSRC-TR-2006-00093, Revision 0, Washington Savannah River Company, Aiken, South Carolina (2006).

30. Jantzen, C. M., J. B. Picket, K. G. Brown, T. B. Edwards and D. C. Beam, **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)**, WSRC-TR-93-672, Revision 1, Westinghouse Savannah River Company, Aiken, South Carolina (1995).

31. Peeler, D. K. and T. B. Edwards, **High B_2O_3/Fe_2O_3-based Frits: MAR Assessments for Sludge Batch 4 (SB4)**, WSRC-TR-2006-00181, Revision 0, Washington Savannah River Company, Aiken, South Carolina (2006).

32. Washburn, F. A., **Technical Task Request: Sludge Batch 4 and MCU Frit Optimization**, HLW/DWPF/TTR-2004-0025, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2004).

33. Peeler, D. K., **Task Technical & QA Plan: Sludge Batch and MCU Frit Optimization**, WSRC-RP-2004-00746, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2004).

34. Peeler, D. K. and T. B. Edwards, **Model Based Assessments for SB4 Washing Options: 1.2M Batch/0.91M Blend and 1.4M Batch/0.96M Blend**, WSRC-STI-2006-00006, Revision 0, Washington Savannah River Company, Aiken, South Carolina (2006).

35. SRNL, **Glass Batching**, SRTC Procedure Manual, L29, ITS-0001, Westinghouse Savannah River Company, Aiken, South Carolina (2002).

36. SRNL, **Glass Melting**, SRTC Procedure Manual, L29, ITS-0003, Westinghouse Savannah River Company, Aiken, South Carolina (2002).

37. Peeler, D. K., C. C. Herman, M. E. Smith, T. H. Lorier, D. R. Best, T. B. Edwards and M. A. Baich, **An Assessment of the Sulfate Solubility Limit for the Frit 418 – Sludge Batch 2/3 System**, WSRC-TR-2004-00081, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2004).

38. Harbour, J. R., T. B. Edwards and R. J. Workman, **Summary of Results for Macrobatch 3 Variability Study**, WSRC-TR-2000-00351, Revision 0, Westinghouse Savannah River Company, Aiken, South Carolina (2000).

This page intentionally left blank.

Appendix A

An Analytical Plan for Measuring the Chemical Compositions of Five Frit 503/SB4 Glasses (U)

(SRNL-SCS-2006-00014)

This page intentionally left blank.

SRNL-SCS-2006-00014

April 7, 2006

To: K. M. Fox, SRNL

cc:
R. A. Baker, 773-42A
D. R. Best, 786-1A (wo)
C. C. Herman, 999-W
D. K. Peeler, 999-W
I. A. Reamer, 999-1W
P. A. Toole, 786-1A (wo)
R. C. Tuckfield, 773-42A
R. J. Workman, 999-1W

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

wo – without glass identifiers

R. A. Baker, Technical Reviewer — 4/17/2006 Date

R. C. Tuckfield, Manager
Statistical Consulting Section — 4/17/2006 Date

AN ANALYTICAL PLAN FOR MEASURING THE CHEMICAL COMPOSITIONS OF FIVE FRIT 503/SB4 GLASSES (U)

1.0 EXECUTIVE SUMMARY

Frit development efforts are underway at the Savannah River National Laboratory (SRNL) to support the processing of Sludge Batch 4 (SB4) at the Defense Waste Processing Facility (DWPF). One of the candidate frits considered during the recent assessments of the baseline preparation plan (Case 15C) was Frit 503, which was called Frit 418-m10 in the assessment. To provide experimental results to support the evaluation of this frit with SB4, five glasses have been fabricated for durability testing. Testing the durability of these glasses will also complement SRNL's ongoing nepheline studies by providing additional insight into the ability of the nepheline discriminator to predict the occurrence of a nepheline primary crystalline phase for SB4 glasses and into the impact of nepheline on the durability of the SB4 glasses.

The chemical compositions of the five Frit 503/SB4 glasses are to be determined by SRNL's Process Science Analytical Laboratory (PSAL). This memorandum provides an analytical plan to direct and support these measurements at PSAL.

2.0 INTRODUCTION

Frit development efforts are underway at the Savannah River National Laboratory (SRNL) to support the processing of Sludge Batch 4 (SB4) at the Defense Waste Processing Facility (DWPF). One of the candidate frits considered during the recent assessments [1] of the baseline preparation plan (Case 15C) was Frit 503, which was called Frit 418-m10 in [1]. To provide experimental results to support the evaluation of this frit with SB4, five glasses have been fabricated for durability testing. Testing the durability of these glasses will also complement SRNL's ongoing nepheline studies by providing additional insight into the ability of the nepheline discriminator [2] to predict the occurrence of a nepheline primary crystalline phase for SB4 glasses and into the impact of nepheline on the durability of the SB4 glasses. In addition, the results from the study of these glasses will contribute needed data to the ComPro™ database [3] in anticipation of a variability study for SB4.

The chemical compositions of the five Frit 503/SB4 glasses are to be determined by SRNL's Process Science Analytical Laboratory (PSAL). This memorandum provides an analytical plan to direct and support these measurements at PSAL.

3.0 ANALYTICAL PLAN

The analytical procedures used by PSAL to determine cation concentrations for a glass sample include steps for sample preparation and for instrument calibration. Each glass is to be prepared in duplicate by each of two dissolution methods: lithium metaborate fusion (LM) and sodium peroxide fusion (PF).

The primary measurements of interest are to be acquired as follows. The samples prepared by LM are to be measured for barium (Ba), calcium (Ca), cerium (Ce), chromium (Cr), copper (Cu), potassium (K), lanthanum (La), magnesium (Mg), manganese (Mn), sodium (Na), lead (Pb), sulfur (S), thorium (Th), titanium (Ti), zinc (Zn), and zirconium (Zr) concentrations. Samples prepared by PF are to be measured for aluminum (Al), boron (B), iron (Fe), lithium (Li), nickel (Ni), silicon (Si), and uranium (U). Samples dissolved by both preparation methods are to be measured using Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES). It should be noted that some of these elements are minor components that may be near detection limits for most, if not all, of the study glasses.

Randomizing the preparation steps and blocking and randomizing the measurements for the ICP-AES are of primary concern in the development of this analytical plan. The sources of uncertainty for the analytical procedure used by PSAL to determine the cation concentrations for the submitted glass samples are dominated by the dissolution step in the preparation of the sample and by the calibrations of the ICP-AES. Samples of glass standards will be included in the analytical plan to provide an opportunity for checking the performance of the instrumentation over the course of the analyses and for potential bias-correction. Specifically, several samples of Waste Compliance Plan (WCP) Batch 1 (BCH) [4] and a uranium standard glass (Ustd) are included in this analytical plan. The reference compositions of these glasses are provided in Table 1.

Table 1: Oxide Compositions of WCP Batch 1 (BCH) and of Ustd (wt%)

Oxide/ Anion	BCH (wt %)	Ustd (wt %)
Al_2O_3	4.877	4.1
B_2O_3	7.777	9.209
BaO	0.151	0
CaO	1.22	1.301
Cr_2O_3	0.107	0
Cs_2O	0.06	0
CuO	0.399	0
Fe_2O_3	12.839	13.196
K_2O	3.327	2.999
Li_2O	4.429	3.057
MgO	1.419	1.21
MnO	1.726	2.892
Na_2O	9.003	11.795
Nd_2O_3	0.147	0
NiO	0.751	1.12
RuO_2	0.0214	0
SiO_2	50.22	45.353
SO_3	0	0
TiO_2	0.677	1.049
U_3O_8	0	2.406
ZrO_2	0.098	0

Each glass sample submitted to PSAL will be prepared in duplicate by the LM and PF dissolution methods. Every prepared sample will be read twice by ICP-AES, with the instrument being calibrated before each of these two sets of readings. This will lead to four measurements for each cation of interest for each submitted glass.

Table 2 presents identifying codes, I01 through I05, for the 5 glasses fabricated for this study. The table provides a naming convention that is to be used in analyzing the glasses and reporting the measurements of their compositions.[4]

---

[4] Renaming these samples helps to ensure that they will be processed as blind samples within PSAL. Table 2 is not shown in its entirety in the copies going to PSAL.

Table 2: Glass Identifiers to Establish Blind Samples for PSAL

Glass ID	Sample ID
NEPHB-01	I03
NEPHB-02	I01
NEPHB-03	I05
NEPHB-04	I02
NEPHB-05	I04

3.1 *PREPARATION OF THE SAMPLES*

Each of the 5 glasses included in this analytical plan is to be prepared in duplicate by the LM and PF dissolution methods. Thus, the total number of prepared glass samples is determined by $5 \cdot 2 \cdot 2 = 20$, not including the samples of the BCH and Ustd glass standards that are to be prepared.

Table 3 provides blocking and (random) sequencing schema for conducting the preparation steps of the analytical procedures. One block of preparation work is provided for each preparation method to facilitate the scheduling of activities by work shift. The identifier for each of the prepared samples indicates the sample identifier (ID), preparation method, and duplicate number.

Table 3: Preparation Blocks by Dissolution Method

LM (Lithium Metaborate)	PF (Peroxide Fusion)
I03LM1	I04PF1
I01LM1	I02PF1
I03LM2	I05PF1
I05LM1	I01PF1
I04LM1	I04PF2
I02LM1	I03PF1
I05LM2	I01PF2
I04LM2	I05PF2
I02LM2	I02PF2
I01LM2	I03PF2

3.2 *ICP-AES Calibration Blocks*

The glass samples prepared by the LM and PF dissolution methods are to be analyzed using ICP-AES instrumentation calibrated for the particular preparation method. After the initial set of cation concentration measurements, the ICP-AES instrumentation is to be recalibrated and a second set of concentration measurements for the cations determined.

Randomized plans for measuring cation concentrations in the LM-prepared and PF-prepared samples are provided in Table 4. The cations to be measured are specified as part of the table. In the tables, the sample identifiers for the 5 study glasses have been modified by the addition of a suffix (a “1”or a “2”) to indicate whether the measurement was made during the first or second (respectively) calibration of the ICP-AES instrumentation. The identifiers for the BCH and Ustd samples have been modified to indicate the ICP-AES calibration and that each of these prepared samples is to be read 2 times (mirrored in the corresponding suffix of 1. 2, or 3) per calibration block.

Table 4: ICP-AES Blocks & Calibration Groups by Preparation Method

LM Glass Samples		**PF Glass Samples**	
Used to Measure Elemental Ba, Ca, Ce, Cr, Cu, K, La, Mg, Mn, Na, Pb, S, Th, Ti, Zn, & Zr		Used to Measure Elemental Al, B, Fe, Li, Ni, Si, & U	
LM Block		**PF Block**	
Calibration 1	**Calibration 2**	**Calibration 1**	**Calibration 2**
BCHLM11	BCHLM21	BCHPF11	BCHPF21
UstdLM11	UstdLM21	UstdPF11	UstdPF21
I02LM11	I01LM22	I04PF21	I05PF22
I04LM21	I02LM22	I03PF11	I01PF22
I02LM21	I03LM12	I05PF11	I02PF12
I01LM11	I05LM12	I03PF21	I05PF12
I05LM21	I03LM22	I01PF21	I04PF22
BCHLM12	BCHLM22	BCHPF12	BCHPF22
UstdLM12	UstdLM22	UstdPF12	UstdPF22
I04LM11	I02LM12	I02PF21	I03PF22
I05LM11	I01LM12	I02PF11	I04PF12
I03LM21	I04LM22	I01PF11	I02PF22
I03LM11	I04LM12	I04PF11	I03PF12
I01LM21	I05LM22	I05PF21	I01PF12
BCHLM13	BCHLM23	BCHPF13	BCHPF23
UstdLM13	UstdLM23	UstdPF13	UstdPF23

4.0 CONCLUDING COMMENTS

In summary, this analytical plan identifies two preparation blocks in Table 3 and four ICP-AES calibration blocks in Table 4 for use by PSAL. The sequencing of the activities associated with each of the steps in the analytical procedures has been randomized. The size of each of the blocks was selected so that it could be completed in a single work shift.

If a problem is discovered while measuring samples in a calibration block, the instrument should be re-calibrated and the block of samples re-measured in its entirety. If for some reason the measurements are not conducted in the sequences presented in this report, a record should be made of the actual order used along with any explanative comments.

The analytical plan indicated in the preceding tables should be modified by the personnel of PSAL to include any calibration check standards and/or other standards that are part of their routine operating procedures. It is also recommended that the solutions resulting from each of the prepared samples be archived for some period, considering the “shelf-life” of the solutions, in case questions arise during data analysis. This would allow for the solutions to be rerun without additional preparations, thus minimizing cost.

5.0 REFERENCES

[1] Peeler, D.K. and T.B. Edwards, “Model Based Assessments for the Baseline Sludge Batch 4 (Case 15C) Preparation Plan,” WSRC-TR-2006-00049, Revision 0, 2006.

[2] Li, H., P. Hrma, J.D. Vienna, M. Qian, Y. Su, and D.E. Smith, “Effects of Al_2O_3, B_2O_3, Na_2O, and SiO_2 on Nepheline Formation in Borosilicate Glasses: Chemical and Physical Correlations,” **Journal of Non-Crystalline Solids**, 331, pgs. 202-216, 2003.

[3] Taylor, A.S., T.B. Edwards, J.C. George, T.K. Snyder, and D.K. Peeler, “The SRNL Glass Composition – Properties (ComPro) Database,” WSRC-RP-2004-00704, Revision 0, 2004.

[4] Jantzen, C.M., J.B. Pickett, K.G. Brown, T.B. Edwards, and D.C. Beam, “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^{TM}) (U),” WSRC-TR-93-673, Revision 1, Volume 2, Table B.1, pp. B.9, 1995.

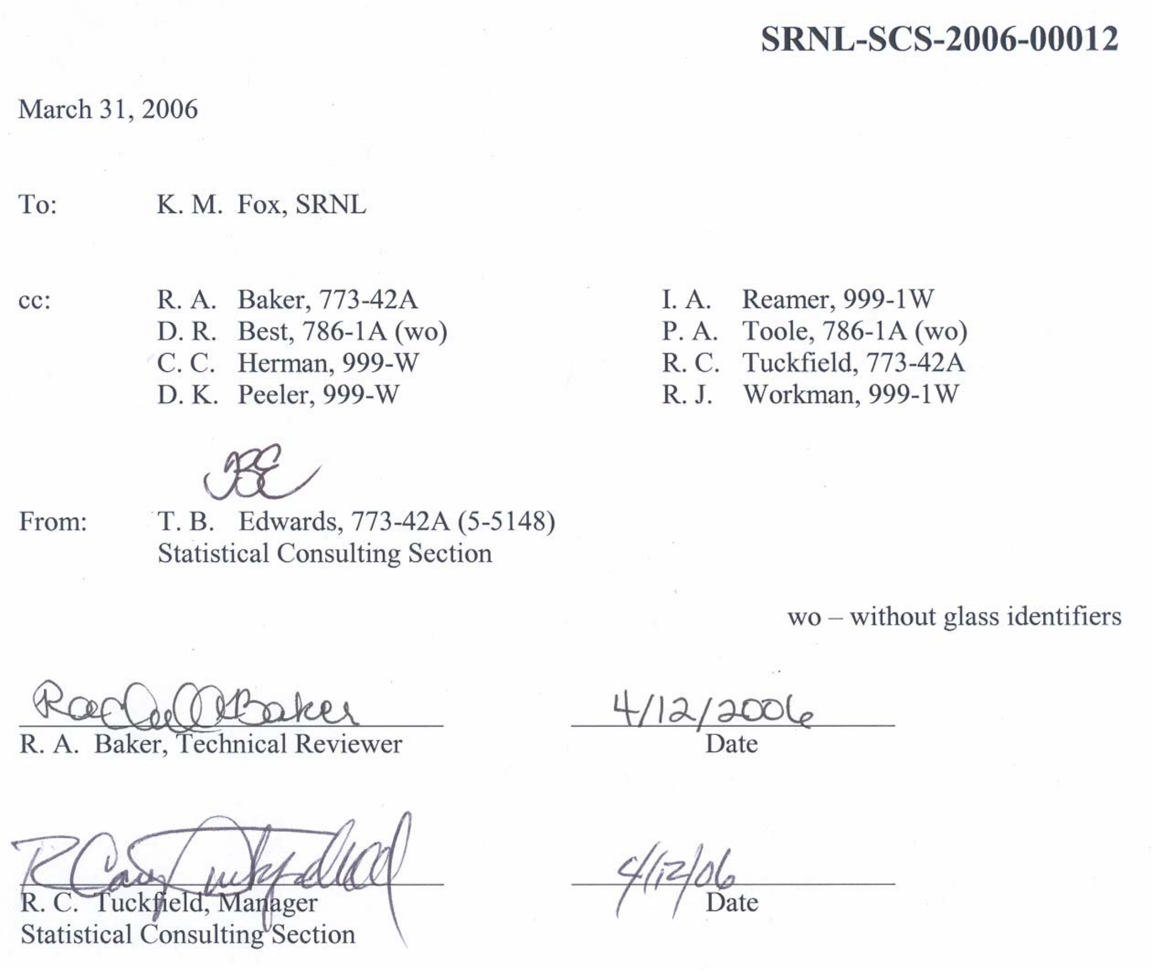
This page intentionally left blank.

Appendix B

An Analytical Plan for Measuring PCT Solutions for a Set of Five Frit 503/SB4 Glasses (U)

(SRNL-SCS-2006-00012)

This page intentionally left blank.



SRNL-SCS-2006-00012

March 31, 2006

To: K. M. Fox, SRNL

cc:
R. A. Baker, 773-42A
D. R. Best, 786-1A (wo)
C. C. Herman, 999-W
D. K. Peeler, 999-W
I. A. Reamer, 999-1W
P. A. Toole, 786-1A (wo)
R. C. Tuckfield, 773-42A
R. J. Workman, 999-1W

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

wo – without glass identifiers

R. A. Baker, Technical Reviewer

4/12/2006
Date

R. C. Tuckfield, Manager
Statistical Consulting Section

4/12/06
Date

AN ANALYTICAL PLAN FOR MEASURING PCT SOLUTIONS FOR A SET OF FIVE FRIT 503/SB4 GLASSES (U)

1.0 EXECUTIVE SUMMARY

Frit development efforts are underway at the Savannah River National Laboratory (SRNL) to support the processing of Sludge Batch 4 (SB4) at the Defense Waste Processing Facility (DWPF). One of the candidate frits considered during the recent assessments of the baseline preparation plan (Case 15C) was Frit 503, which was called Frit 418-m10 in the assessment. To provide experimental results to support the evaluation of this frit with SB4, five glasses have been fabricated for durability testing. Testing the durability of these glasses will also complement SRNL's ongoing nepheline studies by providing additional insight into the ability of the nepheline discriminator to predict the occurrence of a nepheline primary crystalline phase for SB4 glasses and into the impact of nepheline on the durability of the SB4 glasses.

The durability of the glasses is to be measured using the Product Consistency Test (PCT) as defined in ASTM C-1285-2002. Two heat treatments were utilized during the fabrication of each of these glasses. Specifically, each of the 5 glasses was quenched (i.e., rapidly cooled) and cooled in accordance with the centerline-canister-cooling (ccc) regime. Both heat treatments of each glass are to be subjected to the PCT.

The PCTs are to be submitted to SRNL's Process Science Analytical Laboratory (PSAL) for measurement. This memorandum provides an analytical plan for the measurement of the PCTs by PSAL.

2.0 INTRODUCTION

Frit development efforts are underway at the Savannah River National Laboratory (SRNL) to support the processing of Sludge Batch 4 (SB4) at the Defense Waste Processing Facility (DWPF). One of the candidate frits considered during the recent assessments [1] of the baseline preparation plan (Case 15C) was Frit 503, which was called Frit 418-m10 in [1]. The specific SB4 option being considered is Case 15C Blend 1 as defined by Shah [2]. To provide experimental results to support the evaluation of this frit with SB4, five glasses have been fabricated for durability testing. Testing the durability of these glasses will also complement SRNL's ongoing nepheline studies by providing additional insight into the ability of the nepheline discriminator [3] to predict the occurrence of a nepheline primary crystalline phase for SB4 glasses and into the impact of nepheline on the durability of the SB4 glasses. In addition, the results from the study of these glasses will contribute needed data to the ComPro™ database [4] in anticipation of a variability study for SB4.

The durability of the glasses is to be measured using the Product Consistency Test (PCT) as defined in ASTM C-1285-2002 [5]. Two heat treatments were utilized during the fabrication of each of these glasses. Specifically, each of the 5 glasses was quenched (i.e., rapidly cooled) and cooled in accordance with the centerline-canister-cooling (ccc) regime. Both heat treatments of each glass are to be subjected to the PCT.

The PCTs are to be submitted to SRNL's Process Science Analytical Laboratory (PSAL) for measurement. This memorandum provides an analytical plan for the measurement of the PCTs by PSAL. Table 1 presents a listing of the glasses covered by this memorandum.

Table 1: Identifiers for Glasses Covered by this Plan

NEPHB-01
NEPHB-01ccc
NEPHB-02
NEPHB-02ccc
NEPHB-03
NEPHB-03ccc
NEPHB-04
NEPHB-04ccc
NEPHB-05
NEPHB-05ccc

3.0 DISCUSSION

Each of the study glasses of Table 1 is to be subjected to the PCT in triplicate. In addition to PCTs for the study glasses, triplicate PCTs are to be conducted on a sample of the Approved Reference Material – One (ARM-1) glass and a sample of the Environmental Assessment (EA) glass. Two reagent blank samples are also to be included in these tests. This results in 38 sample solutions being required to complete these PCTs.

The leachates from these tests will be diluted by adding 4 mL of 0.4 M HNO_3 to 6 mL of the leachate (a 6:10 volume to volume, v:v, dilution) before being submitted to PSAL. The leachates of EA will be further diluted (1:10 v:v) with deionized water prior to submission to PSAL in order to prevent problems with the nebulizer. Note that additional dilutions for the ccc versions of one or more of the study glasses may be needed due to a possible low durability of some of the glasses. Upon termination of the PCT, a decision is to be made (by the technicians and a PSAL representative, if called by the technician) as to whether any other dilution is needed for these solutions to mitigate any potential gelling issues. Any extra dilutions are to be reported, and guidance is to be given as to how the dilutions are to be handled in the statistical assessment of the measurement data. More specifically, PSAL will be responsible for indicating if any additional dilutions were made and how they were, or how they should be, accounted for in the reported measurements.

Table 2 presents identifying codes, J01 through J38, for the individual solutions required for the PCTs of the select study glasses and of the standards (EA, ARM-1, and blanks). This provides a naming convention that is to be used by PSAL in analyzing the solutions and reporting the relevant concentration measurements.[a]

Table 2: Identifiers for the PCT Solutions Covered by this Plan

Original Sample	Solution Identifier	Original Sample	Solution Identifier
NEPHB-01	J24	NEPHB-04ccc	J37
NEPHB-01	J05	NEPHB-04ccc	J08
NEPHB-01	J18	NEPHB-04ccc	J09
NEPHB-01ccc	J26	NEPHB-05	J25
NEPHB-01ccc	J36	NEPHB-05	J10
NEPHB-01ccc	J17	NEPHB-05	J20
NEPHB-02	J30	NEPHB-05ccc	J12
NEPHB-02	J32	NEPHB-05ccc	J06
NEPHB-02	J07	NEPHB-05ccc	J27
NEPHB-02ccc	J04	EA	J16
NEPHB-02ccc	J13	EA	J31
NEPHB-02ccc	J22	EA	J23
NEPHB-03	J28	ARM-1	J15
NEPHB-03	J35	ARM-1	J14
NEPHB-03	J01	ARM-1	J19
NEPHB-03ccc	J29	blank	J02
NEPHB-03ccc	J11	blank	J21
NEPHB-03ccc	J03		
NEPHB-04	J34		
NEPHB-04	J38		
NEPHB-04	J33		

---

[a] Renaming these samples ensures that they will be processed as blind samples by PSAL. This table does not contain the solution identifiers for those on the distribution list with a “wo” following their names.

4.0 ANALYTICAL PLAN

The analytical plan for PSAL is provided in this section. Each of the solution samples submitted to PSAL is to be analyzed only once for each of the following: boron (B), barium (Ba), cadmium (Cd), chromium (Cr), iron (Fe), lithium (Li), sodium (Na), lead (Pb), silicon (Si), thorium (Th), and uranium (U). B, Li, Na, and Si are the elements that are to be used in the assessment of glass durability; the other elements are being monitored to address solution disposal issues in SRNL upon termination of the PCTs. The measurements are to be made in parts per million (ppm). The analytical procedure used by PSAL to determine the concentrations utilizes an Inductively Coupled Plasma – Atomic Emission Spectrometer (ICP-AES). The PCT solutions (as identified in Table 2) are grouped in three ICP-AES blocks for processing by PSAL in Table 3. Each block requires a different calibration of the ICP-AES.

Table 3: ICP-AES Calibration Blocks for Leachate Measurements

Block 1	Block 2	Block 3
std-b1-1	std-b2-1	std-b3-1
J02	J11	J17
J15	J36	J22
J34	J31	J20
J37	J06	J07
J25	J14	J19
J28	J35	J27
J26	J32	J03
std-b1-2	std-b2-2	std-b3-2
J16	J10	J33
J29	J08	J21
J04	J38	J09
J24	J13	J23
J30	J05	J18
J12	std-b2-3	J01
std-b1-3		std-b3-3

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

5.0 SUMMARY

In summary, this analytical plan provides identifiers for the PCT solutions in Table 2 and three ICP-AES calibration blocks in Table 3 for PSAL to use in conducting the boron (B), barium (Ba), cadmium (Cd), chromium (Cr), iron (Fe), lithium (Li), sodium (Na), lead (Pb), silicon (Si), thorium (Th), and uranium (U) concentration measurements for the PCT study of this select subset of glasses for SB4. The sequencing of the activities associated with each of the steps in the analytical procedure has been randomized. The size of the blocks was selected so that each block could be completed in a single work shift. If for some reason the measurements are not conducted in the sequence presented in this memorandum, the actual order should be recorded along with any explanative comments.

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

6.0 REFERENCES

[1] Peeler, D.K. and T.B. Edwards, “Model Based Assessments for the Baseline Sludge Batch 4 (Case 15C) Preparation Plan,” WSRC-TR-2006-00049, Revision 0, 2006.

[2] Shah, H.B., “Estimate of Sludge Batch 4 Calcine Composition Additional Cases for Final Recommendation,” CBU-PIT-2006-00011, Revision 0, 2006.

[3] Li, H., P. Hrma, J.D. Vienna, M. Qian, Y. Su, and D.E. Smith, “Effects of Al_2O_3, B_2O_3, Na_2O, and SiO_2 on Nepheline Formation in Borosilicate Glasses: Chemical and Physical Correlations,” **Journal of Non-Crystalline Solids**, 331, pgs. 202-216, 2003.

[4] Taylor, A.S., T.B. Edwards, J.C. George, T.K. Snyder, and D.K. Peeler, “The SRNL Glass Composition – Properties (ComPro) Database,” WSRC-RP-2004-00704, Revision 0, 2004.

[5] ASTM C-1285-2002, “Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT),” ASTM, 2002.

Appendix C

An Analytical Plan for Measuring a Second Set of PCT Solutions for the Five CCC Frit 503/SB4 Glasses (U)

(SRNL-SCS-2006-00018)

This page intentionally left blank.

SRNL-SCS-2006-00018

May 19, 2006

To: K. M. Fox, SRNL

cc:
R. A. Baker, 773-42A
D. R. Best, 786-1A (wo)
C. C. Herman, 999-W
D. K. Peeler, 999-W
I. A. Reamer, 999-1W
P. A. Toole, 786-1A (wo)
R. C. Tuckfield, 773-42A
R. J. Workman, 999-1W

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

wo – without glass identifiers

R. A. Baker, Technical Reviewer
5/22/2006
Date

R. C. Tuckfield, Manager
Statistical Consulting Section
5/23/06
Date

AN ANALYTICAL PLAN FOR MEASURING A SECOND SET OF PCT SOLUTIONS FOR THE FIVE CCC FRIT 503/SB4 GLASSES (U)

1.0 EXECUTIVE SUMMARY

Frit development efforts are underway at the Savannah River National Laboratory (SRNL) to support the processing of Sludge Batch 4 (SB4) at the Defense Waste Processing Facility (DWPF). One of the candidate frits considered during the recent assessments of the baseline preparation plan (Case 15C) was Frit 503, which was called Frit 418-m10 in the assessment. To provide experimental results to support the evaluation of this frit with SB4, five glasses were fabricated for durability testing. Testing the durability of these glasses will also complement SRNL's ongoing nepheline studies by providing additional insight into the ability of the nepheline discriminator to predict the occurrence of a nepheline primary crystalline phase for SB4 glasses and into the impact of nepheline on the durability of the SB4 glasses.

The durability of the glasses was measured using the Product Consistency Test (PCT) as defined in ASTM C-1285-2002. Two heat treatments were utilized during the fabrication of each of these glasses. Specifically, each of the 5 glasses was quenched (i.e., rapidly cooled) and cooled in accordance with the centerline-canister-cooling (ccc) regime. Both heat treatments of each glass were subjected to the PCT. The interpretation of the ccc results was somewhat unclear; so a decision was made to repeat the ccc tests.

The PCTs for the ccc glasses are to be submitted to SRNL's Process Science Analytical Laboratory (PSAL) for measurement. This memorandum provides an analytical plan for the measurement of the PCTs by PSAL.

2.0 INTRODUCTION

Frit development efforts are underway at the Savannah River National Laboratory (SRNL) to support the processing of Sludge Batch 4 (SB4) at the Defense Waste Processing Facility (DWPF). One of the candidate frits considered during the recent assessments [1] of the baseline preparation plan (Case 15C) was Frit 503, which was called Frit 418-m10 in [1]. The specific SB4 option being considered is Case 15C Blend 1 as defined by Shah [2]. To provide experimental results to support the evaluation of this frit with SB4, five glasses were fabricated for durability testing. Testing the durability of these glasses will also complement SRNL's ongoing nepheline studies by providing additional insight into the ability of the nepheline discriminator [3] to predict the occurrence of a nepheline primary crystalline phase for SB4 glasses and into the impact of nepheline on the durability of the SB4 glasses. In addition, the results from the study of these glasses will contribute needed data to the ComPro™ database [4] in anticipation of a variability study for SB4.

The durability of the glasses was measured using the Product Consistency Test (PCT) as defined in ASTM C-1285-2002 [5]. Two heat treatments were utilized during the fabrication of each of these glasses. Specifically, each of the 5 glasses was quenched (i.e., rapidly cooled) and cooled in accordance with the centerline-canister-cooling (ccc) regime. Both heat treatments of each glass were subjected to the PCT. The PCT solutions were analyzed under the auspices of the pertinent analytical plan [6], but the interpretation of the ccc results was somewhat unclear; so a decision was made to repeat the ccc tests. Specifically, the PCT response for NEPHB-04ccc and NEPHB-05ccc appeared to be inconsistent with previous trends with respect to the impact of waste loading on durability. Although the data would not have an impact on the conclusions drawn, the data did imply that the glasses may have been inadvertently switched during the durability assessment. To address this issue, the PCT responses for all five "NEPHB" ccc glasses are being reevaluated.

The PCTs for the ccc glasses are to be submitted to SRNL's Process Science Analytical Laboratory (PSAL) for measurement. This memorandum provides an analytical plan for the measurement of the PCTs by PSAL. Table 1 presents a listing of the glasses covered by this memorandum.

Table 1: Identifiers for Glasses Covered by this Plan

NEPHB-01ccc
NEPHB-02ccc
NEPHB-03ccc
NEPHB-04ccc
NEPHB-05ccc

3.0 DISCUSSION

Each of the study glasses of Table 1 is to be subjected to the PCT in triplicate. In addition to PCTs for the study glasses, triplicate PCTs are to be conducted on a sample of the Approved Reference Material – One (ARM-1) glass and a sample of the Environmental Assessment (EA) glass. Two reagent blank samples are also to be included in these tests. This results in 23 sample solutions being required to complete these PCTs.

The leachates from these tests will be diluted by adding 4 mL of 0.4 M HNO_3 to 6 mL of the leachate (a 6:10 volume to volume, v:v, dilution) before being submitted to PSAL. The leachates of EA will be further diluted (1:10 v:v) with deionized water prior to submission to PSAL in order to prevent problems with the nebulizer. Note that additional dilutions for the ccc versions of one or more of the study glasses may be needed due to a possible low durability of some of the glasses. Upon termination of the PCT, a decision is to be made (by the technicians and a PSAL representative, if called by the technician) as to whether any other dilutions are needed for these solutions to mitigate any potential gelling issues. Any extra dilutions are to be reported, and guidance is to be given as to how the dilutions are to be handled in the statistical assessment of the measurement data. More specifically, PSAL will be responsible for indicating if any additional dilutions were made and how they were, or how they should be, accounted for in the reported measurements.

Table 2 presents identifying codes, K01 through K23, for the individual solutions required for the PCTs of the select study glasses and of the standards (EA, ARM-1, and blanks). This provides a naming convention that is to be used by PSAL in analyzing the solutions and reporting the relevant concentration measurements.[6]

Table 2: Identifiers for the PCT Solutions Covered by this Plan

Original Sample	Solution Identifier	Original Sample	Solution Identifier
NEPHB-01ccc	K17	EA	K05
NEPHB-01ccc	K01	EA	K15
NEPHB-01ccc	K14	EA	K20
NEPHB-02ccc	K22	ARM-1	K18
NEPHB-02ccc	K06	ARM-1	K02
NEPHB-02ccc	K10	ARM-1	K04
NEPHB-03ccc	K23	blank	K08
NEPHB-03ccc	K16	blank	K19
NEPHB-03ccc	K11		
NEPHB-04ccc	K21		
NEPHB-04ccc	K09		
NEPHB-04ccc	K03		
NEPHB-05ccc	K13		
NEPHB-05ccc	K12		
NEPHB-05ccc	K07		

4.0 ANALYTICAL PLAN

The analytical plan for PSAL is provided in this section. Each of the solution samples submitted to PSAL is to be analyzed only once for each of the following: boron (B), barium (Ba), cadmium (Cd), chromium (Cr), iron (Fe), lithium (Li), sodium (Na), lead (Pb), silicon (Si), thorium (Th), and uranium (U). B, Li, Na, and Si are the elements that are to be used in the assessment of glass durability; the other elements are being monitored to address solution disposal issues in SRNL upon termination of the PCTs. The

---

[6] Renaming these samples ensures that they will be processed as blind samples by PSAL. This table does not contain the solution identifiers for those on the distribution list with a “wo” following their names.

measurements are to be made in parts per million (ppm). The analytical procedure used by PSAL to determine the concentrations utilizes an Inductively Coupled Plasma – Atomic Emission Spectrometer (ICP-AES). The PCT solutions (as identified in Table 2) are grouped in three ICP-AES blocks for processing by PSAL in Table 3. Each block requires a different calibration of the ICP-AES.

Table 3: ICP-AES Calibration Blocks for Leachate Measurements

Block 1	Block 2	Block 3
std-b1-1	std-b2-1	std-b3-1
K05	K16	K03
K17	K02	K10
K22	K01	K20
K18	K06	K11
std-b1-2	std-b2-2	std-b3-2
K08	K09	K04
K21	K15	K19
K23	K12	K07
K13	std-b2-3	K14
std-b1-3		std-b3-3

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

5.0 SUMMARY

In summary, this analytical plan provides identifiers for the PCT solutions in Table 2 and three ICP-AES calibration blocks in Table 3 for PSAL to use in conducting the boron (B), barium (Ba), cadmium (Cd), chromium (Cr), iron (Fe), lithium (Li), sodium (Na), lead (Pb), silicon (Si), thorium (Th), and uranium (U) concentration measurements for the PCT study of this select subset of glasses for SB4. The sequencing of the activities associated with each of the steps in the analytical procedure has been randomized. The size of the blocks was selected so that each block could be completed in a single work shift. If for some reason the measurements are not conducted in the sequence presented in this memorandum, the actual order should be recorded along with any explanative comments.

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

6.0 REFERENCES

[1] Peeler, D.K. and T.B. Edwards, “Model Based Assessments for the Baseline Sludge Batch 4 (Case 15C) Preparation Plan,” WSRC-TR-2006-00049, Revision 0, 2006.

[2] Shah, H.B., “Estimate of Sludge Batch 4 Calcine Composition Additional Cases for Final Recommendation,” CBU-PIT-2006-00011, Revision 0, 2006.

[3] Li, H., P. Hrma, J.D. Vienna, M. Qian, Y. Su, and D.E. Smith, “Effects of Al_2O_3, B_2O_3, Na_2O, and SiO_2 on Nepheline Formation in Borosilicate Glasses: Chemical and Physical Correlations,” **Journal of Non-Crystalline Solids**, 331, pgs. 202-216, 2003.

[4] Taylor, A.S., T.B. Edwards, J.C. George, T.K. Snyder, and D.K. Peeler, “The SRNL Glass Composition – Properties (ComPro™) Database,” WSRC-RP-2004-00704, Revision 0, 2004.

[5] ASTM C-1285-2002, “Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT),” ASTM, 2002.

[6] Edwards, T.B., “An Analytical Plan for Measuring PCT Solutions for a Set of Five Frit 503/SB4 Glasses,” SRNL-SCS-2006-00012, March 31, 2006.

Appendix D

Tables and Exhibits Supporting the Analysis of the Chemical Composition Measurements of the Frit 503 – SB4 Study Glasses

Table D1. Targeted Oxide Concentrations (as wt%'s) for the Frit 503/SB4 Study Glasses

Glass #	Al2O3	B2O3	BaO	CaO	Ce2O3	Cr2O3	CuO	Fe2O3	K2O	La2O3	Li2O	MgO	MnO	Na2O	NiO	PbO	SO4	SiO2	ThO2	TiO2	U3O8	ZnO	ZrO2	Sum
NEPHB-01	7.4417	9.8000	0.0378	0.7162	0.0449	0.0636	0.0179	7.9697	0.1032	0.0325	5.6000	0.7486	1.6440	9.4239	0.4731	0.0271	0.4014	53.0340	0.0197	0.0080	2.2921	0.0293	0.0711	100.000
NEPHB-02	8.6820	9.1000	0.0441	0.8356	0.0524	0.0742	0.0209	9.2979	0.1204	0.0379	5.2000	0.8734	1.9180	10.3279	0.5520	0.0317	0.4683	49.5396	0.0230	0.0093	2.6741	0.0342	0.0830	100.000
NEPHB-03	9.9223	8.4000	0.0504	0.9550	0.0599	0.0848	0.0239	10.6262	0.1376	0.0434	4.8000	0.9982	2.1920	11.2318	0.6308	0.0362	0.5352	46.0453	0.0263	0.0107	3.0561	0.0390	0.0948	100.000
NEPHB-04	11.1626	7.7000	0.0567	1.0743	0.0674	0.0954	0.0269	11.9545	0.1548	0.0488	4.4000	1.1229	2.4660	12.1358	0.7097	0.0407	0.6021	42.5509	0.0296	0.0120	3.4381	0.0439	0.1067	100.000
NEPHB-05	12.4029	7.0000	0.0630	1.1937	0.0749	0.1061	0.0299	13.2827	0.1721	0.0542	4.0000	1.2477	2.7401	13.0398	0.7886	0.0452	0.6690	39.0566	0.0329	0.0133	3.8201	0.0488	0.1185	100.000

Table D2. Measured Elemental Concentrations (wt%) for Samples Prepared Using Lithium Metaborate

Glass ID	Laboratory ID	Block	Analytical Sequence	Ba	Ca	Ce	Cr	Cu	K	La	Mg	Mn	Na	Pb	S	Th	Ti	Zn	Zr
Batch 1	BCHLM11	1	1	0.127	0.816	<0.010	0.067	0.294	2.44	<0.010	0.816	1.36	6.91	<0.020	<0.100	<0.010	0.393	0.035	0.066
Ustd	UstdLM11	1	2	<0.010	0.852	<0.010	0.154	<0.010	2.33	<0.010	0.672	2.29	9.20	<0.020	<0.100	0.048	0.551	<0.010	<0.010
NEPHB-04	I02LM11	1	3	0.047	0.691	0.044	0.044	0.029	0.133	0.037	0.639	2.01	9.11	0.037	0.187	0.088	<0.010	0.063	0.069
NEPHB-05	I04LM21	1	4	0.054	0.771	0.057	0.048	0.029	0.148	0.041	0.712	2.20	9.65	0.044	0.197	0.103	<0.010	0.047	0.082
NEPHB-04	I02LM21	1	5	0.047	0.690	0.044	0.044	0.034	0.133	0.037	0.641	1.98	8.98	0.038	0.180	0.090	<0.010	0.047	0.070
NEPHB-02	I01LM11	1	6	0.037	0.545	0.038	0.037	0.021	0.099	0.028	0.500	1.52	7.69	0.031	0.144	0.071	<0.010	0.032	0.046
NEPHB-03	I05LM21	1	7	0.043	0.625	0.034	0.042	0.026	0.114	0.031	0.576	1.70	8.38	0.033	0.167	0.081	<0.010	0.046	0.066
Batch 1	BCHLM12	1	8	0.126	0.811	<0.010	0.067	0.297	2.46	<0.010	0.820	1.33	6.85	<0.020	<0.100	<0.010	0.390	0.035	0.066
Ustd	UstdLM12	1	9	<0.010	0.880	<0.010	0.154	<0.010	2.39	<0.010	0.679	2.19	8.89	<0.020	<0.100	0.049	0.554	<0.010	<0.010
NEPHB-05	I04LM11	1	10	0.055	0.778	0.056	0.049	0.030	0.144	0.041	0.713	2.22	9.84	0.044	0.197	0.103	<0.010	0.045	0.083
NEPHB-03	I05LM11	1	11	0.044	0.642	0.035	0.042	0.026	0.120	0.032	0.586	1.78	8.31	0.033	0.171	0.083	<0.010	0.047	0.068
NEPHB-01	I03LM21	1	12	0.033	0.483	0.027	0.025	0.020	0.083	0.023	0.436	1.39	7.11	0.036	0.128	0.063	<0.010	0.027	0.041
NEPHB-01	I03LM11	1	13	0.032	0.504	0.027	0.025	0.019	0.082	0.023	0.434	1.36	7.06	0.035	0.130	0.062	<0.010	0.040	0.040
NEPHB-02	I01LM21	1	14	0.037	0.555	0.039	0.037	0.022	0.104	0.028	0.498	1.59	7.40	0.031	0.147	0.071	<0.010	0.032	0.047
Batch 1	BCHLM13	1	15	0.127	0.837	<0.010	0.067	0.300	2.48	<0.010	0.818	1.42	6.84	<0.020	<0.100	<0.010	0.394	0.035	0.067
Ustd	UstdLM13	1	16	<0.010	0.871	<0.010	0.154	<0.010	2.38	<0.010	0.670	2.29	9.07	<0.020	<0.100	0.049	0.550	<0.010	<0.010
Batch 1	BCHLM21	2	1	0.127	0.824	<0.010	0.066	0.297	2.45	<0.010	0.821	1.38	7.13	<0.020	<0.100	<0.010	0.392	0.034	0.066
Ustd	UstdLM21	2	2	<0.010	0.883	<0.010	0.154	<0.010	2.37	<0.010	0.682	2.27	9.47	<0.020	<0.100	0.049	0.557	<0.010	<0.010
NEPHB-02	I01LM22	2	3	0.037	0.546	0.039	0.036	0.021	0.101	0.028	0.506	1.49	7.76	0.030	0.148	0.072	<0.010	0.031	0.046
NEPHB-04	I02LM22	2	4	0.046	0.704	0.045	0.043	0.034	0.134	0.036	0.646	1.94	9.22	0.037	0.178	0.089	<0.010	0.046	0.070
NEPHB-01	I03LM12	2	5	0.032	0.508	0.027	0.024	0.018	0.080	0.023	0.435	1.32	7.25	0.033	0.125	0.062	<0.010	0.039	0.040
NEPHB-03	I05LM12	2	6	0.043	0.628	0.035	0.041	0.024	0.115	0.031	0.582	1.78	8.44	0.032	0.164	0.082	<0.010	0.046	0.066
NEPHB-01	I03LM22	2	7	0.032	0.481	0.027	0.024	0.019	0.080	0.023	0.439	1.34	7.33	0.034	0.126	0.062	<0.010	0.026	0.040
Batch 1	BCHLM22	2	8	0.127	0.815	<0.010	0.066	0.296	2.47	<0.010	0.824	1.39	7.03	<0.020	<0.100	<0.010	0.390	0.033	0.066
Ustd	UstdLM22	2	9	<0.010	0.876	<0.010	0.153	<0.010	2.36	<0.010	0.678	2.25	9.05	<0.020	<0.100	0.047	0.551	<0.010	<0.010
NEPHB-04	I02LM12	2	10	0.046	0.699	0.044	0.043	0.028	0.132	0.036	0.642	1.98	9.47	0.036	0.179	0.087	<0.010	0.062	0.069
NEPHB-02	I01LM12	2	11	0.037	0.541	0.039	0.037	0.021	0.101	0.028	0.505	1.54	7.78	0.031	0.144	0.073	<0.010	0.031	0.046
NEPHB-05	I04LM22	2	12	0.054	0.788	0.057	0.047	0.029	0.147	0.040	0.718	2.12	10.2	0.043	0.196	0.103	<0.010	0.046	0.082
NEPHB-05	I04LM12	2	13	0.055	0.787	0.057	0.048	0.031	0.145	0.041	0.726	2.13	10.1	0.044	0.197	0.105	<0.010	0.044	0.083
NEPHB-03	I05LM22	2	14	0.043	0.629	0.035	0.041	0.025	0.115	0.031	0.577	1.74	8.48	0.032	0.167	0.082	<0.010	0.048	0.066
Batch 1	BCHLM23	2	15	0.126	0.832	<0.010	0.066	0.299	2.48	<0.010	0.820	1.39	7.14	<0.020	<0.100	<0.010	0.394	0.033	0.066
Ustd	UstdLM23	2	16	<0.010	0.873	<0.010	0.154	<0.010	2.39	<0.010	0.688	2.24	9.26	<0.020	<0.100	0.048	0.556	<0.010	<0.010

Table D3. Measured Elemental Concentrations (wt%) for Samples Prepared Using Peroxide Fusion

Glass ID	PSAL ID	Block	Analytical Sequence	Al	B	Fe	Li	Ni	Si	U
Batch 1	BCHPF11	1	1	2.47	2.56	8.70	2.13	0.500	22.3	<0.100
Ustd	UstdPF11	1	2	2.12	2.91	8.80	1.40	0.716	19.6	1.88
NEPHB-05	I04PF21	1	3	6.37	2.08	9.02	1.77	0.532	17.9	3.12
NEPHB-01	I03PF11	1	4	3.98	3.00	5.39	2.46	0.298	24.4	1.89
NEPHB-03	I05PF11	1	5	5.27	2.58	7.27	2.13	0.426	21.3	2.38
NEPHB-01	I03PG21	1	6	3.98	2.99	5.36	2.49	0.308	24.6	1.82
NEPHB-02	I01PF21	1	7	4.60	2.88	6.47	2.32	0.387	23.1	2.15
Batch 1	BCHPF12	1	8	2.51	2.44	9.72	2.00	0.555	23.6	<0.100
Ustd	UstdPF12	1	9	2.14	2.84	9.74	1.40	0.794	20.7	1.88
NEPHB-04	I02PF21	1	10	5.93	2.74	8.24	2.12	0.502	20.2	2.79
NEPHB-04	I02PF11	1	11	5.98	2.46	8.22	1.99	0.493	20.2	2.78
NEPHB-02	I01PF11	1	12	4.57	2.86	6.34	2.28	0.371	22.9	2.13
NEPHB-05	I04PF11	1	13	6.50	2.22	9.30	1.78	0.556	18.4	3.08
NEPHB-03	I05PF21	1	14	5.17	2.61	7.45	2.12	0.425	21.4	2.39
Batch 1	BCHPF13	1	15	2.48	2.56	9.57	2.00	0.550	23.2	<0.100
Ustd	Ustdpf113	1	16	2.07	2.80	9.55	1.37	0.773	20.3	1.85
Batch 1	BCHPF21	2	1	2.55	2.43	9.54	2.04	0.521	23.5	<0.100
Ustd	UstdPF21	2	2	2.25	2.95	9.32	1.41	0.748	20.4	1.98
NEPHB-03	I05PF22	2	3	5.30	2.72	7.44	2.16	0.393	21.6	2.47
NEPHB-02	I01PF22	2	4	4.70	2.94	6.39	2.35	0.363	23.1	2.23
NEPHB-04	I02PF12	2	5	6.07	2.43	8.08	2.02	0.467	20.1	2.76
NEPHB-03	I05PF12	2	6	5.36	2.65	7.36	2.18	0.401	21.6	2.40
NEPHB-05	I04PF22	2	7	6.55	2.20	9.25	1.80	0.520	18.2	3.11
Batch 1	BCHPF22	2	8	2.56	2.44	9.66	2.02	0.524	23.5	<0.100
Ustd	UstdPF22	2	9	2.16	2.85	9.69	1.41	0.772	20.5	1.93
NEPHB-01	I03PF22	2	10	4.08	3.09	5.28	2.54	0.288	24.5	1.88
NEPHB-05	I04PF12	2	11	6.63	2.22	8.92	1.81	0.493	18.1	3.20
NEPHB-04	I02PF22	2	12	5.98	2.62	7.76	2.12	0.442	19.8	2.80
NEPHB-01	I03PF12	2	13	4.04	3.04	5.33	2.50	0.274	24.4	1.87
NEPHB-02	I01PF12	2	14	4.64	2.84	6.21	2.32	0.344	22.8	2.15
Batch 1	BCHPF23	2	15	2.57	2.61	9.48	2.06	0.520	23.6	<0.100
Ustd	UstdPF23	2	16	2.18	2.82	9.47	1.42	0.757	20.5	1.91

Table D4. Average Measured and Bias-Corrected Chemical Compositions Versus Targeted Compositions by Oxide by Study Glass

(100 -Batch 1; 200 -U std)

Glass ID	Glass #	Oxide	Measured (wt%)	Measured Bias-Corrected (wt%)	Targeted (wt%)	Diff of Measured	Diff of Meas BC	% Diff of Measured	% Diff of Meas BC
NEPHB-01	1	Al2O3 (wt%)	7.5958	7.7702	7.4417	0.1541	0.3285	2.1%	4.4%
NEPHB-01	1	B2O3 (wt%)	9.7563	9.4015	9.8000	-0.0437	-0.3985	-0.4%	-4.1%
NEPHB-01	1	BaO (wt%)	0.0360	0.0384	0.0378	-0.0018	0.0006	-4.7%	1.7%
NEPHB-01	1	CaO (wt%)	0.6912	0.7327	0.7162	-0.0250	0.0165	-3.5%	2.3%
NEPHB-01	1	Ce2O3 (wt%)	0.0316	0.0316	0.0449	-0.0133	-0.0133	-29.6%	-29.6%
NEPHB-01	1	Cr2O3 (wt%)	0.0358	0.0394	0.0636	-0.0278	-0.0242	-43.7%	-38.0%
NEPHB-01	1	CuO (wt%)	0.0238	0.0255	0.0179	0.0059	0.0076	32.9%	42.5%
NEPHB-01	1	Fe2O3 (wt%)	7.6346	7.2606	7.9697	-0.3351	-0.7091	-4.2%	-8.9%
NEPHB-01	1	K2O (wt%)	0.0979	0.1097	0.1032	-0.0053	0.0065	-5.2%	6.3%
NEPHB-01	1	La2O3 (wt%)	0.0270	0.0270	0.0325	-0.0055	-0.0055	-17.0%	-17.0%
NEPHB-01	1	Li2O (wt%)	5.3769	5.4179	5.6000	-0.2231	-0.1821	-4.0%	-3.3%
NEPHB-01	1	MgO (wt%)	0.7230	0.7546	0.7486	-0.0256	0.0060	-3.4%	0.8%
NEPHB-01	1	MnO (wt%)	1.7463	1.6939	1.6440	0.1023	0.0499	6.2%	3.0%
NEPHB-01	1	Na2O (wt%)	9.6888	9.2666	9.4239	0.2649	-0.1573	2.8%	-1.7%
NEPHB-01	1	NiO (wt%)	0.3716	0.4149	0.4731	-0.1015	-0.0582	-21.5%	-12.3%
NEPHB-01	1	PbO (wt%)	0.0372	0.0372	0.0271	0.0101	0.0101	37.1%	37.1%
NEPHB-01	1	SO4 (wt%)	0.3812	0.3812	0.4014	-0.0202	-0.0202	-5.0%	-5.0%
NEPHB-01	1	SiO2 (wt%)	52.3594	52.7970	53.0340	-0.6746	-0.2370	-1.3%	-0.4%
NEPHB-01	1	ThO2 (wt%)	0.0708	0.0708	0.0197	0.0511	0.0511	259.6%	259.6%
NEPHB-01	1	TiO2 (wt%)	0.0083	0.0086	0.0080	0.0003	0.0006	4.3%	7.9%
NEPHB-01	1	U3O8 (wt%)	2.1992	2.3560	2.2921	-0.0929	0.0639	-4.1%	2.8%
NEPHB-01	1	ZnO (wt%)	0.0411	0.0411	0.0293	0.0118	0.0118	40.2%	40.2%
NEPHB-01	1	ZrO2 (wt%)	0.0544	0.0544	0.0711	-0.0167	-0.0167	-23.5%	-23.5%
NEPHB-01	1	Sum (wt%)	98.9881	98.7310	99.9998	-1.0117	-1.2688	-1.0%	-1.3%
NEPHB-02	2	Al2O3 (wt%)	8.7437	8.9445	8.6820	0.0617	0.2625	0.7%	3.0%
NEPHB-02	2	B2O3 (wt%)	9.2733	8.9357	9.1000	0.1733	-0.1643	1.9%	-1.8%
NEPHB-02	2	BaO (wt%)	0.0413	0.0441	0.0441	-0.0028	0.0000	-6.3%	0.0%
NEPHB-02	2	CaO (wt%)	0.7650	0.8110	0.8356	-0.0706	-0.0246	-8.4%	-2.9%
NEPHB-02	2	Ce2O3 (wt%)	0.0454	0.0454	0.0524	-0.0070	-0.0070	-13.4%	-13.4%
NEPHB-02	2	Cr2O3 (wt%)	0.0537	0.0591	0.0742	-0.0205	-0.0151	-27.6%	-20.3%
NEPHB-02	2	CuO (wt%)	0.0266	0.0285	0.0209	0.0057	0.0076	27.3%	36.5%
NEPHB-02	2	Fe2O3 (wt%)	9.0822	8.6374	9.2979	-0.2157	-0.6605	-2.3%	-7.1%
NEPHB-02	2	K2O (wt%)	0.1220	0.1367	0.1204	0.0016	0.0163	1.3%	13.6%
NEPHB-02	2	La2O3 (wt%)	0.0328	0.0328	0.0379	-0.0051	-0.0051	-13.4%	-13.4%
NEPHB-02	2	Li2O (wt%)	4.9893	5.0274	5.2000	-0.2107	-0.1726	-4.1%	-3.3%
NEPHB-02	2	MgO (wt%)	0.8329	0.8693	0.8734	-0.0405	-0.0041	-4.6%	-0.5%
NEPHB-02	2	MnO (wt%)	1.9820	1.9224	1.9180	0.0640	0.0044	3.3%	0.2%
NEPHB-02	2	Na2O (wt%)	10.3223	9.8725	10.3279	-0.0056	-0.4554	-0.1%	-4.4%
NEPHB-02	2	NiO (wt%)	0.4661	0.5205	0.5520	-0.0859	-0.0315	-15.6%	-5.7%
NEPHB-02	2	PbO (wt%)	0.0331	0.0331	0.0317	0.0014	0.0014	4.5%	4.5%
NEPHB-02	2	SO4 (wt%)	0.4367	0.4367	0.4683	-0.0316	-0.0316	-6.8%	-6.8%
NEPHB-02	2	SiO2 (wt%)	49.1504	49.5612	49.5396	-0.3892	0.0216	-0.8%	0.0%
NEPHB-02	2	ThO2 (wt%)	0.0816	0.0816	0.0230	0.0586	0.0586	255.0%	255.0%
NEPHB-02	2	TiO2 (wt%)	0.0083	0.0086	0.0093	-0.0010	-0.0007	-10.3%	-7.2%
NEPHB-02	2	U3O8 (wt%)	2.5530	2.7347	2.6741	-0.1211	0.0606	-4.5%	2.3%
NEPHB-02	2	ZnO (wt%)	0.0392	0.0392	0.0342	0.0050	0.0050	14.7%	14.7%
NEPHB-02	2	ZrO2 (wt%)	0.0625	0.0625	0.0830	-0.0205	-0.0205	-24.7%	-24.7%
NEPHB-02	2	Sum (wt%)	99.1434	98.8451	99.9999	-0.8565	-1.1548	-0.9%	-1.2%
NEPHB-03	3	Al2O3 (wt%)	9.9671	10.1959	9.9223	0.0448	0.2736	0.5%	2.8%
NEPHB-03	3	B2O3 (wt%)	8.5005	8.1916	8.4000	0.1005	-0.2084	1.2%	-2.5%
NEPHB-03	3	BaO (wt%)	0.0483	0.0516	0.0504	-0.0021	0.0012	-4.2%	2.3%
NEPHB-03	3	CaO (wt%)	0.8829	0.9360	0.9550	-0.0721	-0.0190	-7.6%	-2.0%
NEPHB-03	3	Ce2O3 (wt%)	0.0407	0.0407	0.0599	-0.0192	-0.0192	-32.0%	-32.0%

Table D4. Average Measured and Bias-Corrected Chemical Compositions Versus Targeted Compositions by Oxide by Study Glass (continued)

(100 -Batch 1; 200 -U std)

Glass ID	Glass #	Oxide	Measured (wt%)	Measured Bias-Corrected (wt%)	Targeted (wt%)	Diff of Measured	Diff of Meas BC	% Diff of Measured	% Diff of Meas BC
NEPHB-03	3	Cr2O3 (wt%)	0.0607	0.0668	0.0848	-0.0241	-0.0180	-28.5%	-21.3%
NEPHB-03	3	CuO (wt%)	0.0316	0.0339	0.0239	0.0077	0.0100	32.3%	41.9%
NEPHB-03	3	Fe2O3 (wt%)	10.5512	10.0331	10.6262	-0.0750	-0.5931	-0.7%	-5.6%
NEPHB-03	3	K2O (wt%)	0.1397	0.1567	0.1376	0.0021	0.0191	1.6%	13.9%
NEPHB-03	3	La2O3 (wt%)	0.0367	0.0367	0.0434	-0.0068	-0.0068	-15.6%	-15.6%
NEPHB-03	3	Li2O (wt%)	4.6234	4.6586	4.8000	-0.1766	-0.1414	-3.7%	-2.9%
NEPHB-03	3	MgO (wt%)	0.9622	1.0043	0.9982	-0.0360	0.0061	-3.6%	0.6%
NEPHB-03	3	MnO (wt%)	2.2596	2.1914	2.1920	0.0676	-0.0006	3.1%	0.0%
NEPHB-03	3	Na2O (wt%)	11.3266	10.8344	11.2318	0.0948	-0.3974	0.8%	-3.5%
NEPHB-03	3	NiO (wt%)	0.5233	0.5844	0.6308	-0.1075	-0.0464	-17.0%	-7.4%
NEPHB-03	3	PbO (wt%)	0.0350	0.0350	0.0362	-0.0012	-0.0012	-3.3%	-3.3%
NEPHB-03	3	SO4 (wt%)	0.5011	0.5011	0.5352	-0.0341	-0.0341	-6.4%	-6.4%
NEPHB-03	3	SiO2 (wt%)	45.9415	46.3220	46.0453	-0.1038	0.2767	-0.2%	0.6%
NEPHB-03	3	ThO2 (wt%)	0.0933	0.0933	0.0263	0.0670	0.0670	254.8%	254.8%
NEPHB-03	3	TiO2 (wt%)	0.0083	0.0086	0.0107	-0.0024	-0.0021	-22.1%	-19.3%
NEPHB-03	3	U3O8 (wt%)	2.8419	3.0443	3.0561	-0.2142	-0.0118	-7.0%	-0.4%
NEPHB-03	3	ZnO (wt%)	0.0582	0.0582	0.0390	0.0192	0.0192	49.2%	49.2%
NEPHB-03	3	ZrO2 (wt%)	0.0898	0.0898	0.0948	-0.0050	-0.0050	-5.2%	-5.2%
NEPHB-03	3	Sum (wt%)	99.5235	99.1684	99.9999	-0.4764	-0.8315	-0.5%	-0.8%
NEPHB-04	4	Al2O3 (wt%)	11.3181	11.5787	11.1626	0.1555	0.4161	1.4%	3.7%
NEPHB-04	4	B2O3 (wt%)	8.2510	7.9498	7.7000	0.5510	0.2498	7.2%	3.2%
NEPHB-04	4	BaO (wt%)	0.0519	0.0554	0.0567	-0.0048	-0.0013	-8.4%	-2.2%
NEPHB-04	4	CaO (wt%)	0.9738	1.0324	1.0743	-0.1005	-0.0419	-9.4%	-3.9%
NEPHB-04	4	Ce2O3 (wt%)	0.0518	0.0518	0.0674	-0.0156	-0.0156	-23.1%	-23.1%
NEPHB-04	4	Cr2O3 (wt%)	0.0636	0.0700	0.0954	-0.0318	-0.0254	-33.4%	-26.6%
NEPHB-04	4	CuO (wt%)	0.0391	0.0420	0.0269	0.0122	0.0151	45.4%	56.0%
NEPHB-04	4	Fe2O3 (wt%)	11.5448	10.9809	11.9545	-0.4097	-0.9736	-3.4%	-8.1%
NEPHB-04	4	K2O (wt%)	0.1602	0.1796	0.1548	0.0054	0.0248	3.5%	16.0%
NEPHB-04	4	La2O3 (wt%)	0.0428	0.0428	0.0488	-0.0060	-0.0060	-12.3%	-12.3%
NEPHB-04	4	Li2O (wt%)	4.4404	4.4742	4.4000	0.0404	0.0742	0.9%	1.7%
NEPHB-04	4	MgO (wt%)	1.0646	1.1112	1.1229	-0.0583	-0.0117	-5.2%	-1.0%
NEPHB-04	4	MnO (wt%)	2.5533	2.4765	2.4660	0.0873	0.0105	3.5%	0.4%
NEPHB-04	4	Na2O (wt%)	12.3949	11.8544	12.1358	0.2591	-0.2814	2.1%	-2.3%
NEPHB-04	4	NiO (wt%)	0.6057	0.6763	0.7097	-0.1040	-0.0334	-14.7%	-4.7%
NEPHB-04	4	PbO (wt%)	0.0399	0.0399	0.0407	-0.0008	-0.0008	-2.1%	-2.1%
NEPHB-04	4	SO4 (wt%)	0.5423	0.5423	0.6021	-0.0598	-0.0598	-9.9%	-9.9%
NEPHB-04	4	SiO2 (wt%)	42.9464	43.3078	42.5509	0.3955	0.7569	0.9%	1.8%
NEPHB-04	4	ThO2 (wt%)	0.1007	0.1007	0.0296	0.0711	0.0711	240.2%	240.2%
NEPHB-04	4	TiO2 (wt%)	0.0083	0.0086	0.0120	-0.0037	-0.0034	-30.5%	-28.1%
NEPHB-04	4	U3O8 (wt%)	3.2811	3.5155	3.4381	-0.1570	0.0774	-4.6%	2.3%
NEPHB-04	4	ZnO (wt%)	0.0678	0.0678	0.0439	0.0239	0.0239	54.5%	54.5%
NEPHB-04	4	ZrO2 (wt%)	0.0939	0.0939	0.1067	-0.0128	-0.0128	-12.0%	-12.0%
NEPHB-04	4	Sum (wt%)	100.6366	100.2526	99.9998	0.6368	0.2528	0.6%	0.3%
NEPHB-05	5	Al2O3 (wt%)	12.3054	12.5876	12.4029	-0.0975	0.1847	-0.8%	1.5%
NEPHB-05	5	B2O3 (wt%)	7.0194	6.7642	7.0000	0.0194	-0.2358	0.3%	-3.4%
NEPHB-05	5	BaO (wt%)	0.0608	0.0650	0.0630	-0.0022	0.0020	-3.4%	3.1%
NEPHB-05	5	CaO (wt%)	1.0928	1.1584	1.1937	-0.1009	-0.0353	-8.5%	-3.0%
NEPHB-05	5	Ce2O3 (wt%)	0.0665	0.0665	0.0749	-0.0084	-0.0084	-11.3%	-11.3%
NEPHB-05	5	Cr2O3 (wt%)	0.0702	0.0772	0.1061	-0.0359	-0.0289	-33.9%	-27.2%
NEPHB-05	5	CuO (wt%)	0.0372	0.0399	0.0299	0.0073	0.0100	24.6%	33.6%
NEPHB-05	5	Fe2O3 (wt%)	13.0424	12.4031	13.2827	-0.2403	-0.8796	-1.8%	-6.6%
NEPHB-05	5	K2O (wt%)	0.1759	0.1972	0.1721	0.0038	0.0251	2.2%	14.6%
NEPHB-05	5	La2O3 (wt%)	0.0478	0.0478	0.0542	-0.0064	-0.0064	-11.8%	-11.8%
NEPHB-05	5	Li2O (wt%)	3.8537	3.8831	4.0000	-0.1463	-0.1169	-3.7%	-2.9%

Table D4. Average Measured and Bias-Corrected Chemical Compositions Versus Targeted Compositions by Oxide by Study Glass (continued)

(100 -Batch 1; 200 -U std)

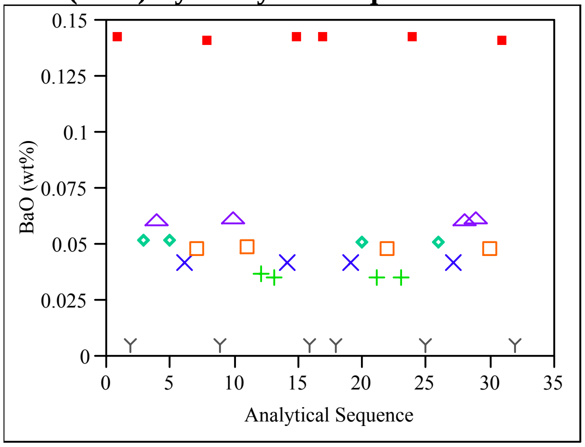
Glass ID	Glass #	Oxide	Measured (wt%)	Measured Bias-Corrected (wt%)	Targeted (wt%)	Diff of Measured	Diff of Meas BC	% Diff of Measured	% Diff of Meas BC
NEPHB-05	5	MgO (wt%)	1.1894	1.2414	1.2477	-0.0583	-0.0063	-4.7%	-0.5%
NEPHB-05	5	MnO (wt%)	2.7987	2.7147	2.7401	0.0586	-0.0254	2.1%	-0.9%
NEPHB-05	5	Na2O (wt%)	13.4092	12.8237	13.0398	0.3694	-0.2161	2.8%	-1.7%
NEPHB-05	5	NiO (wt%)	0.6684	0.7464	0.7886	-0.1202	-0.0422	-15.2%	-5.4%
NEPHB-05	5	PbO (wt%)	0.0471	0.0471	0.0452	0.0019	0.0019	4.3%	4.3%
NEPHB-05	5	SO4 (wt%)	0.5894	0.5894	0.6690	-0.0796	-0.0796	-11.9%	-11.9%
NEPHB-05	5	SiO2 (wt%)	38.8283	39.1524	39.0566	-0.2283	0.0958	-0.6%	0.2%
NEPHB-05	5	ThO2 (wt%)	0.1178	0.1178	0.0329	0.0849	0.0849	258.0%	258.0%
NEPHB-05	5	TiO2 (wt%)	0.0083	0.0086	0.0133	-0.0050	-0.0047	-37.3%	-35.1%
NEPHB-05	5	U3O8 (wt%)	3.6879	3.9507	3.8201	-0.1322	0.1306	-3.5%	3.4%
NEPHB-05	5	ZnO (wt%)	0.0566	0.0566	0.0488	0.0078	0.0078	16.1%	16.1%
NEPHB-05	5	ZrO2 (wt%)	0.1114	0.1114	0.1185	-0.0071	-0.0071	-6.0%	-6.0%
NEPHB-05	5	Sum (wt%)	99.2847	98.8503	100.0001	-0.7154	-1.1498	-0.7%	-1.1%
Batch 1	100	Al2O3 (wt%)	4.7678	4.8770	4.8770	-0.1092	0.0000	-2.2%	0.0%
Batch 1	100	B2O3 (wt%)	8.0712	7.7770	7.7770	0.2942	0.0000	3.8%	0.0%
Batch 1	100	BaO (wt%)	0.1414	0.1510	0.1510	-0.0096	0.0000	-6.3%	0.0%
Batch 1	100	CaO (wt%)	1.1508	1.2200	1.2200	-0.0692	0.0000	-5.7%	0.0%
Batch 1	100	Ce2O3 (wt%)	0.0059	0.0059	0.0000	0.0059	0.0059		
Batch 1	100	Cr2O3 (wt%)	0.0972	0.1070	0.1070	-0.0098	0.0000	-9.2%	0.0%
Batch 1	100	CuO (wt%)	0.3720	0.3990	0.3990	-0.0270	0.0000	-6.8%	0.0%
Batch 1	100	Fe2O3 (wt%)	13.5035	12.8390	12.8390	0.6645	0.0000	5.2%	0.0%
Batch 1	100	K2O (wt%)	2.9673	3.3270	3.3270	-0.3597	0.0000	-10.8%	0.0%
Batch 1	100	La2O3 (wt%)	0.0059	0.0059	0.0000	0.0059	0.0059		
Batch 1	100	Li2O (wt%)	4.3955	4.4290	4.4290	-0.0335	0.0000	-0.8%	0.0%
Batch 1	100	MgO (wt%)	1.3595	1.4190	1.4190	-0.0595	0.0000	-4.2%	0.0%
Batch 1	100	MnO (wt%)	1.7797	1.7260	1.7260	0.0537	0.0000	3.1%	0.0%
Batch 1	100	Na2O (wt%)	9.4135	9.0030	9.0030	0.4105	0.0000	4.6%	0.0%
Batch 1	100	NiO (wt%)	0.6723	0.7510	0.7510	-0.0787	0.0000	-10.5%	0.0%
Batch 1	100	PbO (wt%)	0.0108	0.0108	0.0000	0.0108	0.0108		
Batch 1	100	SO4 (wt%)	0.1498	0.1498	0.0000	0.1498	0.1498		
Batch 1	100	SiO2 (wt%)	49.8100	50.2200	50.2200	-0.4100	0.0000	-0.8%	0.0%
Batch 1	100	ThO2 (wt%)	0.0057	0.0057	0.0000	0.0057	0.0057		
Batch 1	100	TiO2 (wt%)	0.6541	0.6770	0.6770	-0.0229	0.0000	-3.4%	0.0%
Batch 1	100	U3O8 (wt%)	0.0590	0.0632	0.0000	0.0590	0.0632		
Batch 1	100	ZnO (wt%)	0.0425	0.0425	0.0000	0.0425	0.0425		
Batch 1	100	ZrO2 (wt%)	0.0894	0.0894	0.0980	-0.0086	-0.0086	-8.8%	-8.8%
Batch 1	100	Sum (wt%)	99.5249	99.2951	99.0200	0.5049	0.2751	0.5%	0.3%
Ustd	200	Al2O3 (wt%)	4.0687	4.1615	4.1000	-0.0313	0.0615	-0.8%	1.5%
Ustd	200	B2O3 (wt%)	9.2143	8.8788	9.2090	0.0053	-0.3302	0.1%	-3.6%
Ustd	200	BaO (wt%)	0.0056	0.0060	0.0000	0.0056	0.0060		
Ustd	200	CaO (wt%)	1.2208	1.2942	1.3010	-0.0802	-0.0068	-6.2%	-0.5%
Ustd	200	Ce2O3 (wt%)	0.0059	0.0059	0.0000	0.0059	0.0059		
Ustd	200	Cr2O3 (wt%)	0.2248	0.2475	0.0000	0.2248	0.2475		
Ustd	200	CuO (wt%)	0.0063	0.0067	0.0000	0.0063	0.0067		
Ustd	200	Fe2O3 (wt%)	13.4797	12.8172	13.1960	0.2837	-0.3788	2.1%	-2.9%
Ustd	200	K2O (wt%)	2.8549	3.2009	2.9990	-0.1441	0.2019	-4.8%	6.7%
Ustd	200	La2O3 (wt%)	0.0059	0.0059	0.0000	0.0059	0.0059		
Ustd	200	Li2O (wt%)	3.0176	3.0407	3.0570	-0.0394	-0.0163	-1.3%	-0.5%
Ustd	200	MgO (wt%)	1.1246	1.1738	1.2100	-0.0854	-0.0362	-7.1%	-3.0%
Ustd	200	MnO (wt%)	2.9117	2.8239	2.8920	0.0197	-0.0681	0.7%	-2.4%
Ustd	200	Na2O (wt%)	12.3432	11.8060	11.7950	0.5482	0.0110	4.6%	0.1%
Ustd	200	NiO (wt%)	0.9671	1.0805	1.1200	-0.1529	-0.0395	-13.7%	-3.5%
Ustd	200	PbO (wt%)	0.0108	0.0108	0.0000	0.0108	0.0108		
Ustd	200	SO4 (wt%)	0.1498	0.1498	0.0000	0.1498	0.1498		

Table D4. Average Measured and Bias-Corrected Chemical Compositions Versus Targeted Compositions by Oxide by Study Glass (continued)

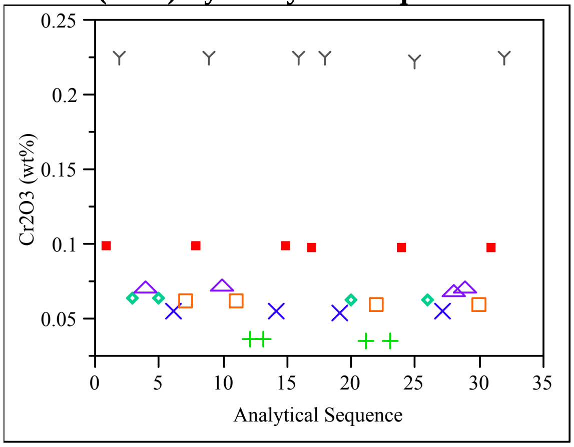
(100 -Batch 1; 200 -U std)

Glass ID	Glass #	Oxide	Measured (wt%)	Measured Bias-Corrected (wt%)	Targeted (wt%)	Diff of Measured	Diff of Meas BC	% Diff of Measured	% Diff of Meas BC
Ustd	200	SiO2 (wt%)	43.4991	43.8591	45.3530	-1.8539	-1.4939	-4.1%	-3.3%
Ustd	200	ThO2 (wt%)	0.0550	0.0550	0.0000	0.0550	0.0550		
Ustd	200	TiO2 (wt%)	0.9227	0.9549	1.0490	-0.1263	-0.0941	-12.0%	-9.0%
Ustd	200	U3O8 (wt%)	2.2464	2.4060	2.4060	-0.1596	0.0000	-6.6%	0.0%
Ustd	200	ZnO (wt%)	0.0062	0.0062	0.0000	0.0062	0.0062		
Ustd	200	ZrO2 (wt%)	0.0068	0.0068	0.0000	0.0068	0.0068		
Ustd	200	Sum (wt%)	98.3477	97.9979	99.6870	-1.3393	-1.6891	-1.3%	-1.7%

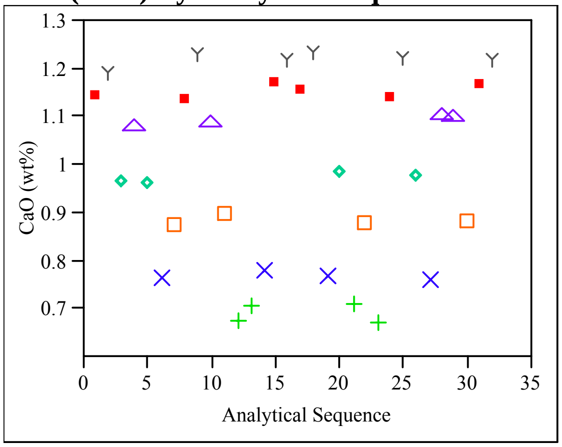
Exhibit D1. Oxide Measurements in Analytical Sequence for Samples Prepared Using the LM Method



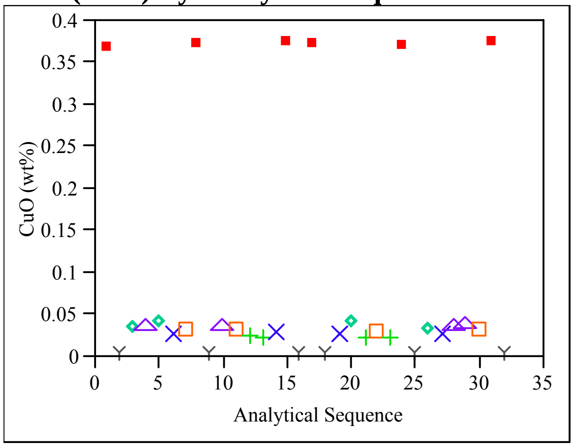
BaO (wt%) By Analytical Sequence



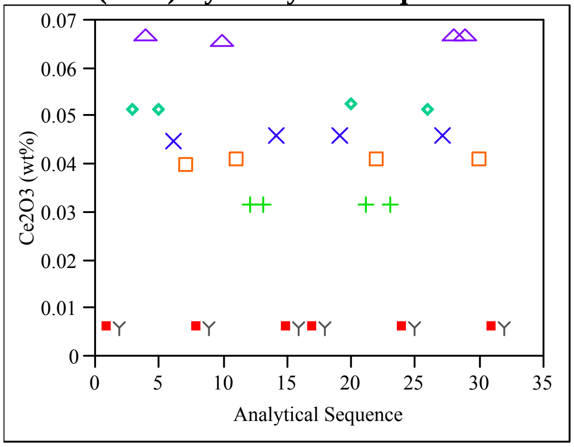
Cr2O3 (wt%) By Analytical Sequence



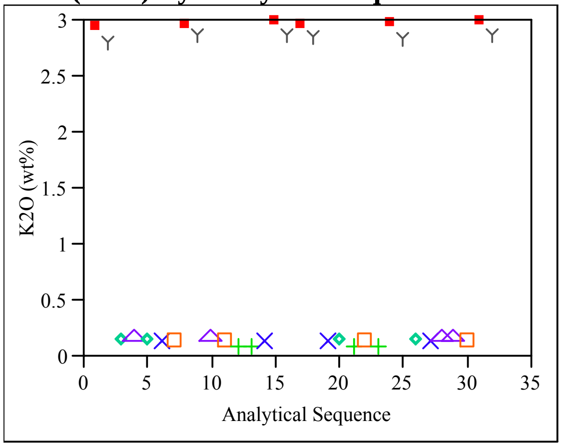
CaO (wt%) By Analytical Sequence



CuO (wt%) By Analytical Sequence



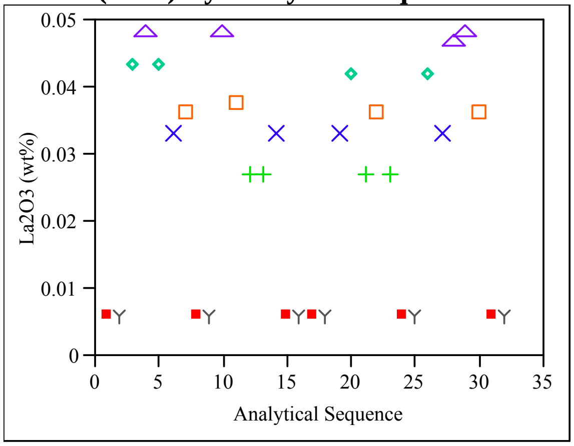
Ce2O3 (wt%) By Analytical Sequence



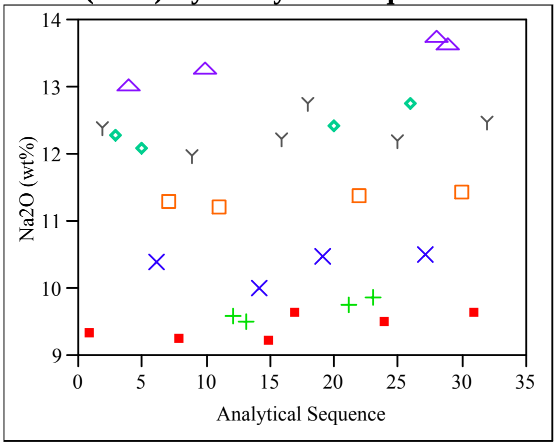
K2O (wt%) By Analytical Sequence

Exhibit D1. Oxide Measurements in Analytical Sequence for Samples Prepared Using the LM Method (continued)

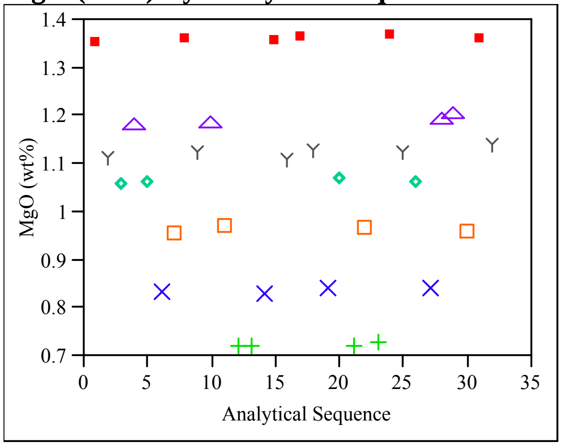
La2O3 (wt%) By Analytical Sequence



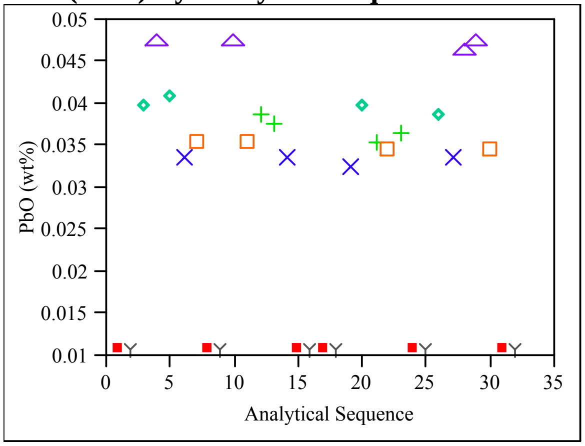
Na2O (wt%) By Analytical Sequence



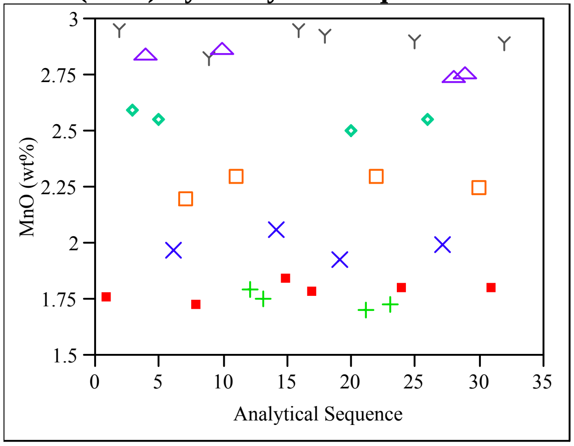
MgO (wt%) By Analytical Sequence



PbO (wt%) By Analytical Sequence



MnO (wt%) By Analytical Sequence



SO4 (wt%) By Analytical Sequence

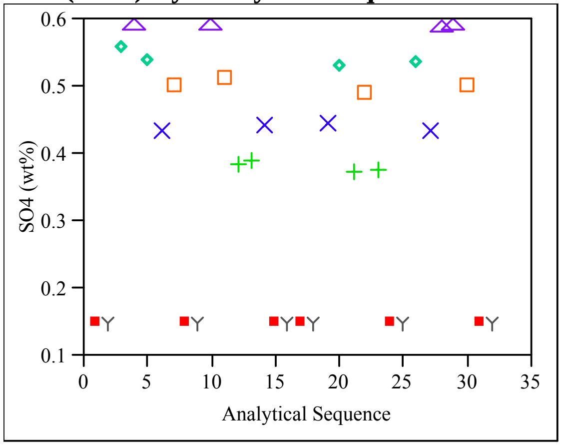
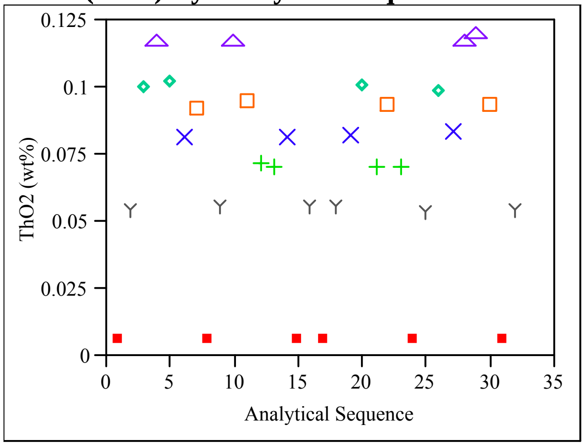
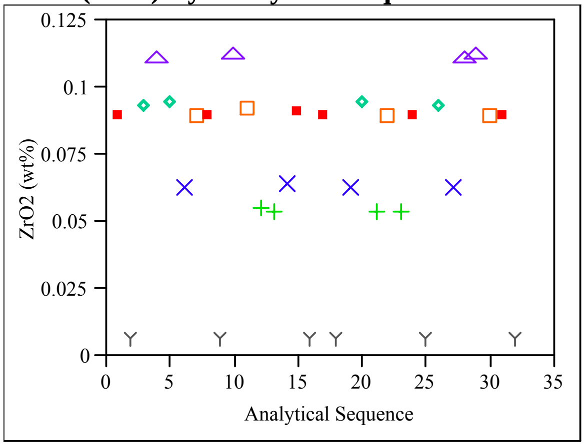


Exhibit D1. Oxide Measurements in Analytical Sequence for Samples Prepared Using the LM Method (continued)

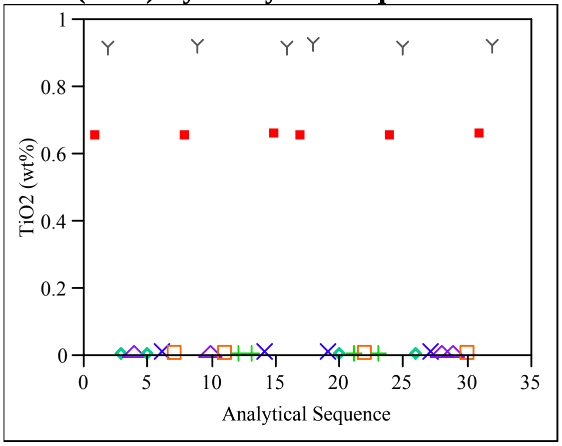
ThO2 (wt%) By Analytical Sequence



ZrO2 (wt%) By Analytical Sequence



TiO2 (wt%) By Analytical Sequence



ZnO (wt%) By Analytical Sequence

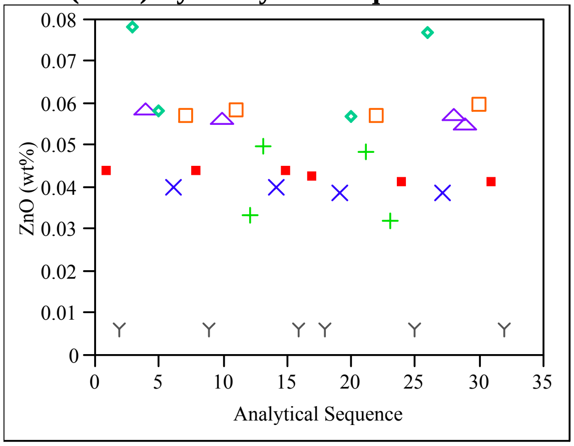
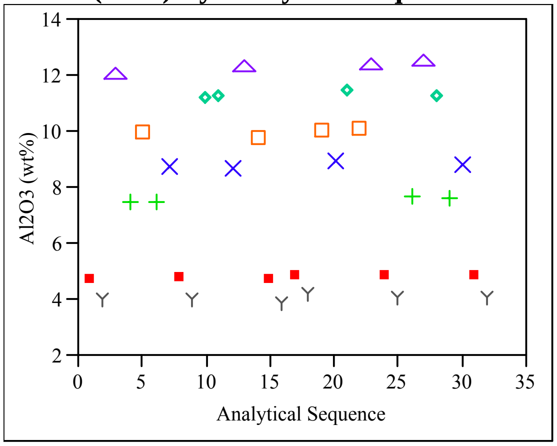
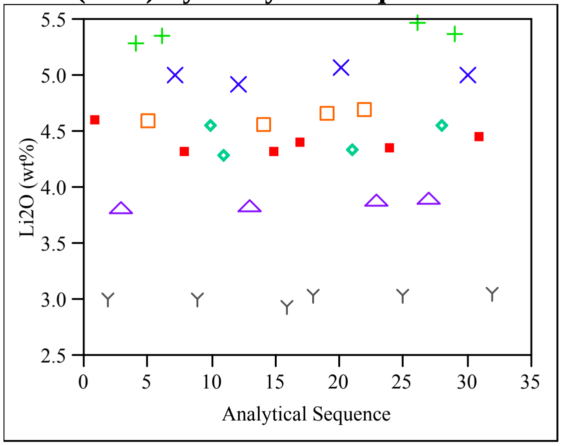


Exhibit D2. Oxide Measurements in Analytical Sequence for Samples Prepared Using the PF Method

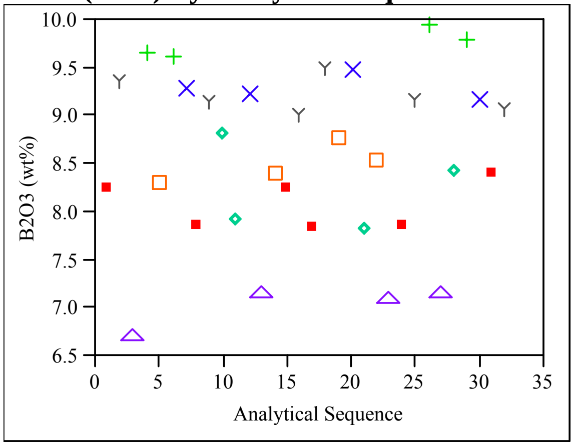
Al2O3 (wt%) By Analytical Sequence



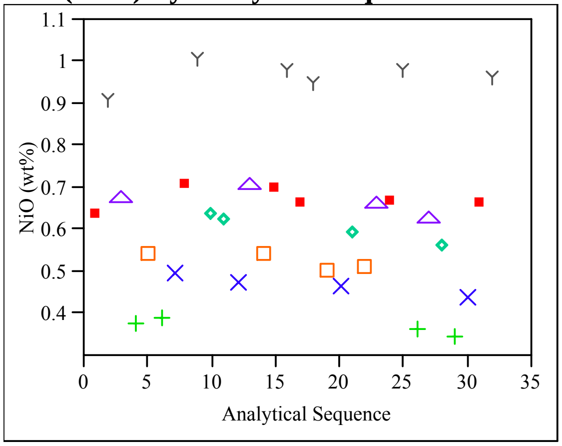
Li2O (wt%) By Analytical Sequence



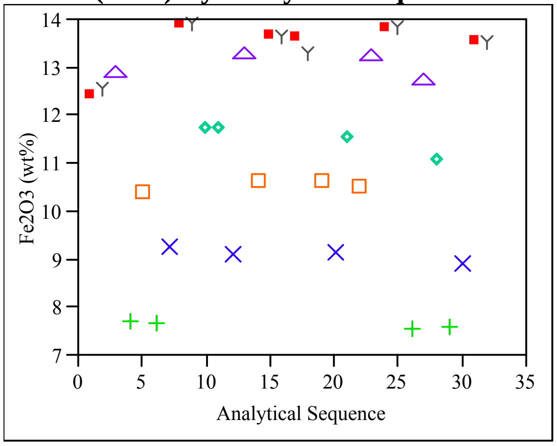
B2O3 (wt%) By Analytical Sequence



NiO (wt%) By Analytical Sequence



Fe2O3 (wt%) By Analytical Sequence



SiO2 (wt%) By Analytical Sequence

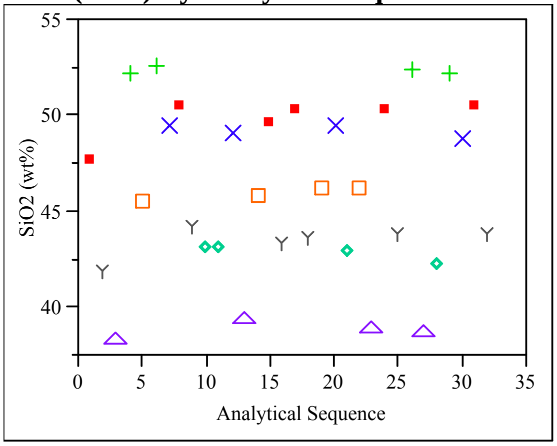


Exhibit D2. Oxide Measurements in Analytical Sequence for Samples Prepared Using the PF Method (continued)

U3O8 (wt%) By Analytical Sequence

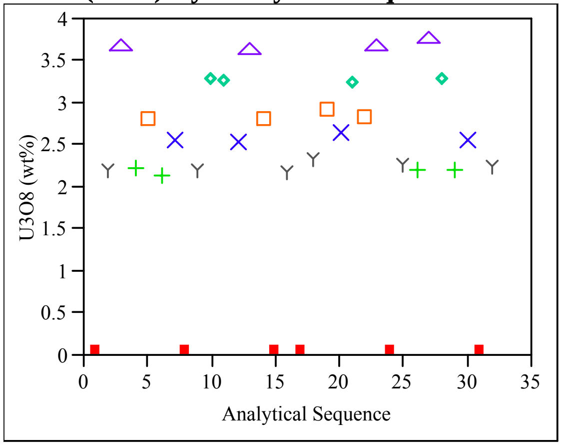
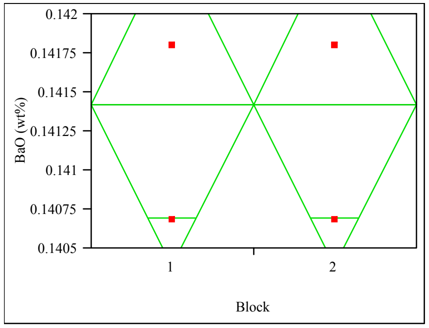


Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method

Glass ID=Batch 1
Oneway Analysis of BaO (wt%) By Block
Reference Value = 0.151 wt%



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	-0.25
Root Mean Square Error	0.000645
Mean of Response	0.141423
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0.00000	t Ratio	0
Std Err Dif	0.00053	DF	4
Upper CL Dif	0.00146	Prob > |t|	1.0000
Lower CL Dif	-0.00146	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

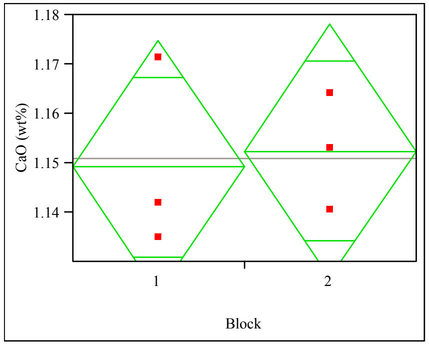
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	0.0000	1.0000
Error	4	0.00000166	4.1552e-7		
C. Total	5	0.00000166			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.141423	0.00037	0.14039	0.14246
2	3	0.141423	0.00037	0.14039	0.14246

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of CaO (wt%) By Block
Reference Value = 1.220 wt%



Oneway Anova
Summary of Fit

Rsquare	0.015308
Adj Rsquare	-0.23087
Root Mean Square Error	0.016035
Mean of Response	1.150842
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.00326	t Ratio	-0.24936
Std Err Dif	0.01309	DF	4
Upper CL Dif	0.03309	Prob > |t|	0.8154
Lower CL Dif	-0.03962	Prob > t	0.5923
Confidence	0.95	Prob < t	0.4077

Analysis of Variance

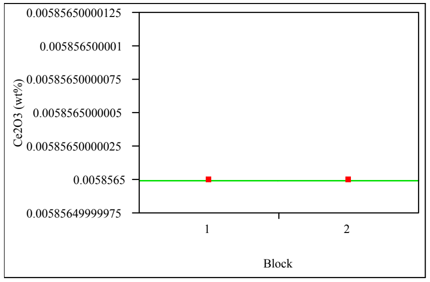
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00001599	0.000016	0.0622	0.8154
Error	4	0.00102848	0.000257		
C. Total	5	0.00104447			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	1.14921	0.00926	1.1235	1.1749
2	3	1.15247	0.00926	1.1268	1.1782

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of Ce2O3 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.005857
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

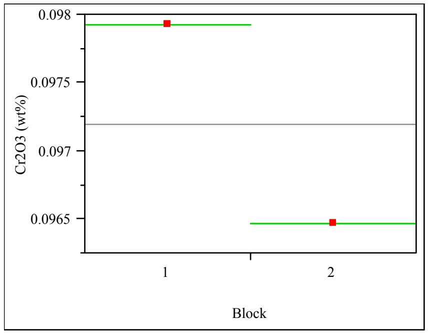
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.005857	0	0.00586	0.00586
2	3	0.005857	0	0.00586	0.00586

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Batch 1
Oneway Analysis of Cr2O3 (wt%) By Block
Reference Value = 0.107 wt%



Oneway Anova
Summary of Fit

Rsquare	1
Adj Rsquare	1
Root Mean Square Error	0
Mean of Response	0.097196
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0.001462	t Ratio	.
Std Err Dif	0.000000	DF	4
Upper CL Dif	0.001462	Prob > |t|	.
Lower CL Dif	0.001462	Prob > t	.
Confidence	0.95	Prob < t	.

Analysis of Variance

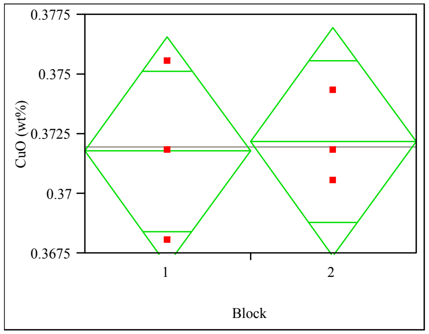
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.0000032	0.0000032	.	.
Error	4	0	0		
C. Total	5	0.0000032			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.097927	0	0.09793	0.09793
2	3	0.096466	0	0.09647	0.09647

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of CuO (wt%) By Block
Reference Value = 0.399 wt%



Oneway Anova
Summary of Fit

Rsquare	0.007299
Adj Rsquare	-0.24088
Root Mean Square Error	0.00298
Mean of Response	0.371993
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.00042	t Ratio	-0.1715
Std Err Dif	0.00243	DF	4
Upper CL Dif	0.00634	Prob > |t|	0.8722
Lower CL Dif	-0.00717	Prob > t	0.5639
Confidence	0.95	Prob < t	0.4361

Analysis of Variance

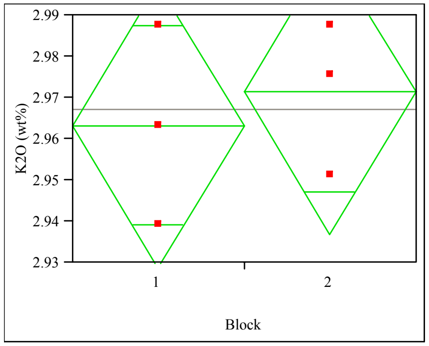
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00000026	2.6117e-7	0.0294	0.8722
Error	4	0.00003552	0.0000089		
C. Total	5	0.00003578			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.371785	0.00172	0.36701	0.37656
2	3	0.372202	0.00172	0.36743	0.37698

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of K2O (wt%) By Block
Reference Value = 3.327 wt%



Oneway Anova
Summary of Fit

Rsquare	0.05
Adj Rsquare	-0.1875
Root Mean Square Error	0.021436
Mean of Response	2.967331
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.00803	t Ratio	-0.45883
Std Err Dif	0.01750	DF	4
Upper CL Dif	0.04056	Prob > |t|	0.6702
Lower CL Dif	-0.05663	Prob > t	0.6649
Confidence	0.95	Prob < t	0.3351

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00009674	0.000097	0.2105	0.6702
Error	4	0.00183801	0.000460		
C. Total	5	0.00193475			

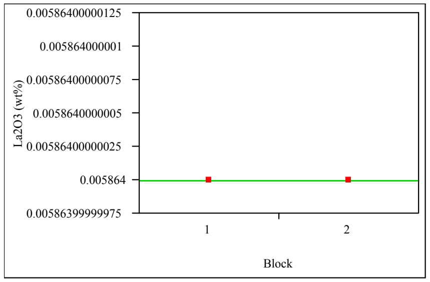
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	2.96332	0.01238	2.9290	2.9977
2	3	2.97135	0.01238	2.9370	3.0057

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Batch 1
Oneway Analysis of La2O3 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.005864
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

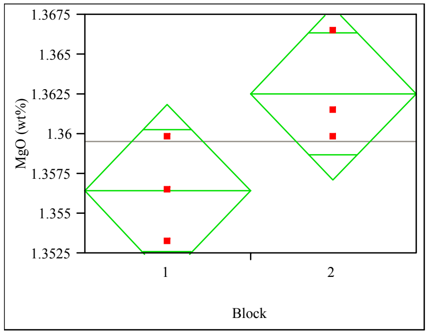
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.005864	0	0.00586	0.00586
2	3	0.005864	0	0.00586	0.00586

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of MgO (wt%) By Block
Reference Value = 1.419 wt%



Oneway Anova
Summary of Fit

Rsquare	0.547511
Adj Rsquare	0.434389
Root Mean Square Error	0.003385
Mean of Response	1.35953
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.00608	t Ratio	-2.2
Std Err Dif	0.00276	DF	4
Upper CL Dif	0.00159	Prob > |t|	0.0927
Lower CL Dif	-0.01375	Prob > t	0.9537
Confidence	0.95	Prob < t	0.0463

Analysis of Variance

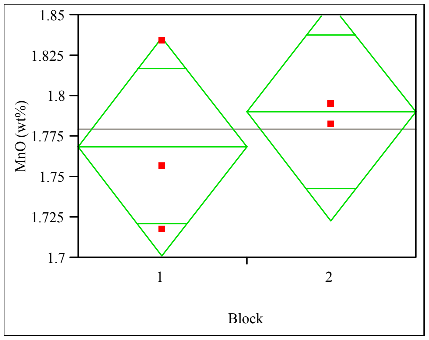
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00005546	0.000055	4.8400	0.0927
Error	4	0.00004583	0.000011		
C. Total	5	0.00010129			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	1.35649	0.00195	1.3511	1.3619
2	3	1.36257	0.00195	1.3571	1.3680

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of MnO (wt%) By Block
Reference Value = 1.726 wt%



Oneway Anova
Summary of Fit

Rsquare	0.088968
Adj Rsquare	-0.13879
Root Mean Square Error	0.04217
Mean of Response	1.779704
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.02152	t Ratio	-0.625
Std Err Dif	0.03443	DF	4
Upper CL Dif	0.07408	Prob > |t|	0.5659
Lower CL Dif	-0.11712	Prob > t	0.7171
Confidence	0.95	Prob < t	0.2829

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00069467	0.000695	0.3906	0.5659
Error	4	0.00711338	0.001778		
C. Total	5	0.00780804			

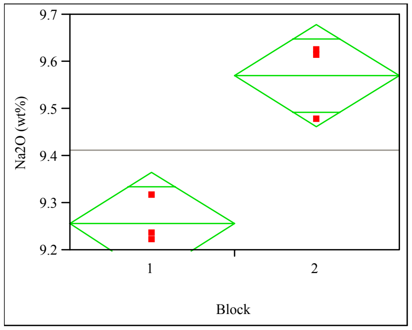
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	1.76894	0.02435	1.7013	1.8365
2	3	1.79046	0.02435	1.7229	1.8581

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Batch 1
Oneway Analysis of Na2O (wt%) By Block
Reference Value = 9.003 wt%



Oneway Anova
Summary of Fit

Rsquare	0.888325
Adj Rsquare	0.860406
Root Mean Square Error	0.068293
Mean of Response	9.413533
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.31453	t Ratio	-5.64076
Std Err Dif	0.05576	DF	4
Upper CL Dif	-0.15972	Prob > |t|	0.0049
Lower CL Dif	-0.46935	Prob > t	0.9976
Confidence	0.95	Prob < t	0.0024

Analysis of Variance

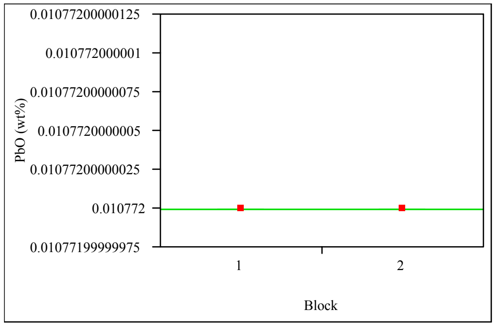
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.14839683	0.148397	31.8182	0.0049
Error	4	0.01865560	0.004664		
C. Total	5	0.16705243			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	9.25627	0.03943	9.1468	9.3657
2	3	9.57080	0.03943	9.4613	9.6803

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of PbO (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	-0.25
Root Mean Square Error	2.12e-18
Mean of Response	0.010772
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	0
Std Err Dif	1.735e-18	DF	4
Upper CL Dif	4.816e-18	Prob > |t|	1.0000
Lower CL Dif	-4.82e-18	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

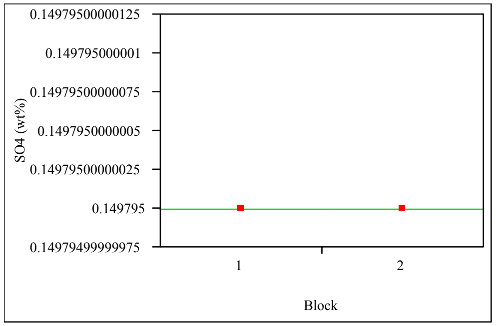
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	0.0000	1.0000
Error	4	1.8056e-35	4.514e-36		
C. Total	5	1.8056e-35			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.010772	1.227e-18	0.01077	0.01077
2	3	0.010772	1.227e-18	0.01077	0.01077

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of SO4 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.149795
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

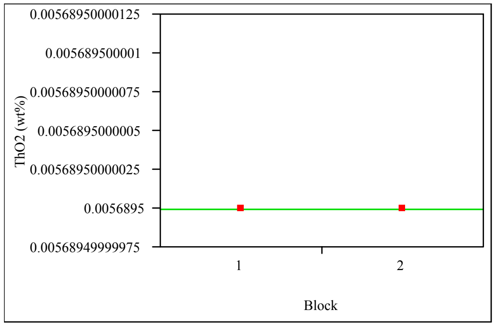
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.149795	0	0.14979	0.14979
2	3	0.149795	0	0.14979	0.14979

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Batch 1
Oneway Analysis of ThO2 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

```
Rsquare                              .
Adj Rsquare                          .
Root Mean Square Error               0
Mean of Response              0.005689
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference      0  t Ratio         .
Std Err Dif     0  DF              4
Upper CL Dif    0  Prob > |t|  1.0000
Lower CL Dif    0  Prob > t    0.5000
Confidence   0.95  Prob < t    0.5000
```

Analysis of Variance

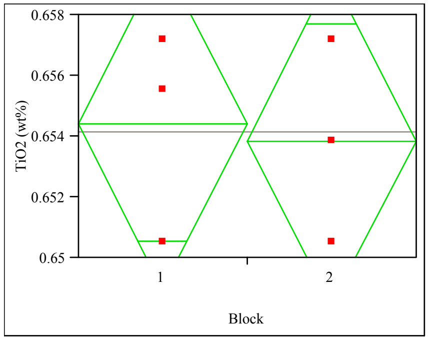
```
Source   DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block     1               0            0        .         .
Error     4               0            0
C. Total  5               0
```

Means for Oneway Anova

```
Level  Number      Mean  Std Error  Lower 95%  Upper 95%
1           3  0.005689          0    0.00569    0.00569
2           3  0.005689          0    0.00569    0.00569
```

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of TiO2 (wt%) By Block
Reference Value = 0.677 wt%



Oneway Anova
Summary of Fit

```
Rsquare                       0.009901
Adj Rsquare                   -0.23762
Root Mean Square Error        0.003405
Mean of Response              0.654134
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference     0.00056  t Ratio        0.2
Std Err Dif    0.00278  DF               4
Upper CL Dif   0.00827  Prob > |t|  0.8512
Lower CL Dif  -0.00716  Prob > t    0.4256
Confidence        0.95  Prob < t    0.5744
```

Analysis of Variance

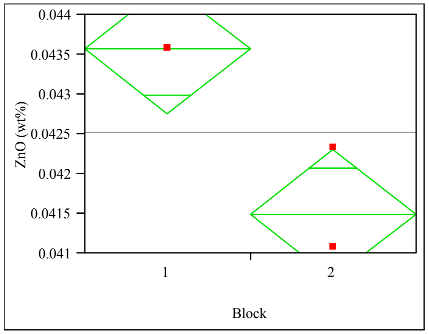
```
Source   DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block     1      0.00000046     4.637e-7   0.0400    0.8512
Error     4      0.00004637     0.000012
C. Total  5      0.00004683
```

Means for Oneway Anova

```
Level  Number      Mean  Std Error  Lower 95%  Upper 95%
1           3  0.654412    0.00197    0.64895    0.65987
2           3  0.653856    0.00197    0.64840    0.65931
```

Std Error uses a pooled estimate of error variance

Glass ID=Batch 1
Oneway Analysis of ZnO (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

```
Rsquare                       0.862069
Adj Rsquare                   0.827586
Root Mean Square Error        0.000508
Mean of Response              0.042531
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference    0.002075  t Ratio          5
Std Err Dif   0.000415  DF               4
Upper CL Dif  0.003227  Prob > |t|  0.0075
Lower CL Dif  0.000923  Prob > t    0.0037
Confidence        0.95  Prob < t    0.9963
```

Analysis of Variance

```
Source   DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block     1      0.00000646    0.0000065  25.0000    0.0075
Error     4      0.00000103    2.5825e-7
C. Total  5      0.00000749
```

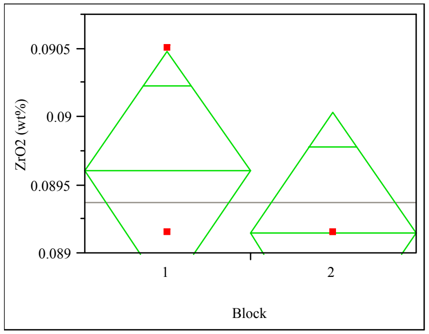
Means for Oneway Anova

```
Level  Number      Mean  Std Error  Lower 95%  Upper 95%
1           3  0.043568    0.00029    0.04275    0.04438
2           3  0.041493    0.00029    0.04068    0.04231
```

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Batch 1
Oneway Analysis of ZrO2 (wt%) By Block
Reference Value = 0.098 wt%



Oneway Anova
Summary of Fit

Rsquare	0.2
Adj Rsquare	2.07e-14
Root Mean Square Error	0.000551
Mean of Response	0.089378
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0.00045	t Ratio	1
Std Err Dif	0.00045	DF	4
Upper CL Dif	0.00170	Prob > |t|	0.3739
Lower CL Dif	-0.00080	Prob > t	0.1870
Confidence	0.95	Prob < t	0.8130

Analysis of Variance

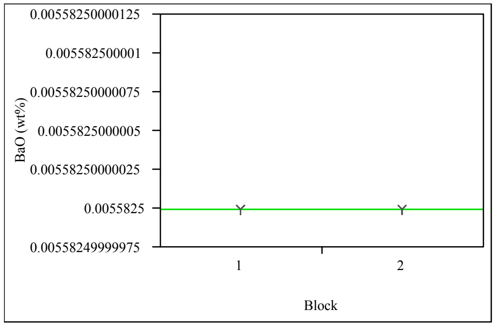
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	3.0411e-7	3.0411e-7	1.0000	0.3739
Error	4	0.00000122	3.0411e-7		
C. Total	5	0.00000152			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.089603	0.00032	0.08872	0.09049
2	3	0.089153	0.00032	0.08827	0.09004

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of BaO (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.005583
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

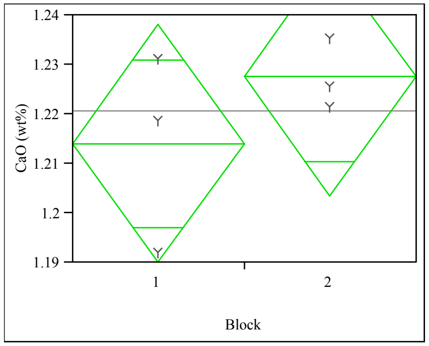
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.005583	0	0.00558	0.00558
2	3	0.005583	0	0.00558	0.00558

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of CaO (wt%) By Block
Reference Value = 1.301 wt%



Oneway Anova
Summary of Fit

Rsquare	0.233029
Adj Rsquare	0.041286
Root Mean Square Error	0.015026
Mean of Response	1.220802
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.01353	t Ratio	-1.10241
Std Err Dif	0.01227	DF	4
Upper CL Dif	0.02054	Prob > |t|	0.3321
Lower CL Dif	-0.04759	Prob > t	0.8339
Confidence	0.95	Prob < t	0.1661

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00027441	0.000274	1.2153	0.3321
Error	4	0.00090318	0.000226		
C. Total	5	0.00117759			

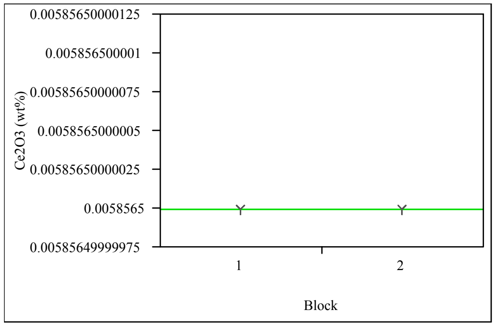
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	1.21404	0.00868	1.1900	1.2381
2	3	1.22756	0.00868	1.2035	1.2517

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Ustd
Oneway Analysis of Ce2O3 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.005857
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

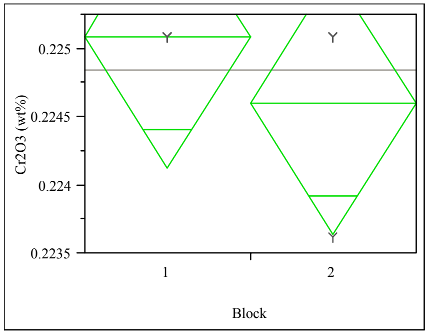
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.005857	0	0.00586	0.00586
2	3	0.005857	0	0.00586	0.00586

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of Cr2O3 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	0.2
Adj Rsquare	-9.5e-15
Root Mean Square Error	0.000597
Mean of Response	0.224843
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0.00049	t Ratio	1
Std Err Dif	0.00049	DF	4
Upper CL Dif	0.00184	Prob > |t|	0.3739
Lower CL Dif	-0.00087	Prob > t	0.1870
Confidence	0.95	Prob < t	0.8130

Analysis of Variance

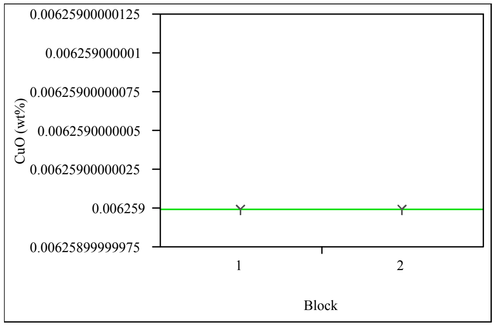
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	3.56046e-7	3.5605e-7	1.0000	0.3739
Error	4	0.00000142	3.5605e-7		
C. Total	5	0.00000178			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.225086	0.00034	0.22413	0.22604
2	3	0.224599	0.00034	0.22364	0.22556

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of CuO (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.006259
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

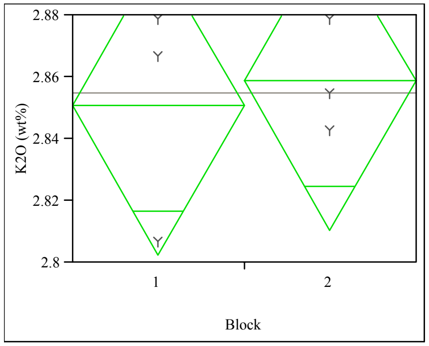
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.006259	0	0.00626	0.00626
2	3	0.006259	0	0.00626	0.00626

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Ustd
Oneway Analysis of K2O (wt%) By Block
Reference Value = 2.999 wt%



Oneway Anova
Summary of Fit

Rsquare	0.025641
Adj Rsquare	-0.21795
Root Mean Square Error	0.030315
Mean of Response	2.854902
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.00803	t Ratio	-0.32444
Std Err Dif	0.02475	DF	4
Upper CL Dif	0.06069	Prob > |t|	0.7619
Lower CL Dif	-0.07675	Prob > t	0.6191
Confidence	0.95	Prob < t	0.3809

Analysis of Variance

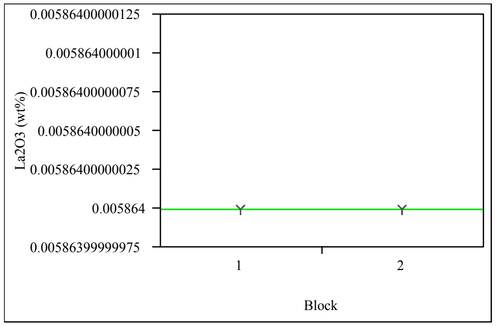
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00009674	0.000097	0.1053	0.7619
Error	4	0.00367602	0.000919		
C. Total	5	0.00377276			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	2.85089	0.01750	2.8023	2.8995
2	3	2.85892	0.01750	2.8103	2.9075

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of La2O3 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.005864
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

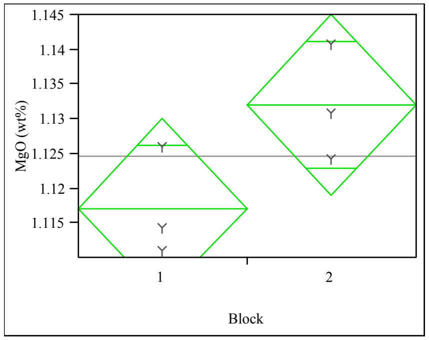
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.005864	0	0.00586	0.00586
2	3	0.005864	0	0.00586	0.00586

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of MgO (wt%) By Block
Reference Value = 1.21 wt%



Oneway Anova
Summary of Fit

Rsquare	0.560338
Adj Rsquare	0.450423
Root Mean Square Error	0.008096
Mean of Response	1.124604
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.01492	t Ratio	-2.25785
Std Err Dif	0.00661	DF	4
Upper CL Dif	0.00343	Prob > |t|	0.0869
Lower CL Dif	-0.03328	Prob > t	0.9566
Confidence	0.95	Prob < t	0.0434

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00033412	0.000334	5.0979	0.0869
Error	4	0.00026216	0.000066		
C. Total	5	0.00059628			

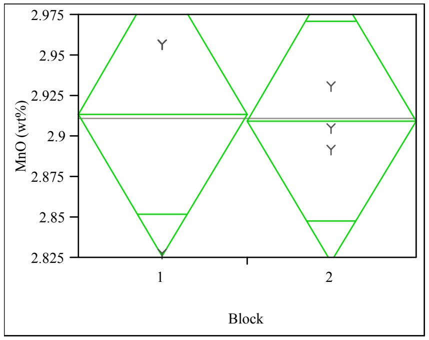
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	1.11714	0.00467	1.1042	1.1301
2	3	1.13207	0.00467	1.1191	1.1450

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Ustd
Oneway Analysis of MnO (wt%) By Block
Reference Value =2.892 wt%



Oneway Anova
Summary of Fit

Rsquare	0.002331
Adj Rsquare	-0.24709
Root Mean Square Error	0.054527
Mean of Response	2.911656
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0.00430	t Ratio	0.096674
Std Err Dif	0.04452	DF	4
Upper CL Dif	0.12791	Prob > |t|	0.9276
Lower CL Dif	-0.11931	Prob > t	0.4638
Confidence	0.95	Prob < t	0.5362

Analysis of Variance

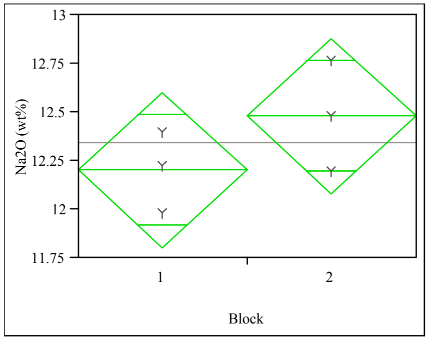
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00002779	0.000028	0.0093	0.9276
Error	4	0.01189268	0.002973		
C. Total	5	0.01192046			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	2.91381	0.03148	2.8264	3.0012
2	3	2.90950	0.03148	2.8221	2.9969

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of Na2O (wt%) By Block
Reference Value = 11.795 wt%



Oneway Anova
Summary of Fit

Rsquare	0.319163
Adj Rsquare	0.148954
Root Mean Square Error	0.249167
Mean of Response	12.34319
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.27859	t Ratio	-1.36935
Std Err Dif	0.20344	DF	4
Upper CL Dif	0.28627	Prob > |t|	0.2427
Lower CL Dif	-0.84344	Prob > t	0.8786
Confidence	0.95	Prob < t	0.1214

Analysis of Variance

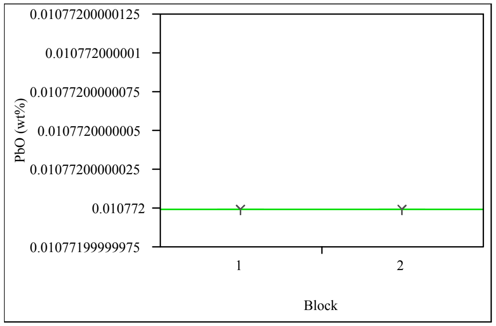
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.11641580	0.116416	1.8751	0.2427
Error	4	0.24833755	0.062084		
C. Total	5	0.36475334			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	12.2039	0.14386	11.804	12.603
2	3	12.4825	0.14386	12.083	12.882

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of PbO (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	-0.25
Root Mean Square Error	2.12e-18
Mean of Response	0.010772
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	0
Std Err Dif	1.735e-18	DF	4
Upper CL Dif	4.816e-18	Prob > |t|	1.0000
Lower CL Dif	-4.82e-18	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	0.0000	1.0000
Error	4	1.8056e-35	4.514e-36		
C. Total	5	1.8056e-35			

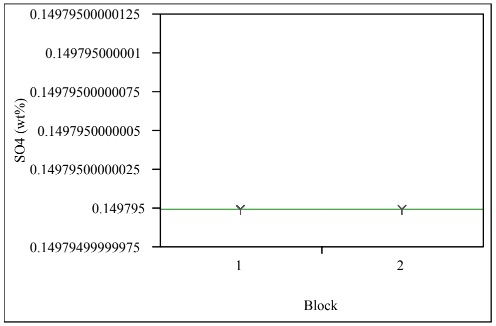
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.010772	1.227e-18	0.01077	0.01077
2	3	0.010772	1.227e-18	0.01077	0.01077

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Ustd
Oneway Analysis of SO4 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.149795
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

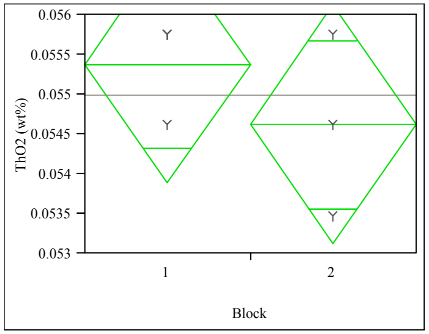
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.149795	0	0.14979	0.14979
2	3	0.149795	0	0.14979	0.14979

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of ThO2 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	0.2
Adj Rsquare	-1.1e-15
Root Mean Square Error	0.000929
Mean of Response	0.054998
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0.00076	t Ratio	1
Std Err Dif	0.00076	DF	4
Upper CL Dif	0.00286	Prob > |t|	0.3739
Lower CL Dif	-0.00135	Prob > t	0.1870
Confidence	0.95	Prob < t	0.8130

Analysis of Variance

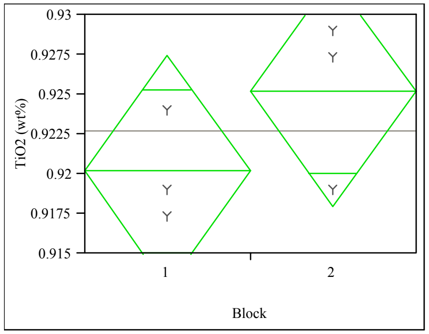
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	8.63211e-7	8.6321e-7	1.0000	0.3739
Error	4	0.00000345	8.6321e-7		
C. Total	5	0.00000432			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.055378	0.00054	0.05389	0.05687
2	3	0.054619	0.00054	0.05313	0.05611

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of TiO2 (wt%) By Block
Reference Value = 1.049 wt%



Oneway Anova
Summary of Fit

Rsquare	0.315175
Adj Rsquare	0.143969
Root Mean Square Error	0.004517
Mean of Response	0.922682
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.00500	t Ratio	-1.3568
Std Err Dif	0.00369	DF	4
Upper CL Dif	0.00524	Prob > |t|	0.2464
Lower CL Dif	-0.01524	Prob > t	0.8768
Confidence	0.95	Prob < t	0.1232

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00003756	0.000038	1.8409	0.2464
Error	4	0.00008161	0.000020		
C. Total	5	0.00011917			

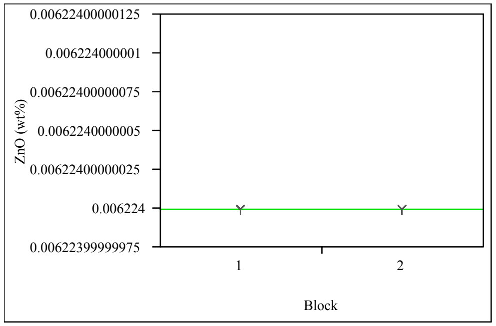
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.920180	0.00261	0.91294	0.92742
2	3	0.925184	0.00261	0.91794	0.93242

Std Error uses a pooled estimate of error variance

Exhibit D3. PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the LM Method (continued)

Glass ID=Ustd
Oneway Analysis of ZnO (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

```
Rsquare                                  .
Adj Rsquare                              .
Root Mean Square Error                   0
Mean of Response                  0.006224
Observations (or Sum Wgts)               6
```

t Test
1-2
Assuming equal variances

```
Difference        0  t Ratio             .
Std Err Dif       0  DF                  4
Upper CL Dif      0  Prob > |t|   1.0000
Lower CL Dif      0  Prob > t     0.5000
Confidence     0.95  Prob < t     0.5000
```

Analysis of Variance

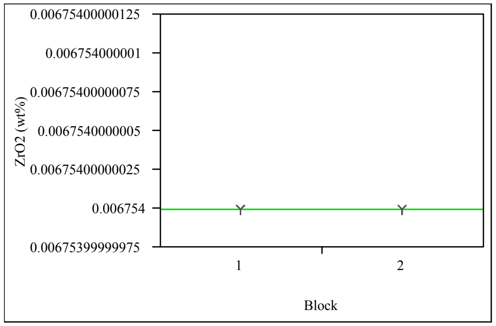
```
Source    DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block      1               0            0        .         .
Error      4               0            0
C. Total   5               0
```

Means for Oneway Anova

```
Level  Number      Mean  Std Error  Lower 95%  Upper 95%
1           3  0.006224          0    0.00622    0.00622
2           3  0.006224          0    0.00622    0.00622
```

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of ZrO2 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

```
Rsquare                                  .
Adj Rsquare                              .
Root Mean Square Error                   0
Mean of Response                  0.006754
Observations (or Sum Wgts)               6
```

t Test
1-2
Assuming equal variances

```
Difference        0  t Ratio             .
Std Err Dif       0  DF                  4
Upper CL Dif      0  Prob > |t|   1.0000
Lower CL Dif      0  Prob > t     0.5000
Confidence     0.95  Prob < t     0.5000
```

Analysis of Variance

```
Source    DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block      1               0            0        .         .
Error      4               0            0
C. Total   5               0
```

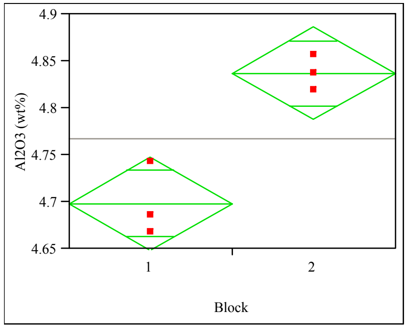
Means for Oneway Anova

```
Level  Number      Mean  Std Error  Lower 95%  Upper 95%
1           3  0.006754          0    0.00675    0.00675
2           3  0.006754          0    0.00675    0.00675
```

Std Error uses a pooled estimate of error variance

Exhibit D4: PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the PF Method

Glass ID=Batch 1
Oneway Analysis of Al2O3 (wt%) By Block
Reference Value = 4.877 wt%



Oneway Anova
Summary of Fit

```
Rsquare                       0.883212
Adj Rsquare                   0.854015
Root Mean Square Error        0.030855
Mean of Response              4.767838
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference     -0.13856  t Ratio        -5.5
Std Err Dif     0.02519  DF                4
Upper CL Dif   -0.06862  Prob > |t|   0.0053
Lower CL Dif   -0.20851  Prob > t     0.9973
Confidence         0.95  Prob < t     0.0027
```

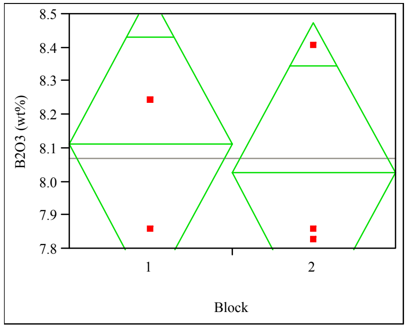
Analysis of Variance

```
Source   DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block     1      0.02879970     0.028800  30.2500    0.0053
Error     4      0.00380822     0.000952
C. Total  5      0.03260792
```

Means for Oneway Anova

```
Level  Number    Mean   Std Error  Lower 95%  Upper 95%
1           3  4.69856    0.01781     4.6491     4.7480
2           3  4.83712    0.01781     4.7877     4.8866
Std Error uses a pooled estimate of error variance
```

Glass ID=Batch 1
Oneway Analysis of B2O3 (wt%) By Block
Reference Value = 7.777 wt%



Oneway Anova
Summary of Fit

```
Rsquare                       0.034261
Adj Rsquare                   -0.20717
Root Mean Square Error        0.279161
Mean of Response              8.071216
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference      0.08586  t Ratio    0.376705
Std Err Dif     0.22793  DF                4
Upper CL Dif    0.71871  Prob > |t|   0.7255
Lower CL Dif   -0.54698  Prob > t     0.3628
Confidence         0.95  Prob < t     0.6372
```

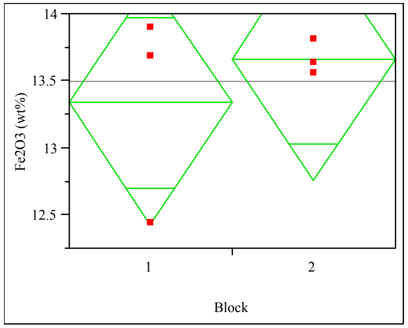
Analysis of Variance

```
Source   DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block     1      0.01105894     0.011059   0.1419    0.7255
Error     4      0.31172386     0.077931
C. Total  5      0.32278280
```

Means for Oneway Anova

```
Level  Number    Mean   Std Error  Lower 95%  Upper 95%
1           3  8.11415    0.16117     7.6667     8.5616
2           3  8.02828    0.16117     7.5808     8.4758
Std Error uses a pooled estimate of error variance
```

Glass ID=Batch 1
Oneway Analysis of Fe2O3 (wt%) By Block
Reference Value = 12.839 wt%



Oneway Anova
Summary of Fit

```
Rsquare                       0.112914
Adj Rsquare                   -0.10886
Root Mean Square Error        0.564415
Mean of Response              13.50352
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference      -0.3288  t Ratio    -0.71354
Std Err Dif      0.4608  DF                4
Upper CL Dif     0.9507  Prob > |t|   0.5149
Lower CL Dif    -1.6083  Prob > t     0.7425
Confidence         0.95  Prob < t     0.2575
```

Analysis of Variance

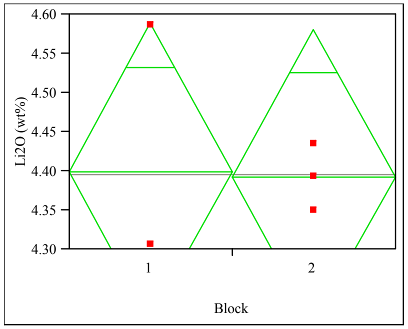
```
Source   DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block     1       0.1621947     0.162195   0.5091    0.5149
Error     4       1.2742558     0.318564
C. Total  5       1.4364506
```

Means for Oneway Anova

```
Level  Number    Mean   Std Error  Lower 95%  Upper 95%
1           3  13.3391    0.32586     12.434     14.244
2           3  13.6679    0.32586     12.763     14.573
Std Error uses a pooled estimate of error variance
```

Exhibit D4: PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the PF Method (continued)

Glass ID=Batch 1
Oneway Analysis of Li2O (wt%) By Block
Reference Value = 4.429 wt%



Oneway Anova
Summary of Fit

```
Rsquare                      0.001379
Adj Rsquare                  -0.24828
Root Mean Square Error       0.118246
Mean of Response             4.395504
Observations (or Sum Wgts)          6
```

t Test
1-2
Assuming equal variances

```
Difference      0.00718  t Ratio     0.074329
Std Err Dif     0.09655  DF                 4
Upper CL Dif    0.27524  Prob > |t|    0.9443
Lower CL Dif   -0.26088  Prob > t      0.4722
Confidence         0.95  Prob < t      0.5278
```

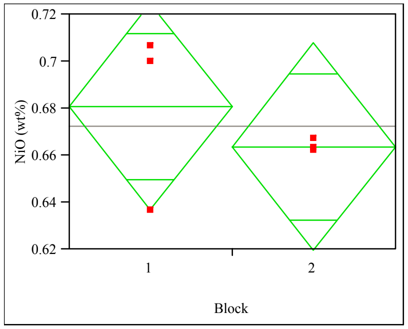
Analysis of Variance

```
Source    DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block      1      0.00007725     0.000077   0.0055    0.9443
Error      4      0.05592874     0.013982
C. Total   5      0.05600599
```

Means for Oneway Anova

```
Level  Number     Mean  Std Error  Lower 95%  Upper 95%
1           3  4.39909    0.06827     4.2095     4.5886
2           3  4.39192    0.06827     4.2024     4.5815
Std Error uses a pooled estimate of error variance
```

Glass ID=Batch 1
Oneway Analysis of NiO (wt%) By Block
Reference Value = 0.751 wt%



Oneway Anova
Summary of Fit

```
Rsquare                      0.125471
Adj Rsquare                  -0.09316
Root Mean Square Error        0.02743
Mean of Response             0.672304
Observations (or Sum Wgts)          6
```

t Test
1-2
Assuming equal variances

```
Difference      0.01697  t Ratio     0.757554
Std Err Dif     0.02240  DF                 4
Upper CL Dif    0.07915  Prob > |t|    0.4909
Lower CL Dif   -0.04522  Prob > t      0.2454
Confidence         0.95  Prob < t      0.7546
```

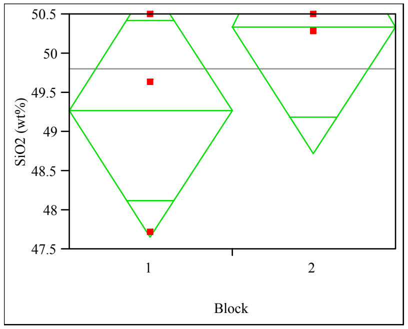
Analysis of Variance

```
Source    DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block      1      0.00043180     0.000432   0.5739    0.4909
Error      4      0.00300966     0.000752
C. Total   5      0.00344146
```

Means for Oneway Anova

```
Level  Number      Mean  Std Error  Lower 95%  Upper 95%
1           3  0.680788    0.01584    0.63682    0.72476
2           3  0.663821    0.01584    0.61985    0.70779
Std Error uses a pooled estimate of error variance
```

Glass ID=Batch 1
Oneway Analysis of SiO2 (wt%) By Block
Reference Value = 50.22 wt%



Oneway Anova
Summary of Fit

```
Rsquare                      0.295664
Adj Rsquare                   0.11958
Root Mean Square Error       1.010994
Mean of Response             49.81003
Observations (or Sum Wgts)          6
```

t Test
1-2
Assuming equal variances

```
Difference      -1.0697  t Ratio      -1.2958
Std Err Dif      0.8255  DF                 4
Upper CL Dif     1.2222  Prob > |t|    0.2648
Lower CL Dif    -3.3615  Prob > t      0.8676
Confidence         0.95  Prob < t      0.1324
```

Analysis of Variance

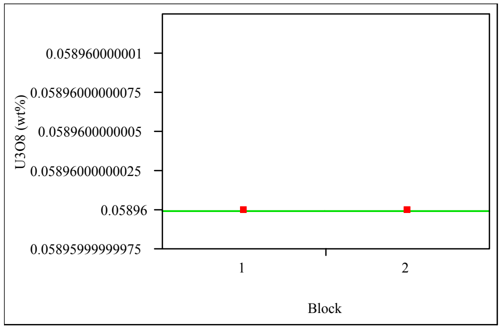
```
Source    DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block      1       1.7162267      1.71623   1.6791    0.2648
Error      4       4.0884333      1.02211
C. Total   5       5.8046600
```

Means for Oneway Anova

```
Level  Number     Mean  Std Error  Lower 95%  Upper 95%
1           3  49.2752    0.58370     47.655     50.896
2           3  50.3449    0.58370     48.724     51.965
Std Error uses a pooled estimate of error variance
```

Exhibit D4: PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the PF Method (continued)

Glass ID=Batch 1
Oneway Analysis of U3O8 (wt%) By Block
Reference Value = 0 wt%



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.05896
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	0	t Ratio	.
Std Err Dif	0	DF	4
Upper CL Dif	0	Prob > |t|	1.0000
Lower CL Dif	0	Prob > t	0.5000
Confidence	0.95	Prob < t	0.5000

Analysis of Variance

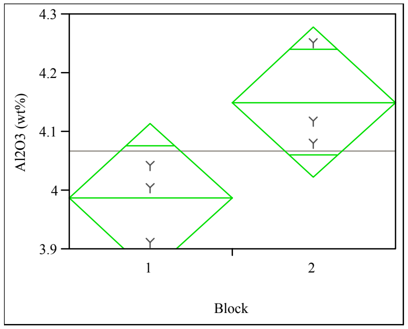
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0	0	.	.
Error	4	0	0		
C. Total	5	0			

Means for Oneway Anova

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

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of Al2O3 (wt%) By Block
Reference Value = 4.1 wt%



Oneway Anova
Summary of Fit

Rsquare	0.614545
Adj Rsquare	0.518182
Root Mean Square Error	0.079419
Mean of Response	4.068723
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.16376	t Ratio	-2.52534
Std Err Dif	0.06485	DF	4
Upper CL Dif	0.01628	Prob > |t|	0.0650
Lower CL Dif	-0.34380	Prob > t	0.9675
Confidence	0.95	Prob < t	0.0325

Analysis of Variance

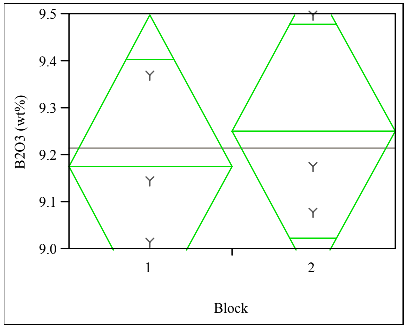
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.04022437	0.040224	6.3774	0.0650
Error	4	0.02522949	0.006307		
C. Total	5	0.06545385			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	3.98685	0.04585	3.8595	4.1142
2	3	4.15060	0.04585	4.0233	4.2779

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of B2O3 (wt%) By Block
Reference Value = 9.209 wt%



Oneway Anova
Summary of Fit

Rsquare	0.050154
Adj Rsquare	-0.18731
Root Mean Square Error	0.200222
Mean of Response	9.21428
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.07513	t Ratio	-0.45957
Std Err Dif	0.16348	DF	4
Upper CL Dif	0.37876	Prob > |t|	0.6697
Lower CL Dif	-0.52902	Prob > t	0.6652
Confidence	0.95	Prob < t	0.3348

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.00846700	0.008467	0.2112	0.6697
Error	4	0.16035463	0.040089		
C. Total	5	0.16882163			

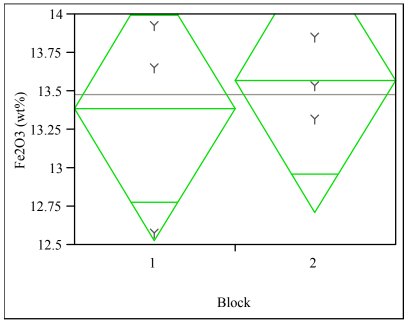
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	9.17671	0.11560	8.8558	9.4977
2	3	9.25185	0.11560	8.9309	9.5728

Std Error uses a pooled estimate of error variance

Exhibit D4: PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the PF Method (continued)

Glass ID=Ustd
Oneway Analysis of Fe2O3 (wt%) By Block
Reference Value =13.196 wt%



Oneway Anova
Summary of Fit

```
Rsquare                       0.043062
Adj Rsquare                   -0.19617
Root Mean Square Error        0.536534
Mean of Response              13.47969
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference    -0.1859  t Ratio    -0.42426
Std Err Dif    0.4381  DF                4
Upper CL Dif   1.0304  Prob > |t|   0.6932
Lower CL Dif  -1.4022  Prob > t     0.6534
Confidence       0.95  Prob < t     0.3466
```

Analysis of Variance

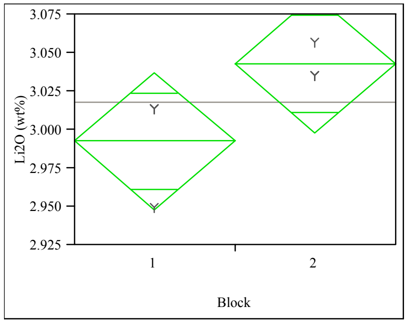
```
Source    DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block      1       0.0518165     0.051816   0.1800    0.6932
Error      4       1.1514770     0.287869
C. Total   5       1.2032935
```

Means for Oneway Anova

```
Level  Number     Mean  Std Error  Lower 95%  Upper 95%
1           3  13.3868    0.30977     12.527     14.247
2           3  13.5726    0.30977     12.713     14.433
```

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of Li2O (wt%) By Block
Reference Value = 3.057 wt%



Oneway Anova
Summary of Fit

```
Rsquare                       0.550562
Adj Rsquare                   0.438202
Root Mean Square Error        0.027794
Mean of Response              3.017648
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference    -0.05023  t Ratio    -2.21359
Std Err Dif    0.02269  DF                4
Upper CL Dif   0.01277  Prob > |t|   0.0913
Lower CL Dif  -0.11324  Prob > t     0.9544
Confidence        0.95  Prob < t     0.0456
```

Analysis of Variance

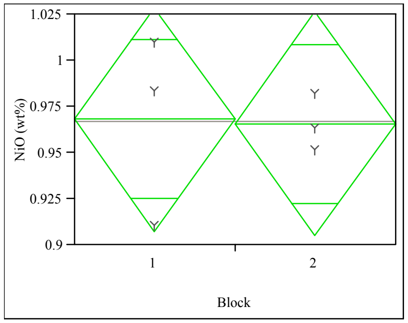
```
Source    DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block      1      0.00378523     0.003785   4.9000    0.0913
Error      4      0.00308999     0.000772
C. Total   5      0.00687522
```

Means for Oneway Anova

```
Level  Number     Mean  Std Error  Lower 95%  Upper 95%
1           3  2.99253    0.01605     2.9480     3.0371
2           3  3.04277    0.01605     2.9982     3.0873
```

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of NiO (wt%) By Block
Reference Value = 1.12 wt%



Oneway Anova
Summary of Fit

```
Rsquare                       0.001686
Adj Rsquare                   -0.24789
Root Mean Square Error         0.03792
Mean of Response                0.9671
Observations (or Sum Wgts)           6
```

t Test
1-2
Assuming equal variances

```
Difference     0.00255  t Ratio    0.082199
Std Err Dif    0.03096  DF                4
Upper CL Dif   0.08851  Prob > |t|   0.9384
Lower CL Dif  -0.08342  Prob > t     0.4692
Confidence        0.95  Prob < t     0.5308
```

Analysis of Variance

```
Source    DF  Sum of Squares  Mean Square  F Ratio  Prob > F
Block      1      0.00000972      0.00001   0.0068    0.9384
Error      4      0.00575160     0.001438
C. Total   5      0.00576131
```

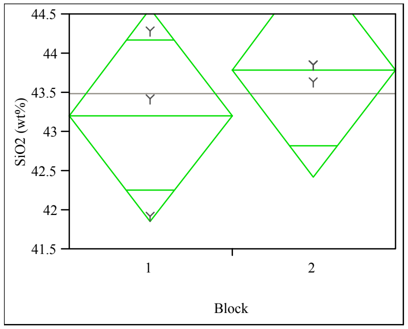
Means for Oneway Anova

```
Level  Number      Mean  Std Error  Lower 95%  Upper 95%
1           3  0.968373    0.02189    0.90759     1.0292
2           3  0.965827    0.02189    0.90504     1.0266
```

Std Error uses a pooled estimate of error variance

Exhibit D4: PSAL Measurements by Analytical Block for Samples of the Standard Glasses Prepared Using the PF Method (continued)

Glass ID=Ustd
Oneway Analysis of SiO2 (wt%) By Block
Reference Value = 45.353 wt%



Oneway Anova
Summary of Fit

Rsquare	0.145455
Adj Rsquare	-0.06818
Root Mean Square Error	0.846759
Mean of Response	43.4991
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.5705	t Ratio	-0.82514
Std Err Dif	0.6914	DF	4
Upper CL Dif	1.3491	Prob > |t|	0.4557
Lower CL Dif	-2.4900	Prob > t	0.7722
Confidence	0.95	Prob < t	0.2278

Analysis of Variance

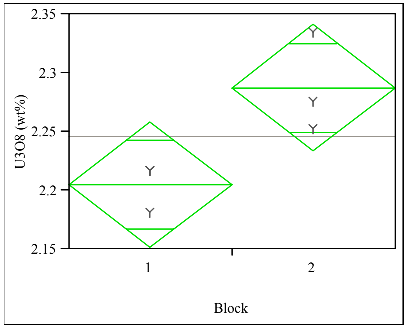
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.4881711	0.488171	0.6809	0.4557
Error	4	2.8680055	0.717001		
C. Total	5	3.3561766			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	43.2139	0.48888	41.857	44.571
2	3	43.7843	0.48888	42.427	45.142

Std Error uses a pooled estimate of error variance

Glass ID=Ustd
Oneway Analysis of U3O8 (wt%) By Block
Reference Value = 2.406 wt%



Oneway Anova
Summary of Fit

Rsquare	0.696682
Adj Rsquare	0.620853
Root Mean Square Error	0.033353
Mean of Response	2.246376
Observations (or Sum Wgts)	6

t Test
1-2
Assuming equal variances

Difference	-0.08254	t Ratio	-3.03109
Std Err Dif	0.02723	DF	4
Upper CL Dif	-0.00693	Prob > |t|	0.0387
Lower CL Dif	-0.15815	Prob > t	0.9806
Confidence	0.95	Prob < t	0.0194

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	1	0.01022027	0.010220	9.1875	0.0387
Error	4	0.00444964	0.001112		
C. Total	5	0.01466991			

Means for Oneway Anova

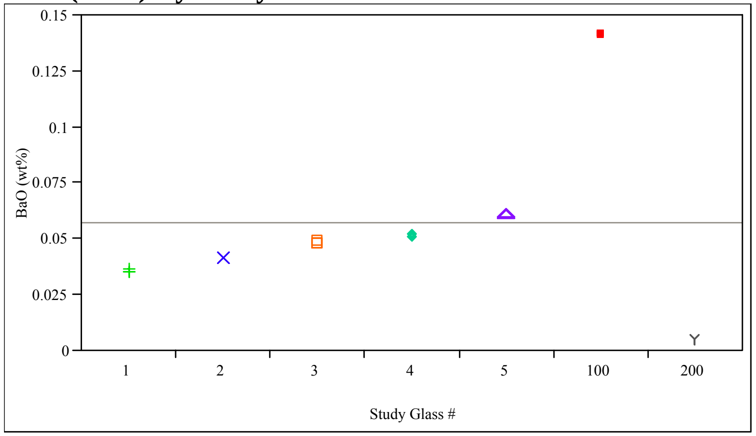
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	2.20510	0.01926	2.1516	2.2586
2	3	2.28765	0.01926	2.2342	2.3411

Std Error uses a pooled estimate of error variance

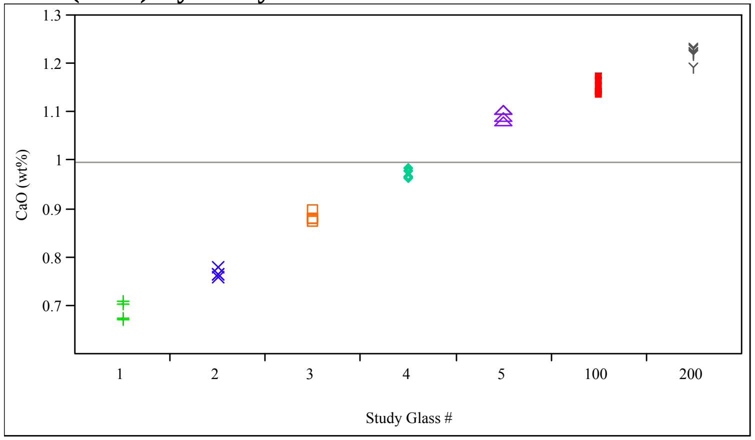
Exhibit D5. Measured and Measured Bias-Corrected Oxide Weight Percents by Glass # for the Glasses Prepared Using the LM Method

(100 – Batch 1; 200 – Ustd)

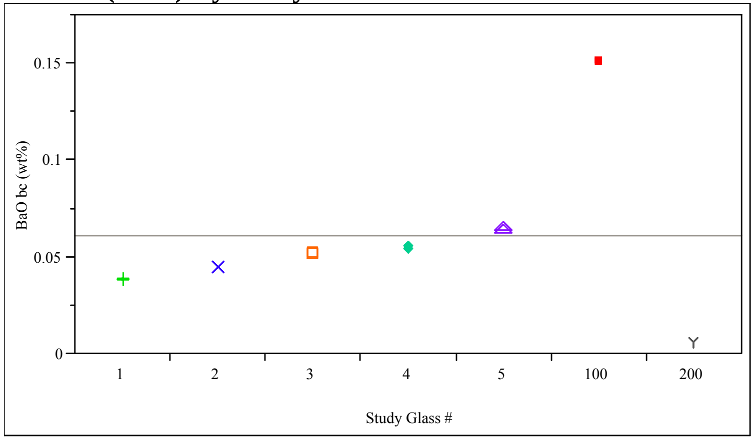
BaO (wt%) By Study Glass #



CaO (wt%) By Study Glass #



BaO bc (wt%) By Study Glass #



CaO bc (wt%) By Study Glass #

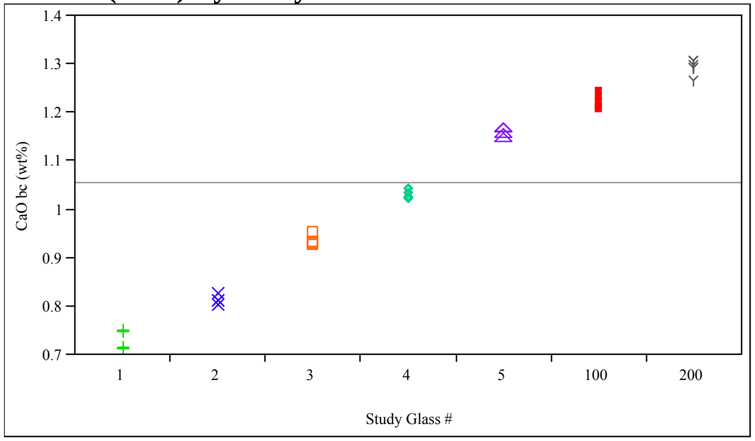
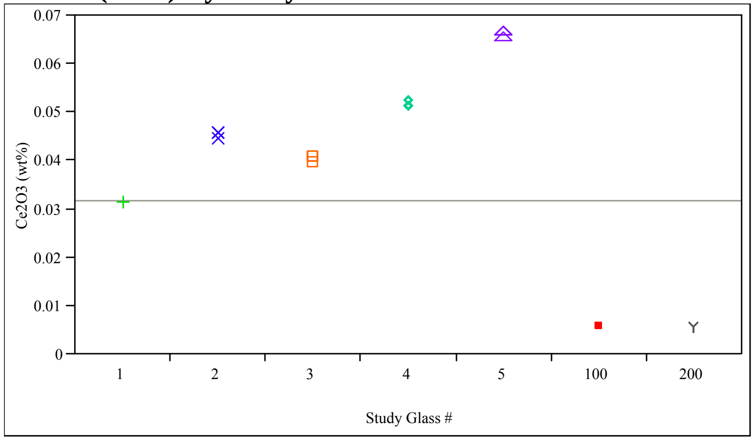


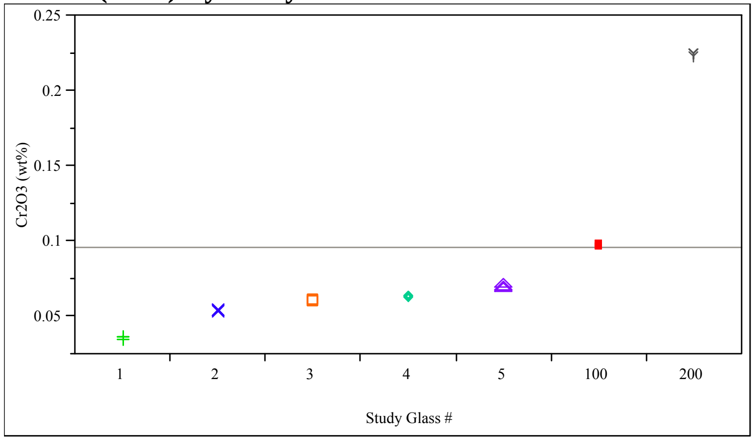
Exhibit D5. Measured and Measured Bias-Corrected Oxide Weight Percents by Glass # for the Glasses Prepared Using the LM Method (continued)

(100 – Batch 1; 200 – Ustd)

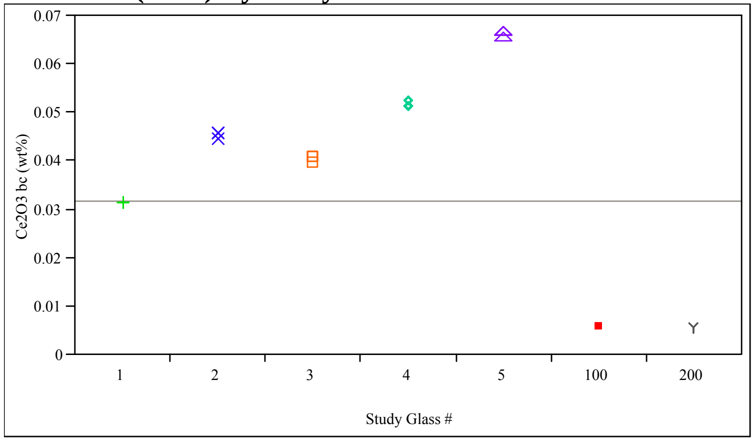
Ce2O3 (wt%) By Study Glass #



Cr2O3 (wt%) By Study Glass #



Ce2O3 bc (wt%) By Study Glass #



Cr2O3 bc (wt%) By Study Glass #

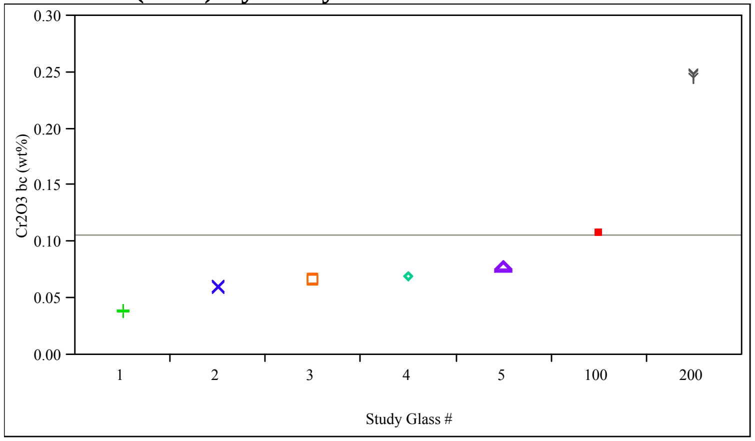
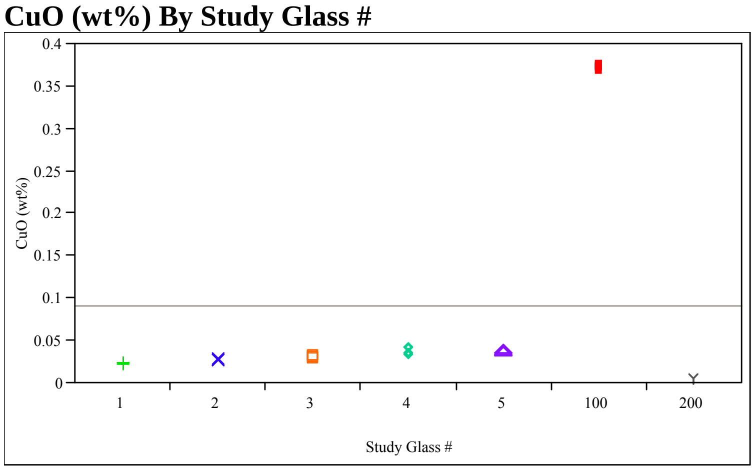
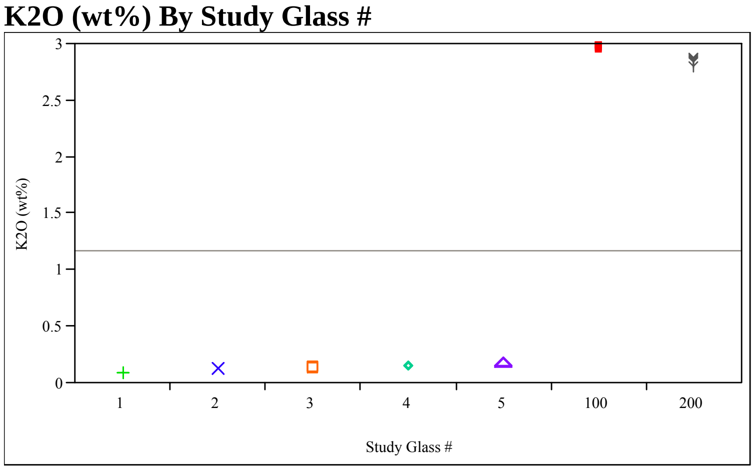
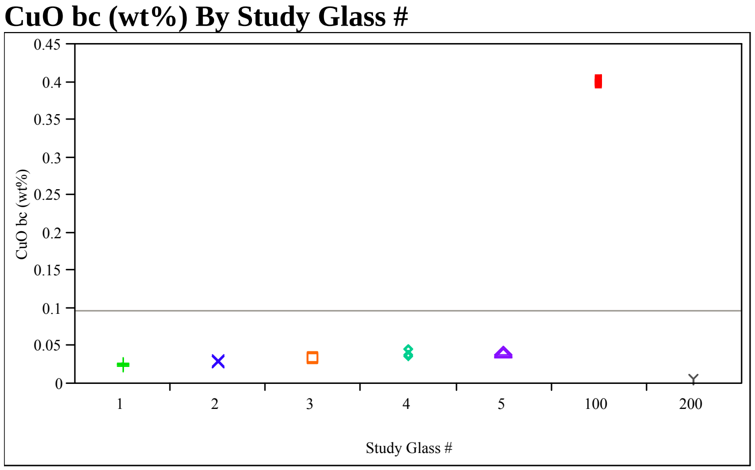


Exhibit D5. Measured and Measured Bias-Corrected Oxide Weight Percents by Glass # for the Glasses Prepared Using the LM Method (continued)

(100 – Batch 1; 200 – Ustd)







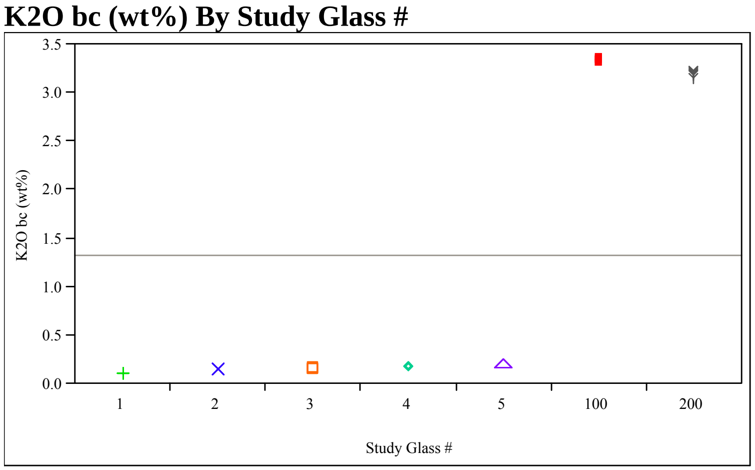
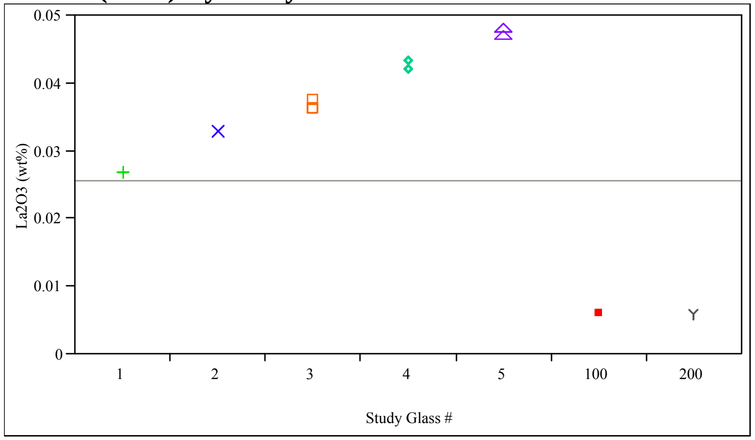


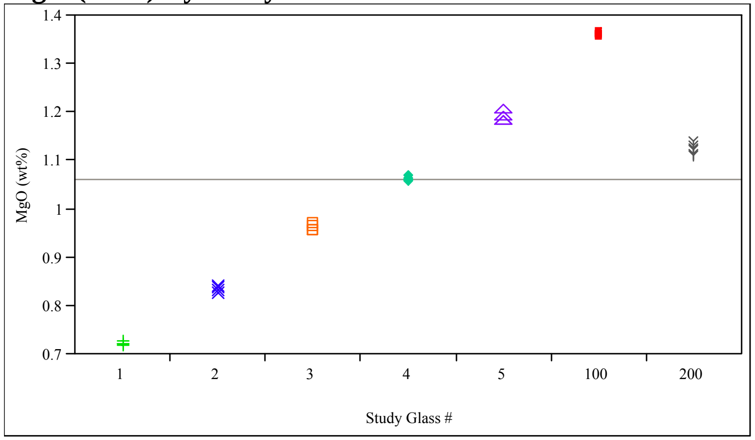
Exhibit D5. Measured and Measured Bias-Corrected Oxide Weight Percents by Glass # for the Glasses Prepared Using the LM Method (continued)

(100 – Batch 1; 200 – Ustd)

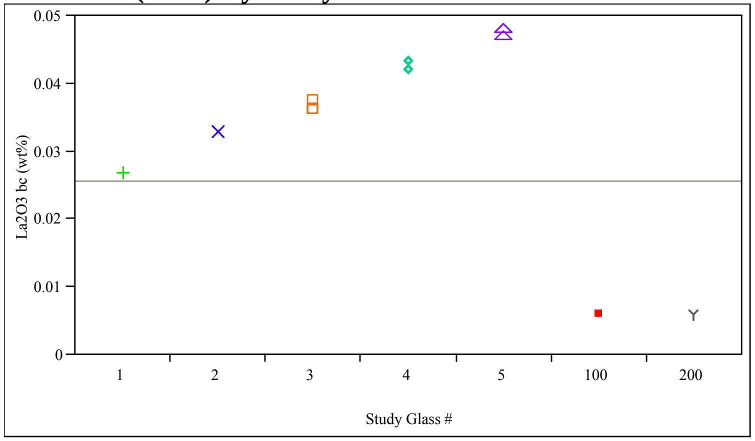
La2O3 (wt%) By Study Glass #



MgO (wt%) By Study Glass #



La2O3 bc (wt%) By Study Glass #



MgO bc (wt%) By Study Glass #

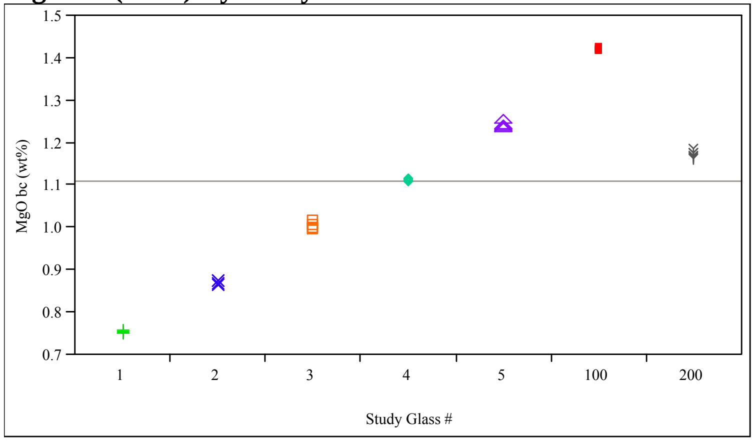
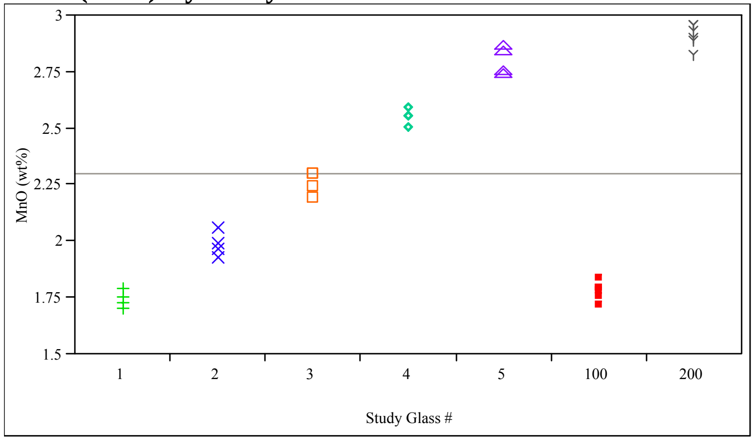


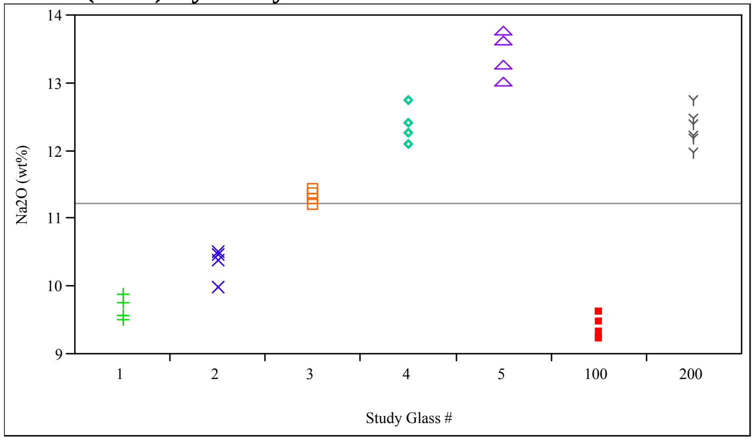
Exhibit D5. Measured and Measured Bias-Corrected Oxide Weight Percents by Glass # for the Glasses Prepared Using the LM Method (continued)

(100 – Batch 1; 200 – Ustd)

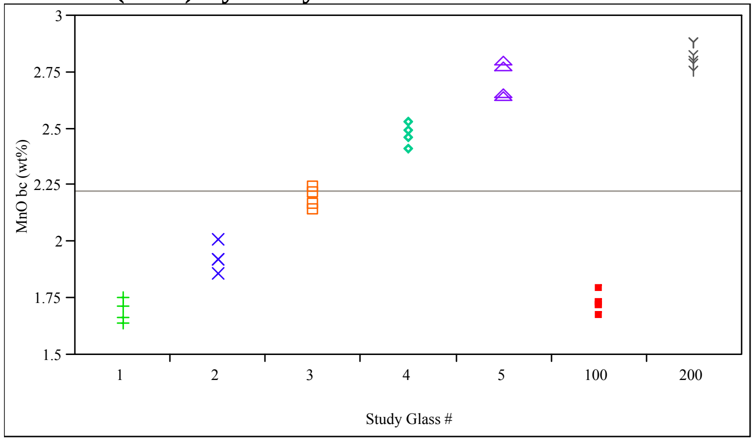
MnO (wt%) By Study Glass #



Na2O (wt%) By Study Glass #



MnO bc (wt%) By Study Glass #



Na2O bc (wt%) By Study Glass #

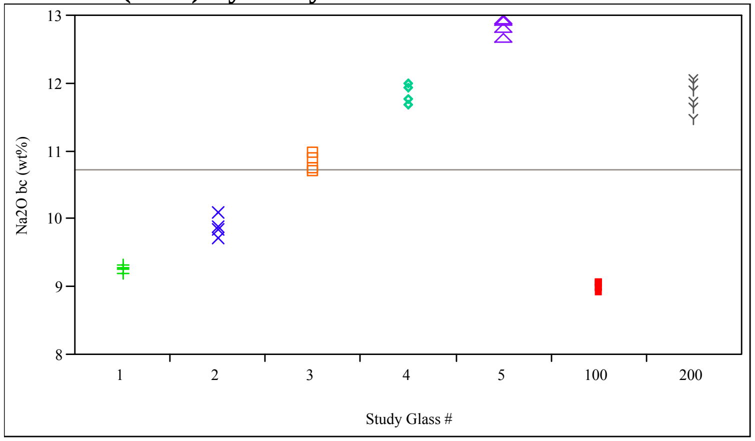
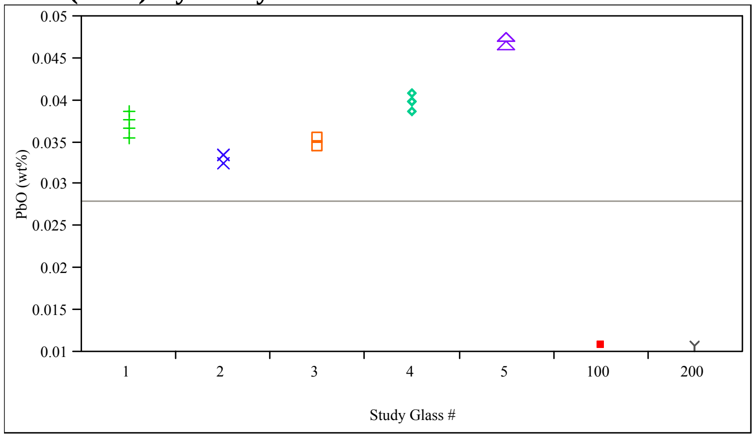


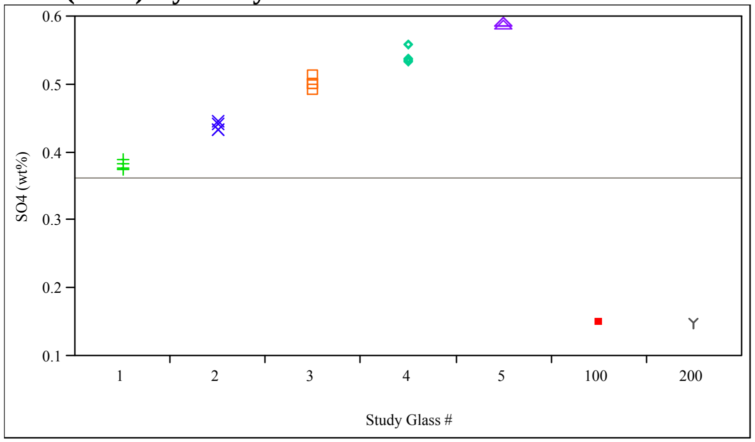
Exhibit D5. Measured and Measured Bias-Corrected Oxide Weight Percents by Glass # for the Glasses Prepared Using the LM Method (continued)

(100 – Batch 1; 200 – Ustd)

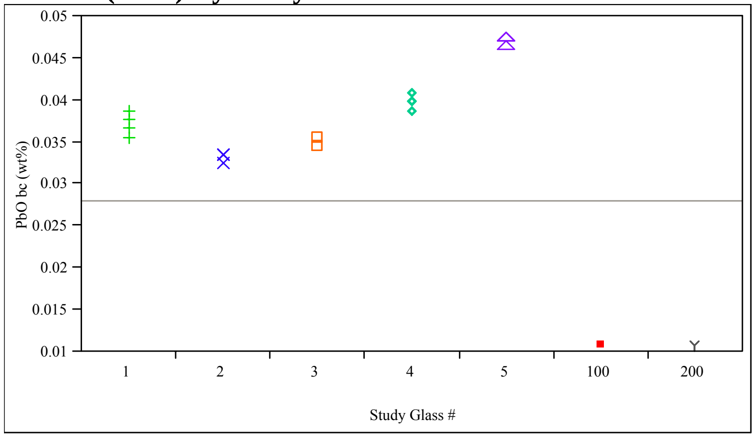
PbO (wt%) By Study Glass #



SO4 (wt%) By Study Glass #



PbO bc (wt%) By Study Glass #



SO4 bc (wt%) By Study Glass #

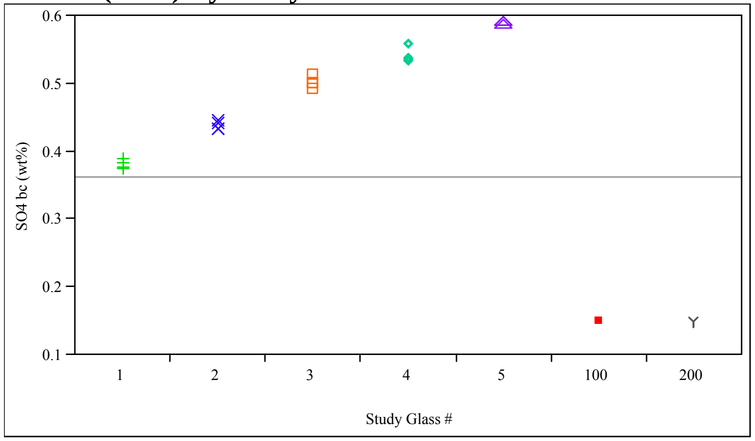
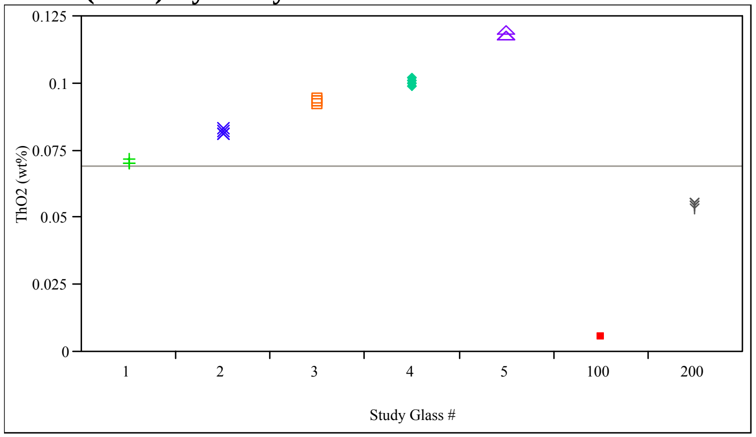
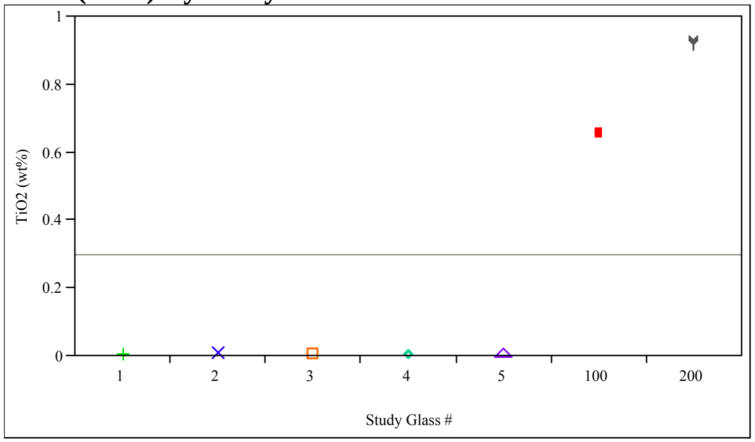
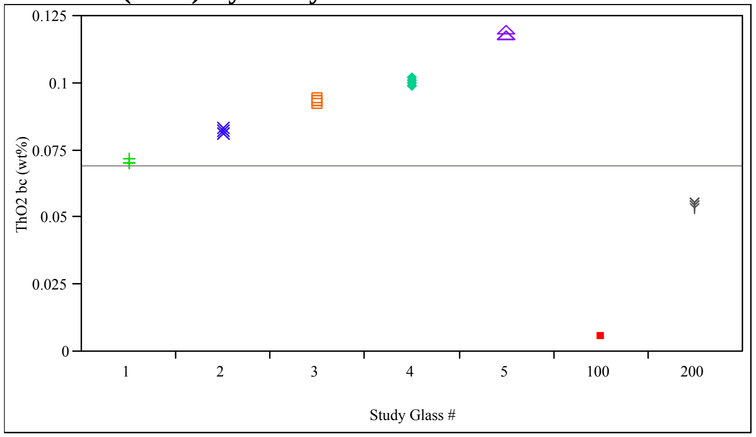


Exhibit D5. Measured and Measured Bias-Corrected Oxide Weight Percents by Glass # for the Glasses Prepared Using the LM Method (continued)

(100 – Batch 1; 200 – Ustd)


ThO2 (wt%) By Study Glass #


TiO2 (wt%) By Study Glass #


ThO2 bc (wt%) By Study Glass #

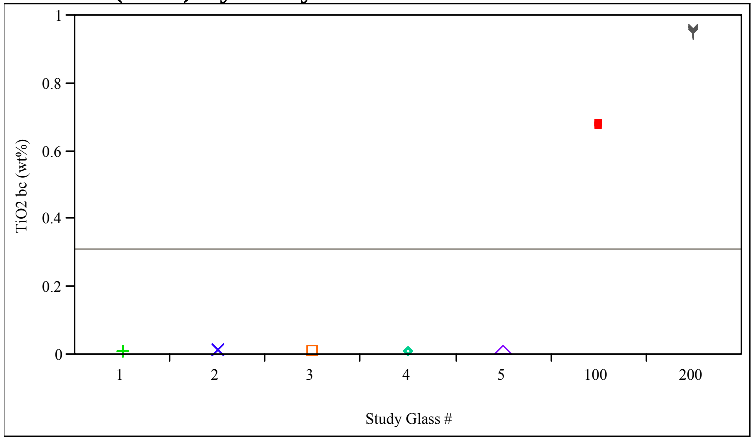
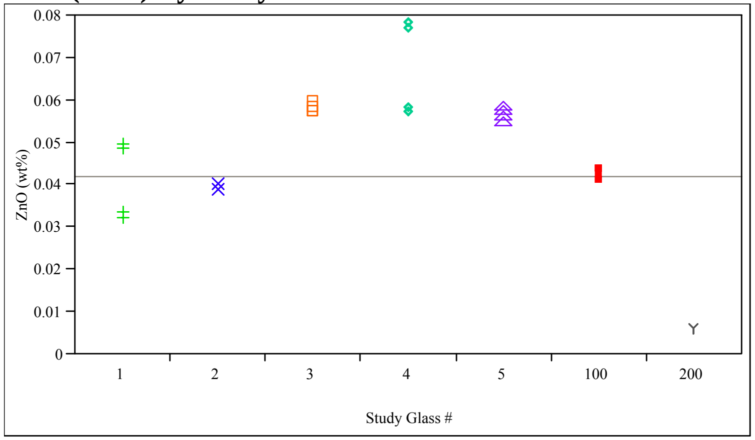

TiO2 bc (wt%) By Study Glass #

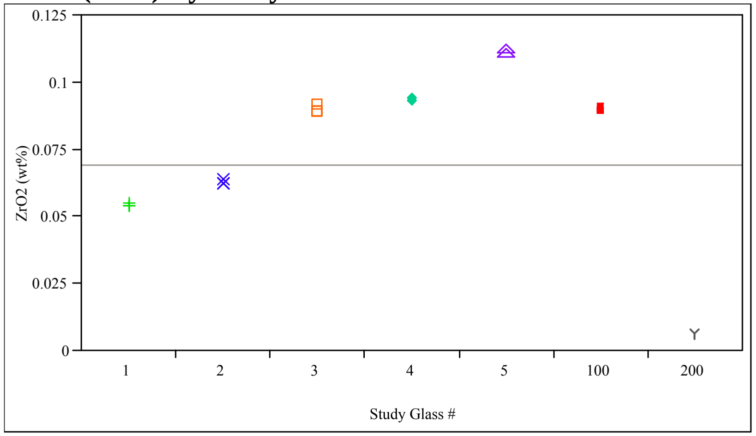
Exhibit D5. Measured and Measured Bias-Corrected Oxide Weight Percents by Glass # for the Glasses Prepared Using the LM Method (continued)

(100 – Batch 1; 200 – Ustd)

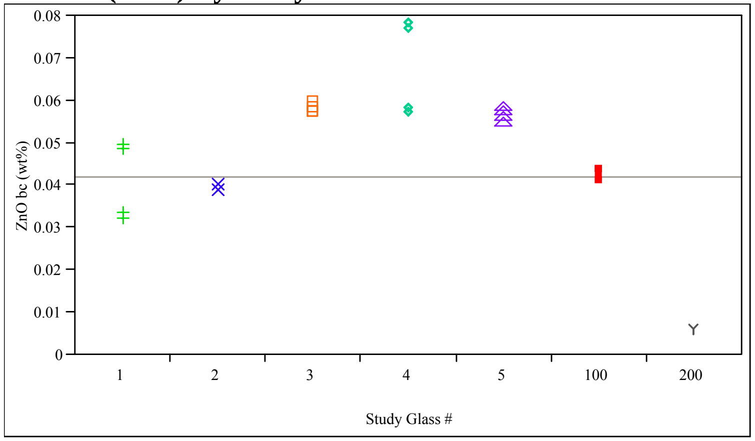
ZnO (wt%) By Study Glass #



ZrO2 (wt%) By Study Glass #



ZnO bc (wt%) By Study Glass #



ZrO2 bc (wt%) By Study Glass #

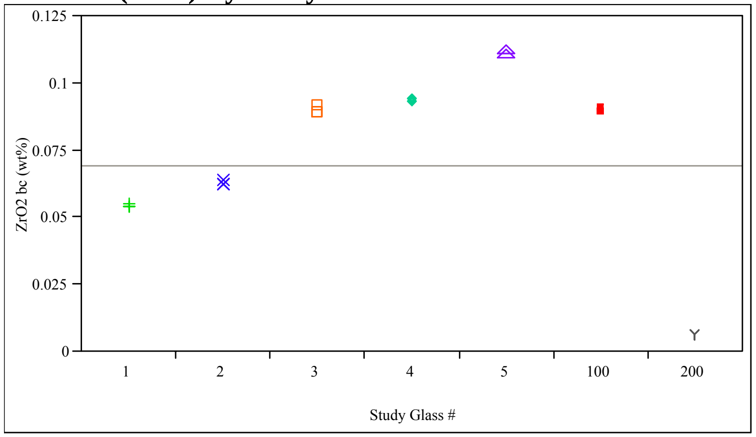
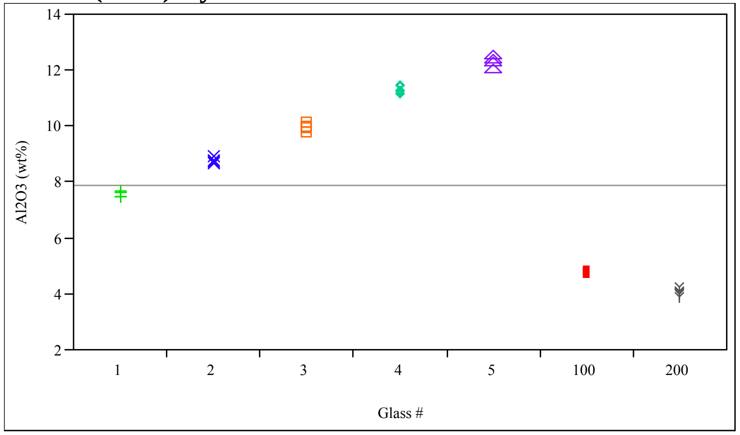


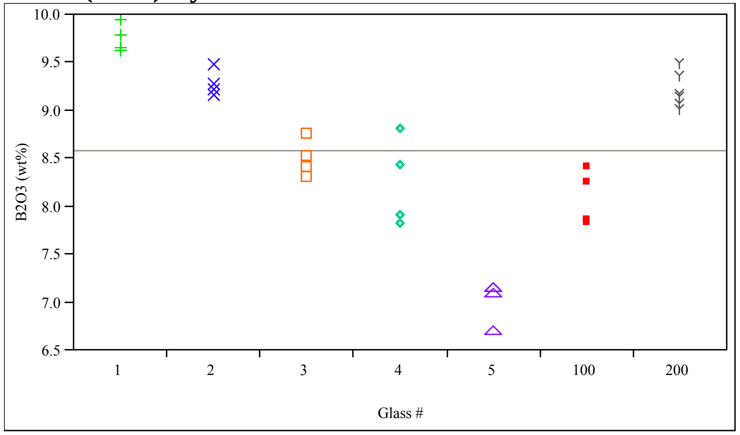
Exhibit D6. Measured and Measured Bias-Corrected Oxide Weight Percent by Glass # for the Glasses Prepared Using the PF Method

(100 – Batch 1; 200 – Ustd)

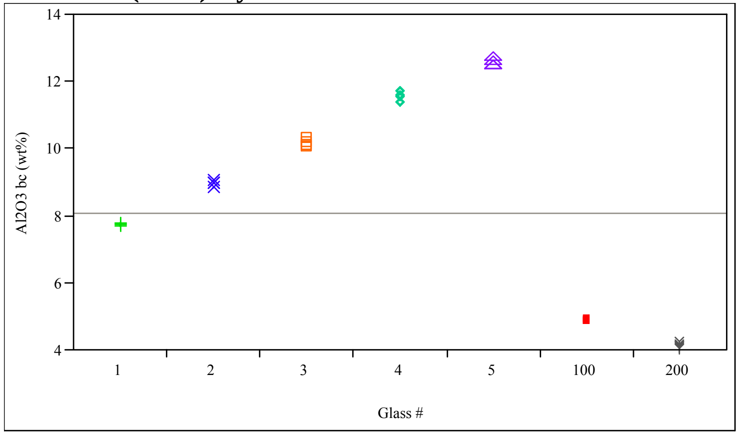
Al2O3 (wt%) By Glass



B2O3 (wt%) By Glass



Al2O3 bc (wt%) By Glass



B2O3 bc (wt%) By Glass

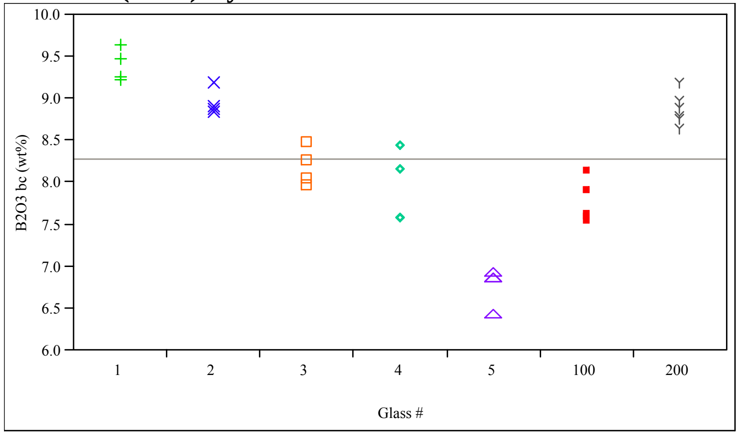
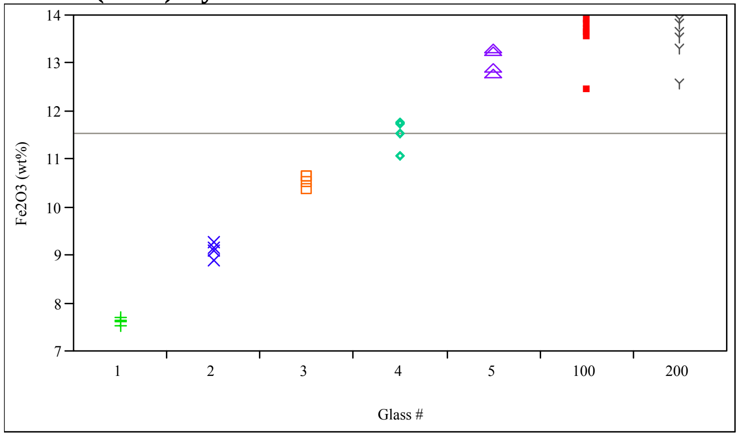


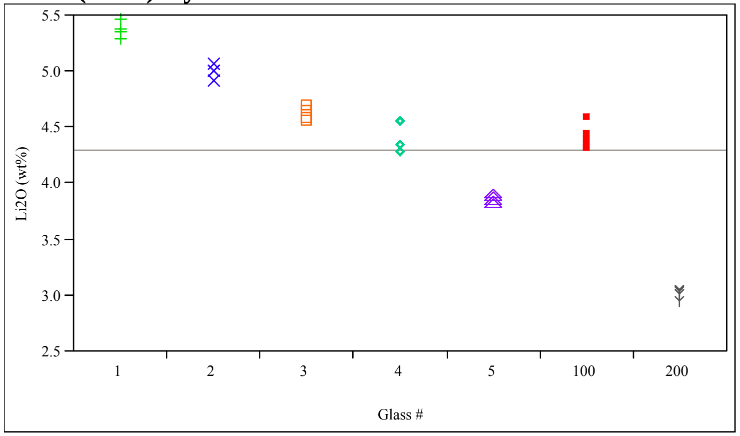
Exhibit D6. Measured and Measured Bias-Corrected Oxide Weight Percent by Glass # for the Glasses Prepared Using the PF Method (continued)

(100 – Batch 1; 200 – Ustd)

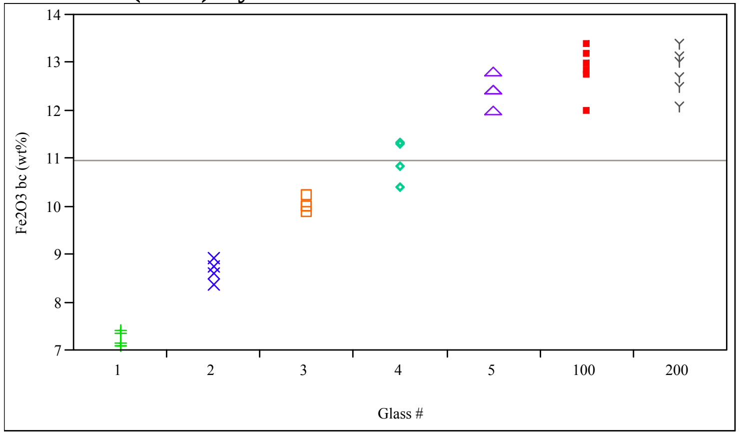
Fe2O3 (wt%) By Glass #



Li2O (wt%) By Glass #



Fe2O3 bc (wt%) By Glass #



Li2O bc (wt%) By Glass #

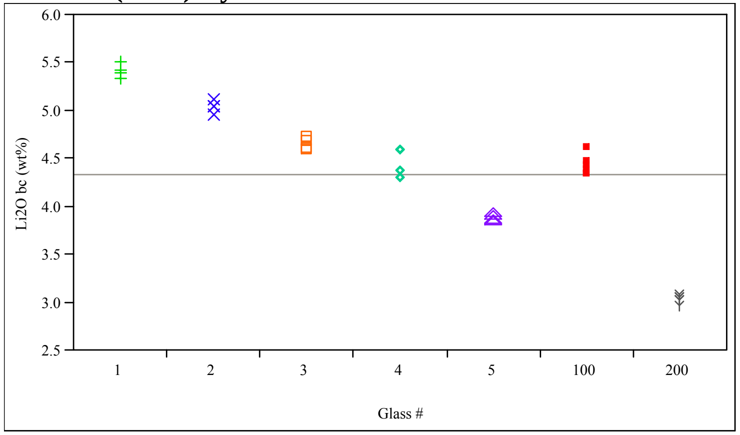
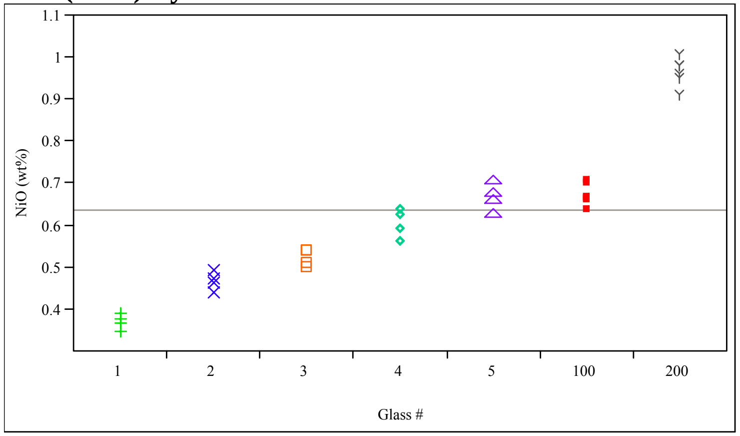


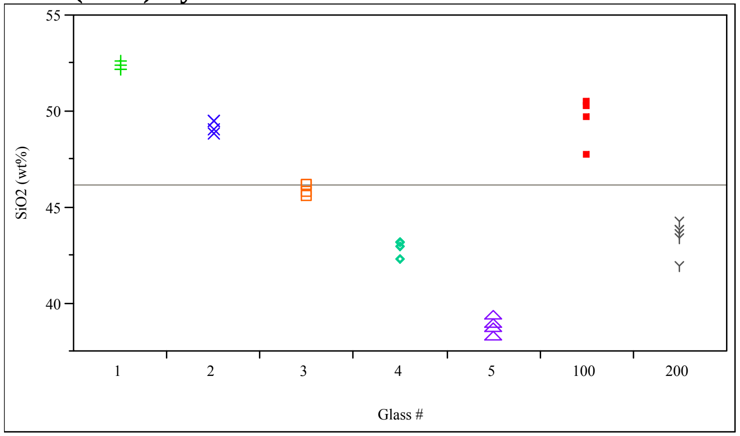
Exhibit D6. Measured and Measured Bias-Corrected Oxide Weight Percent by Glass # for the Glasses Prepared Using the PF Method (continued)

(100 – Batch 1; 200 – Ustd)

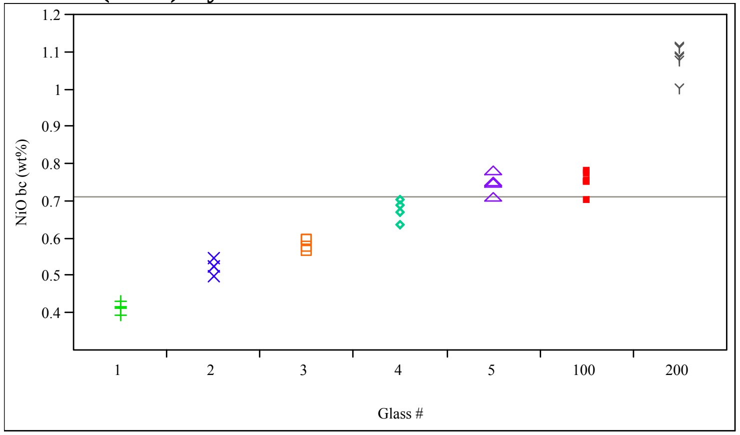
NiO (wt%) By Glass #



SiO2 (wt%) By Glass #



NiO bc (wt%) By Glass #



SiO2 bc (wt%) By Glass #

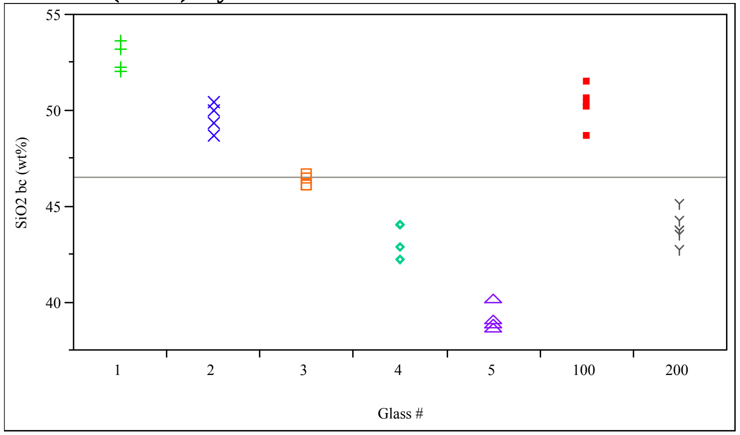
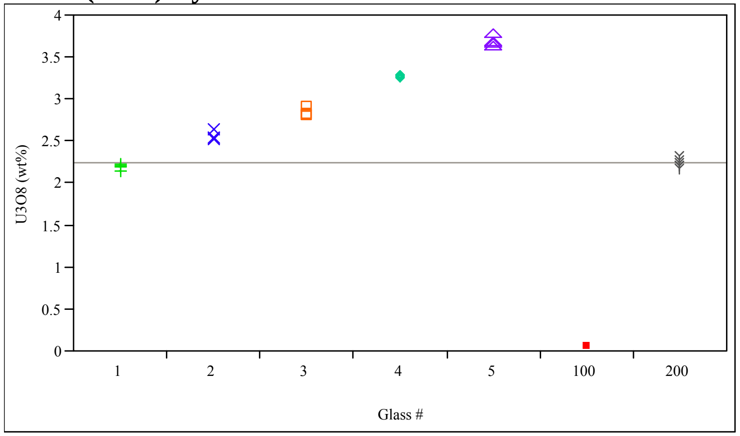


Exhibit D6. Measured and Measured Bias-Corrected Oxide Weight Percent by Glass # for the Glasses Prepared Using the PF Method (continued)

(100 – Batch 1; 200 – Ustd)

U3O8 (wt%) By Glass



U3O8 bc (wt%) By Glass

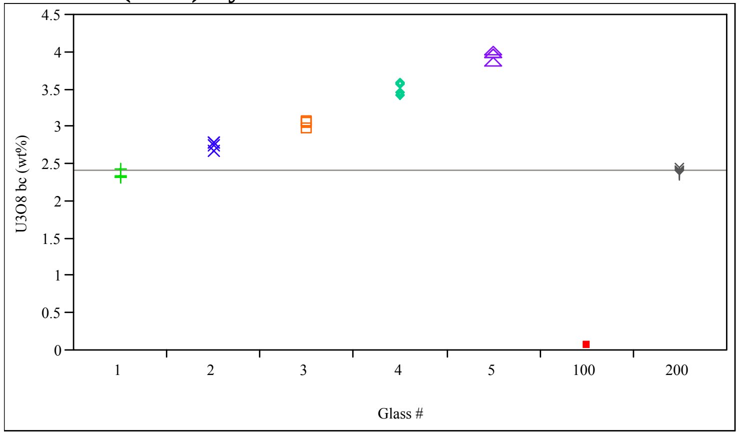


Exhibit D7. Average Measured and Bias-Corrected (bc) Versus Targeted Compositions by Glass # by Oxide

(100 – Batch 1; 200 – Ustd)

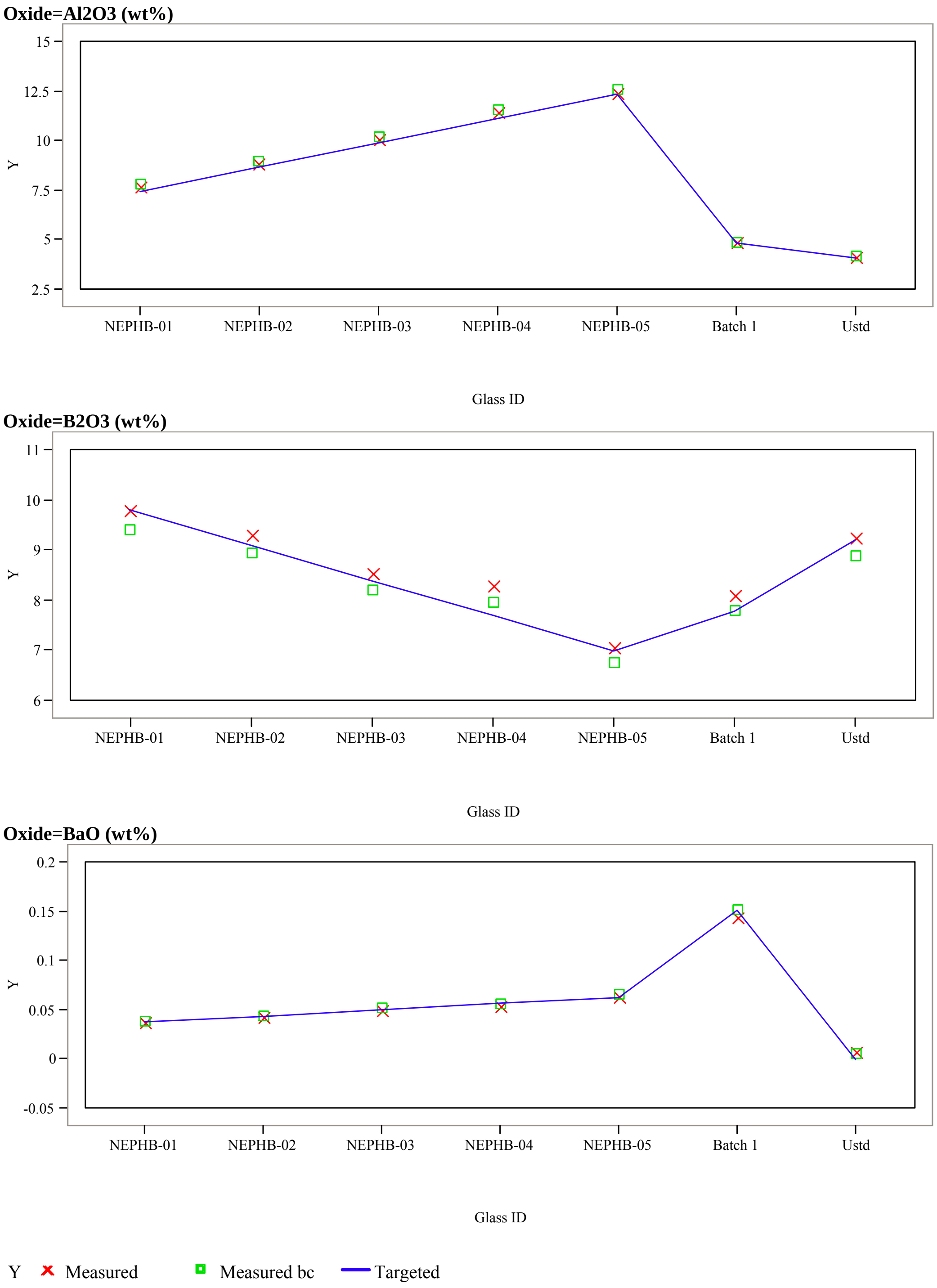
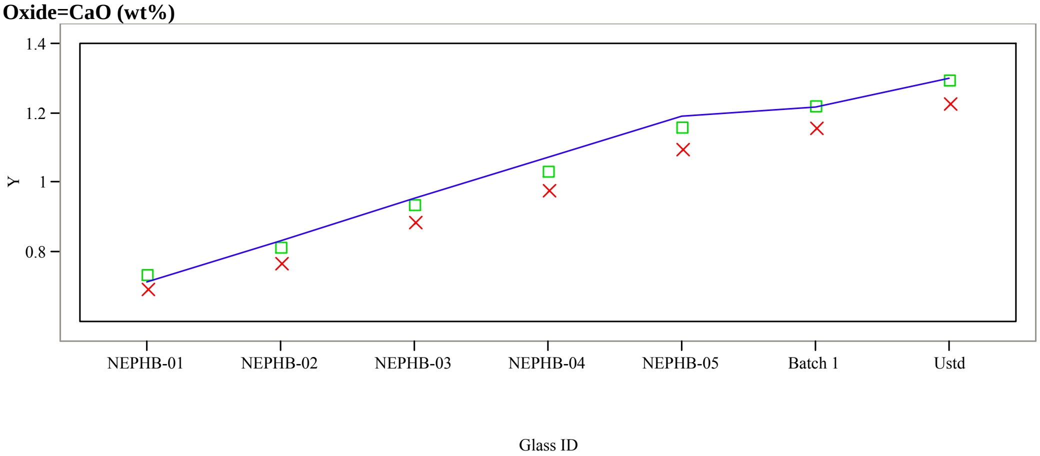


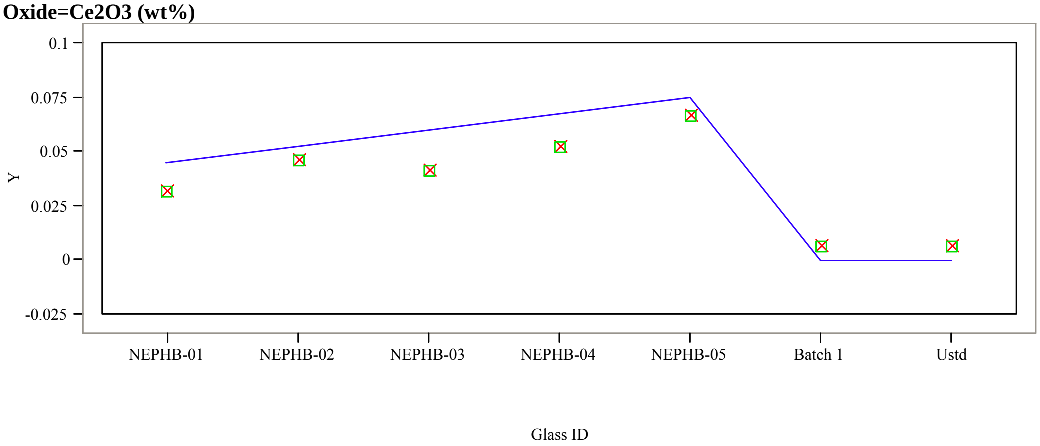
Exhibit D7. Average Measured and Bias-Corrected (bc) Versus Targeted Compositions by Glass # by Oxide (continued)

(100 – Batch 1; 200 – Ustd)

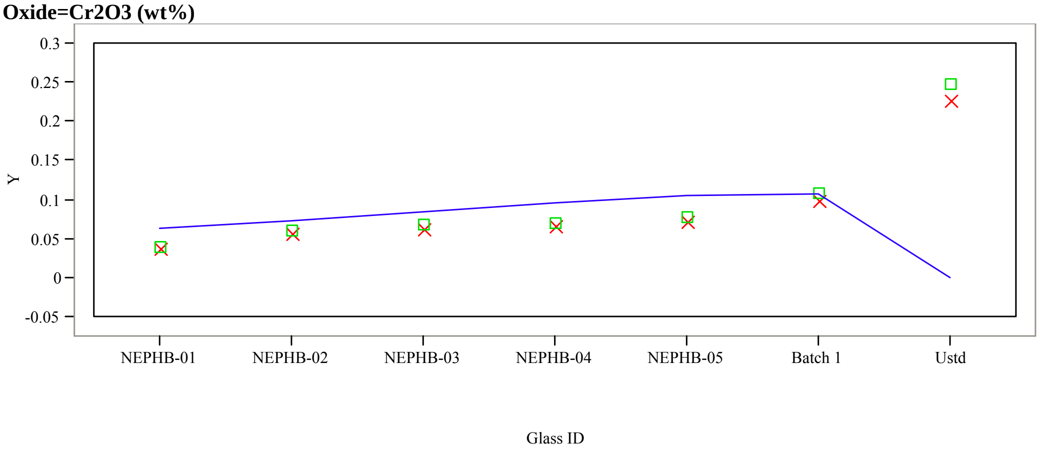
Oxide=CaO (wt%)



Oxide=Ce2O3 (wt%)



Oxide=Cr2O3 (wt%)



Y ✕ Measured 🟩 Measured bc — Targeted

Exhibit D7. Average Measured and Bias-Corrected (bc) Versus Targeted Compositions by Glass # by Oxide (continued)

(100 – Batch 1; 200 – Ustd)

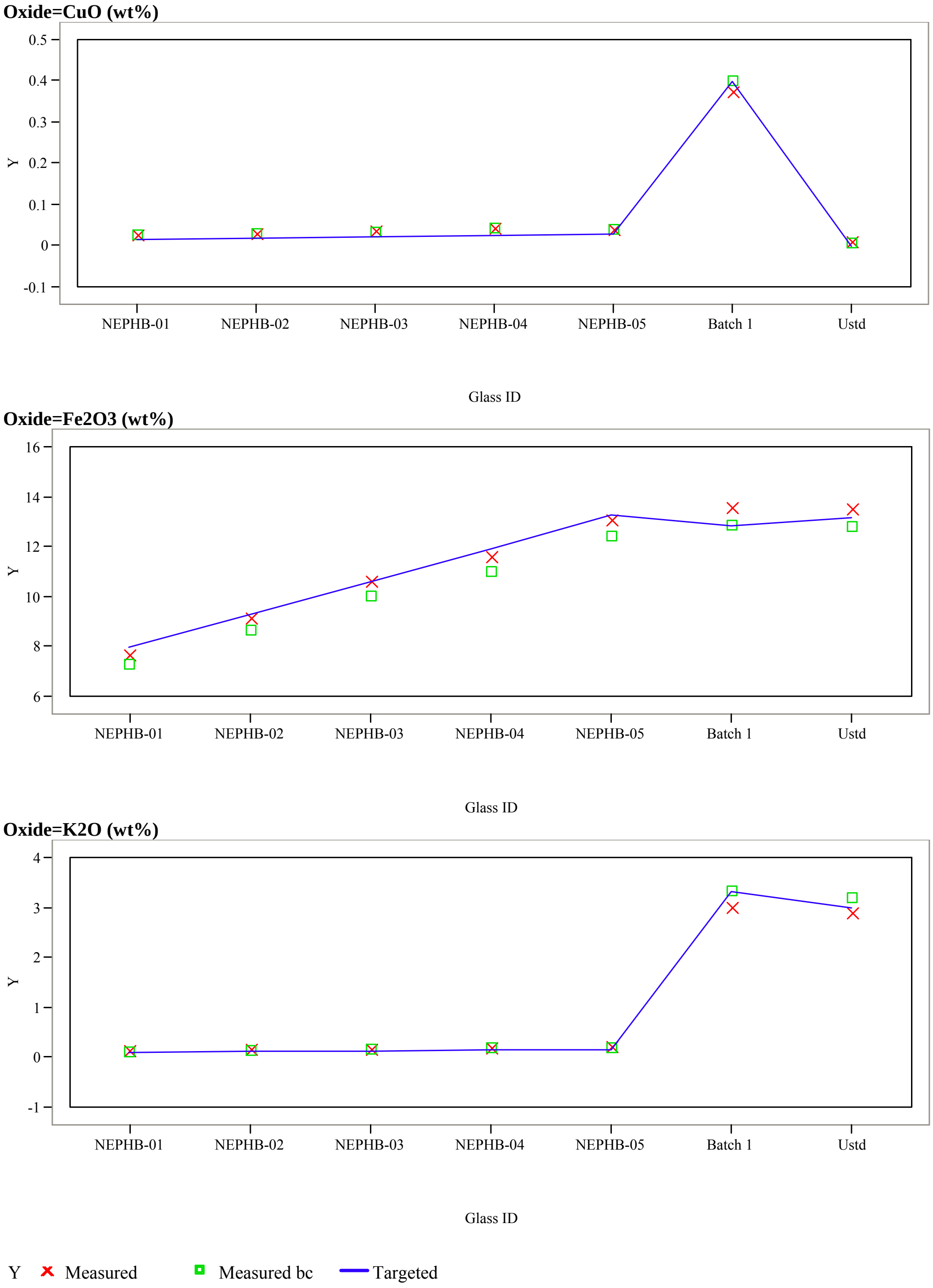


Exhibit D7. Average Measured and Bias-Corrected (bc) Versus Targeted Compositions by Glass # by Oxide (continued)

(100 – Batch 1; 200 – Ustd)

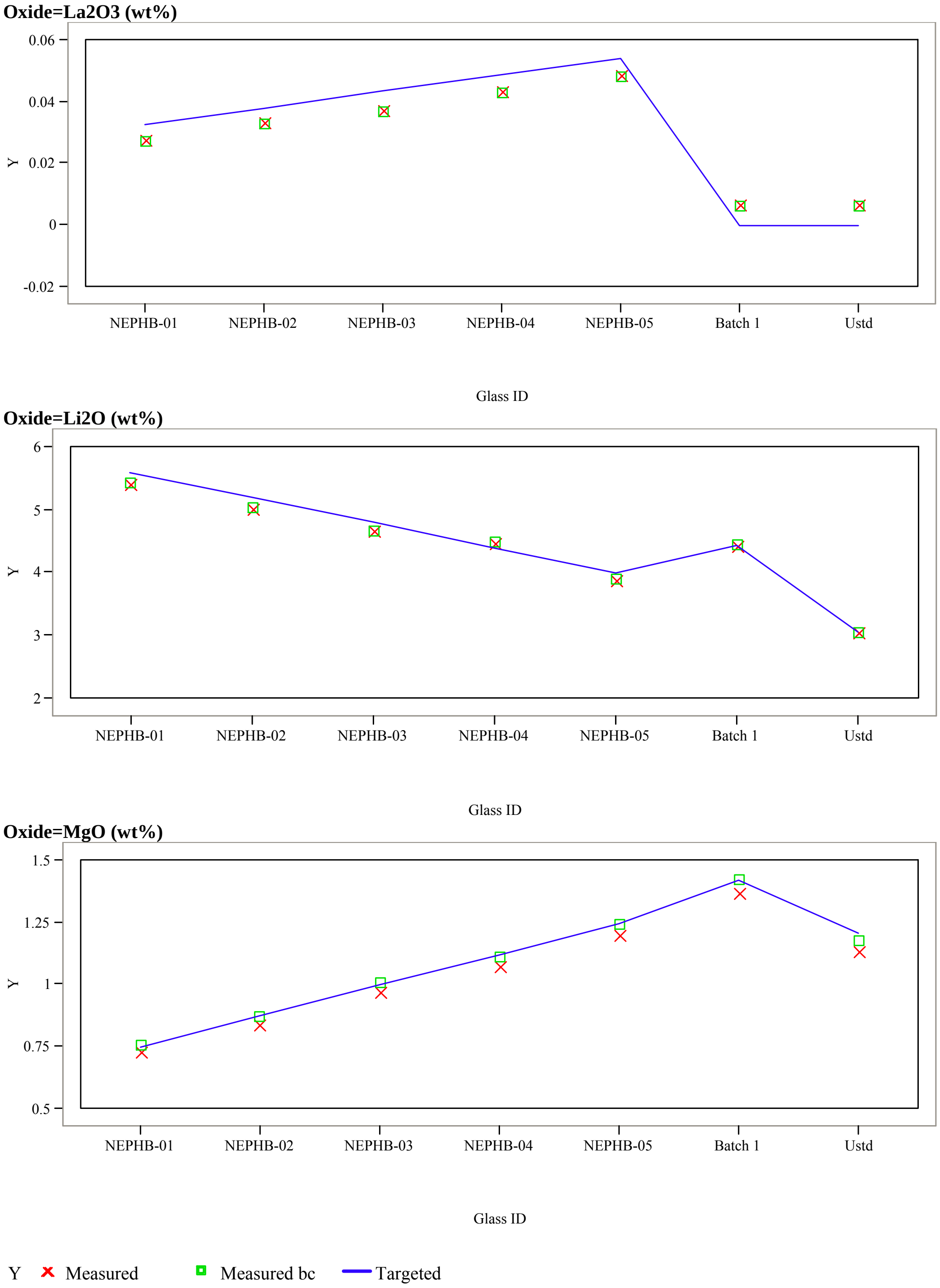
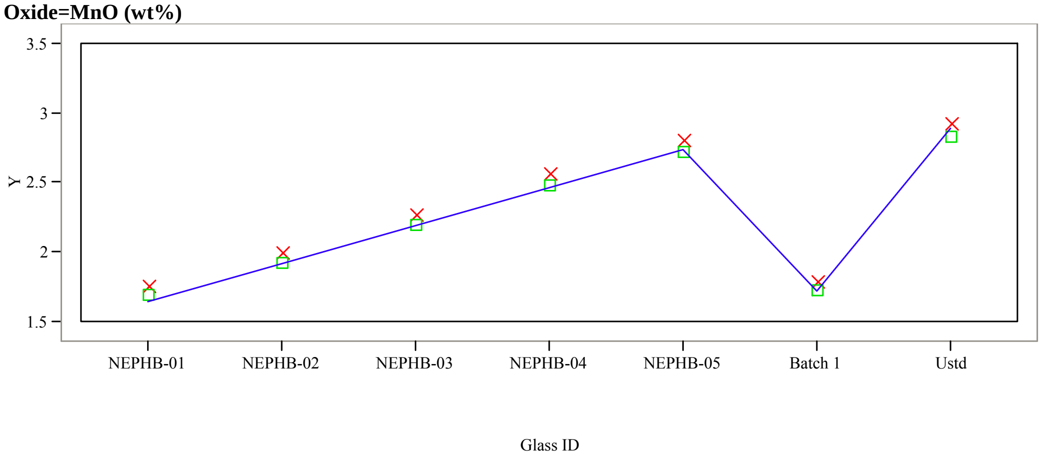


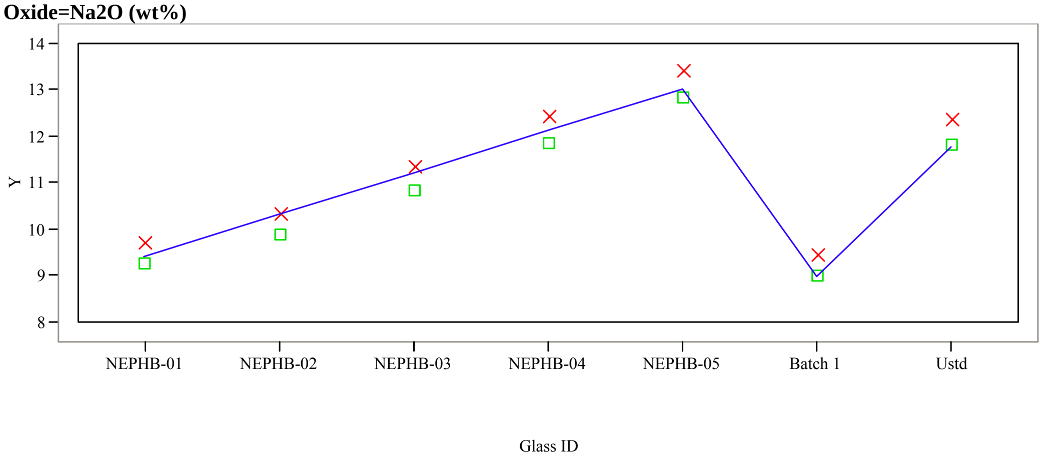
Exhibit D7. Average Measured and Bias-Corrected (bc) Versus Targeted Compositions by Glass # by Oxide (continued)

(100 – Batch 1; 200 – Ustd)

Oxide=MnO (wt%)



Oxide=Na2O (wt%)



Oxide=NiO (wt%)

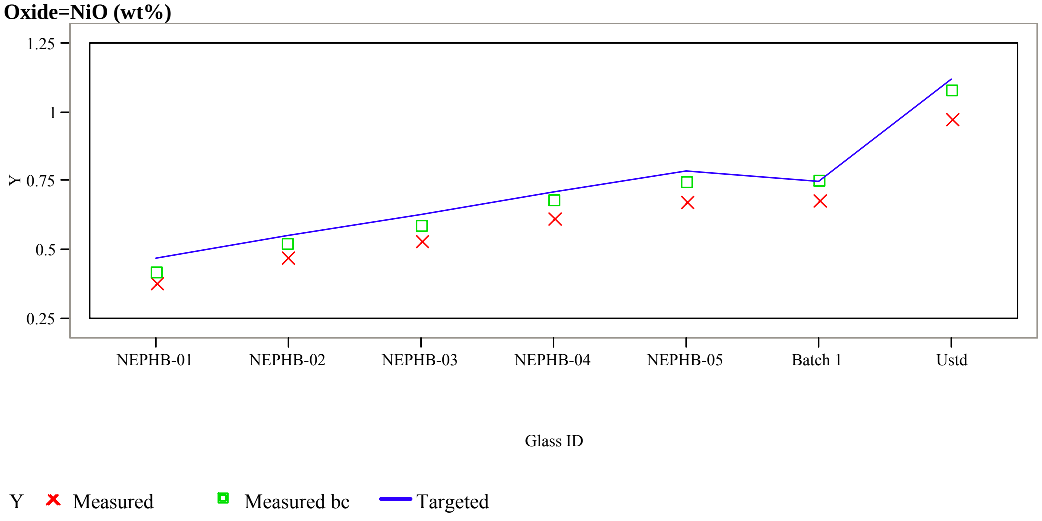
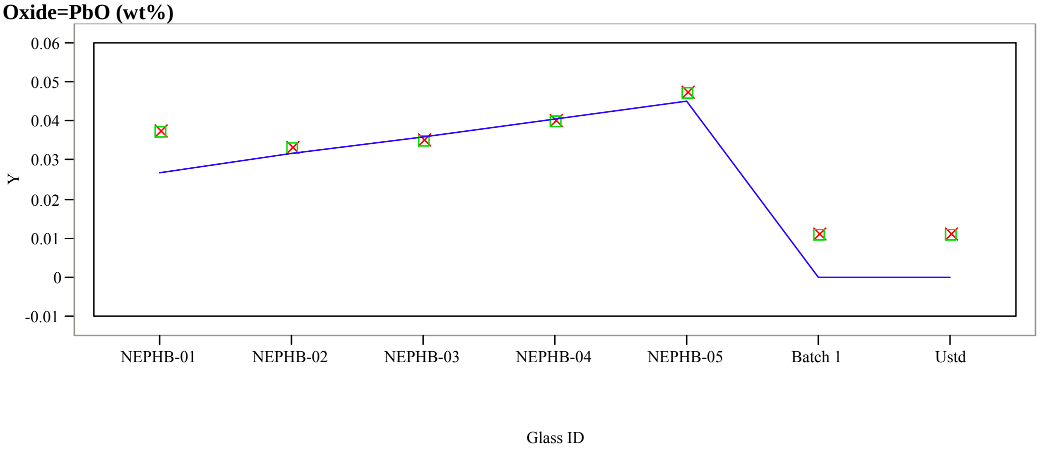


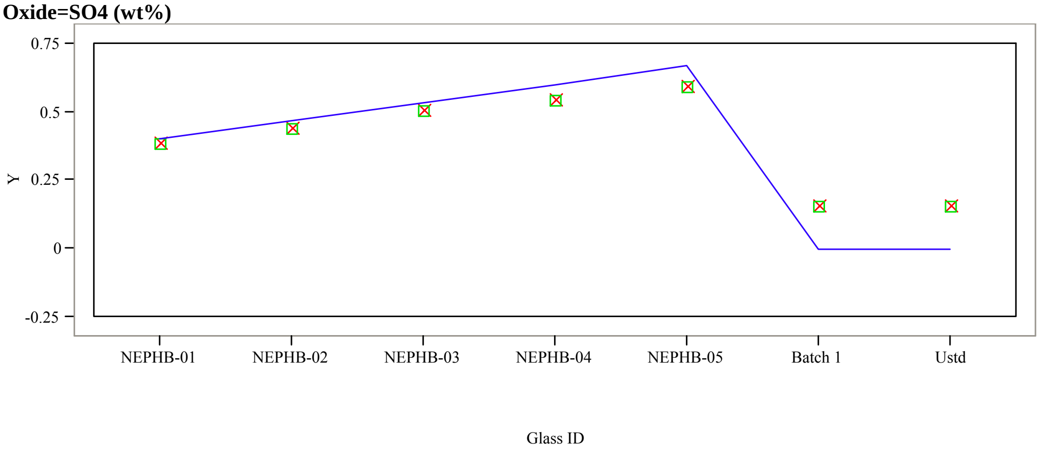
Exhibit D7. Average Measured and Bias-Corrected (bc) Versus Targeted Compositions by Glass # by Oxide (continued)

(100 – Batch 1; 200 – Ustd)

Oxide=PbO (wt%)



Oxide=SO4 (wt%)



Oxide=SiO2 (wt%)

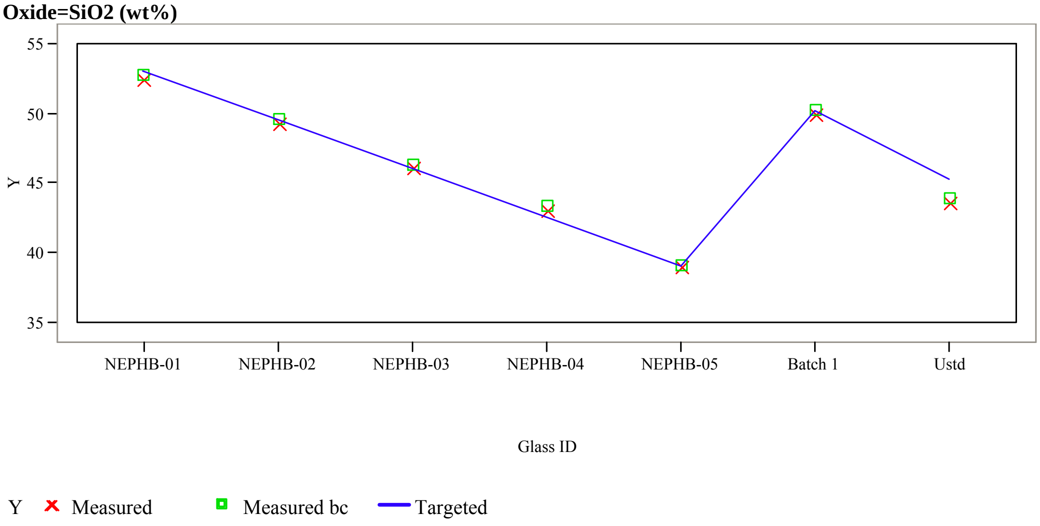
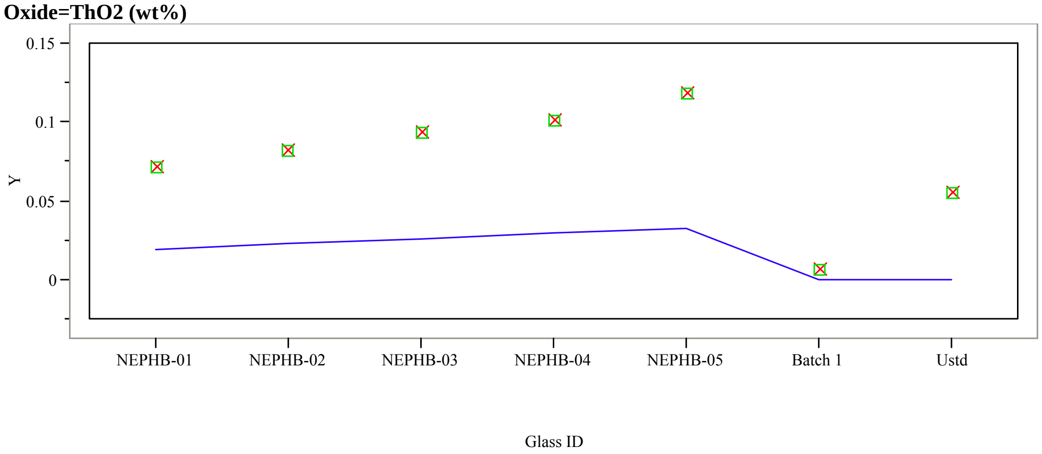


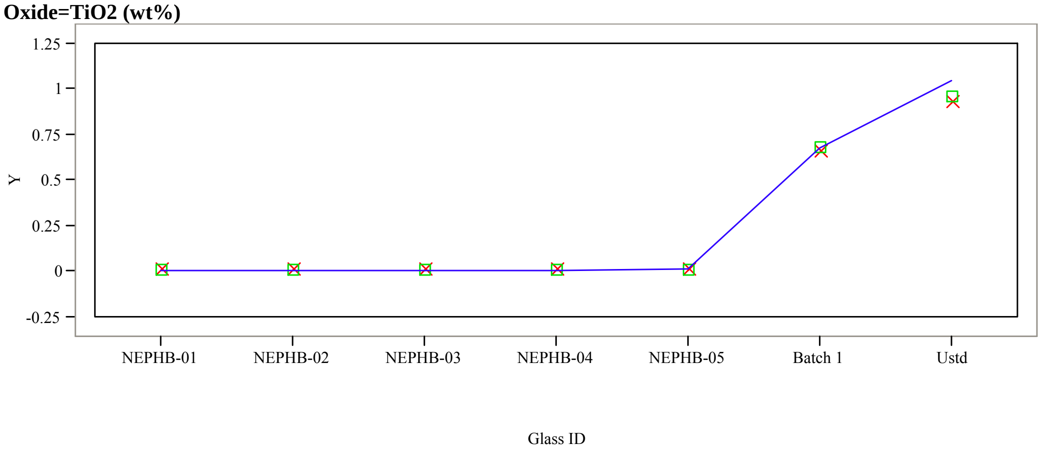
Exhibit D7. Average Measured and Bias-Corrected (bc) Versus Targeted Compositions by Glass # by Oxide (continued)

(100 – Batch 1; 200 – Ustd)

Oxide=ThO2 (wt%)



Oxide=TiO2 (wt%)



Oxide=U3O8 (wt%)

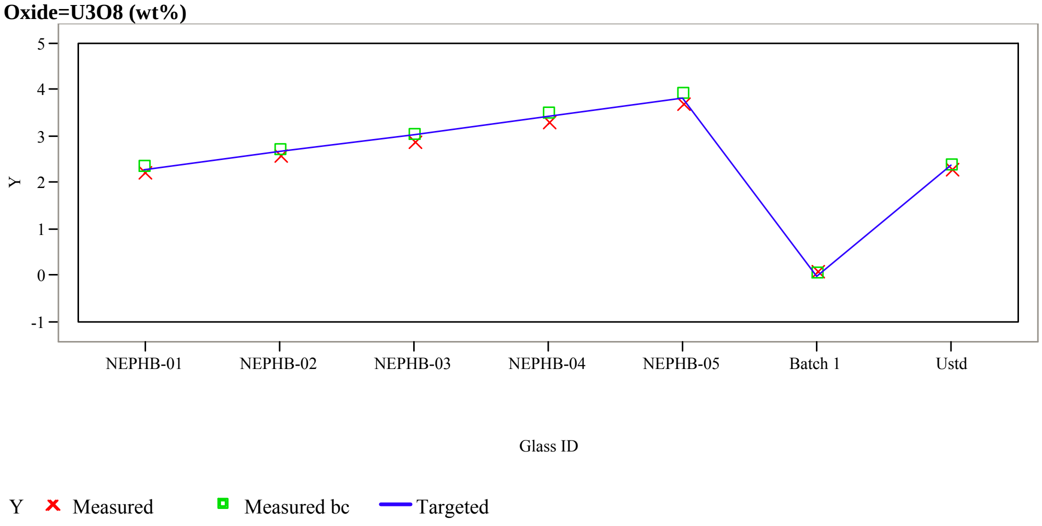
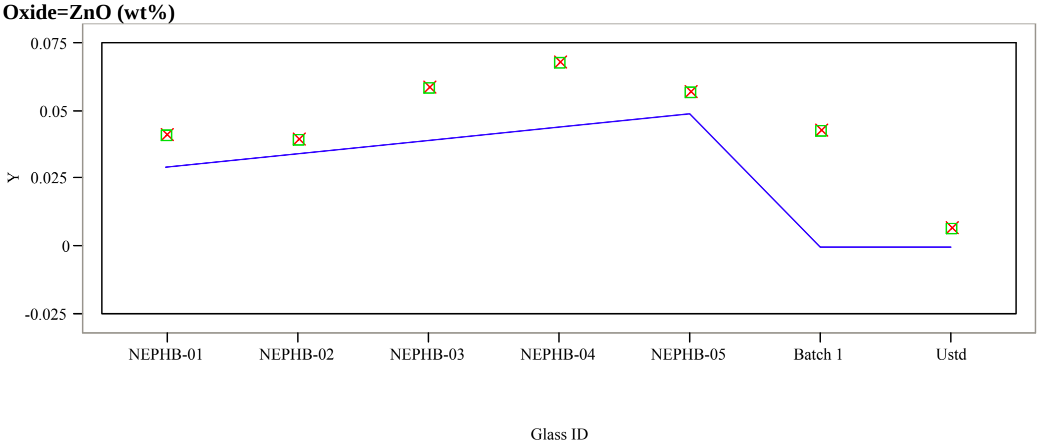


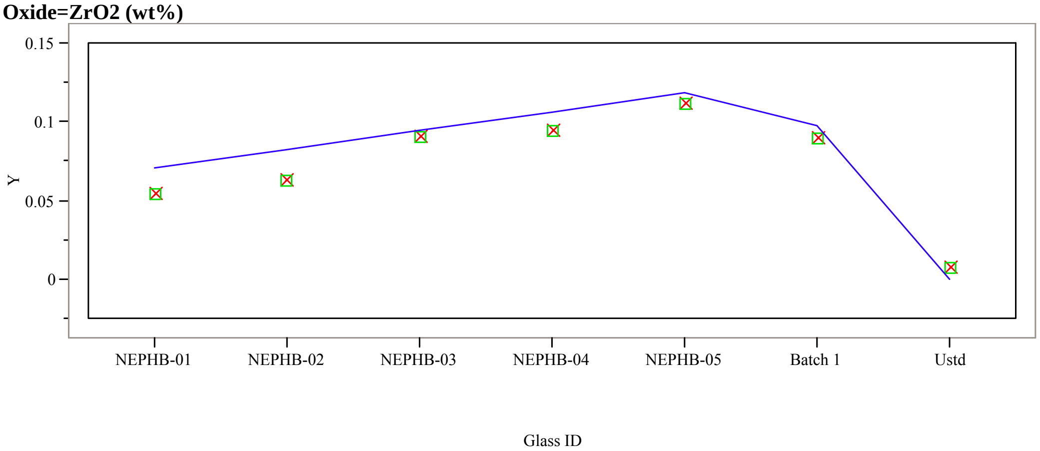
Exhibit D7. Average Measured and Bias-Corrected (bc) Versus Targeted Compositions by Glass # by Oxide (continued)

(100 – Batch 1; 200 – Ustd)

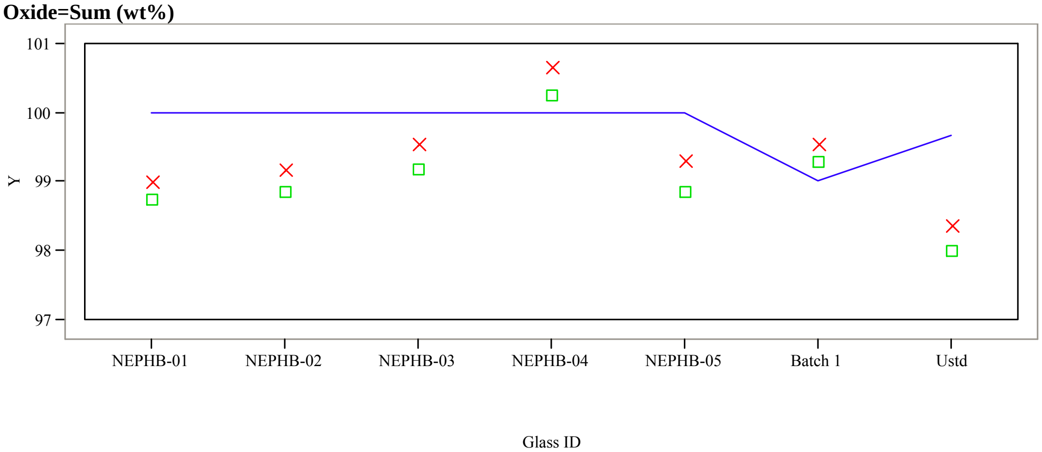
Oxide=ZnO (wt%)



Oxide=ZrO2 (wt%)



Oxide=Sum (wt%)



Y ✕ Measured 🟩 Measured bc — Targeted

This page intentionally left blank.

Appendix E

Tables and Exhibits Supporting the Analysis of the PCT Results for the Frit 503 – SB4 Study Glasses

Table E1. Laboratory Measurements of the PCT Solutions for the Frit 503/SB4 Study Glasses in ppm

Glass ID	Heat Treatment	Laboratory ID	Block	Seq	B ar	Ba ar	Cd ar	Cr ar	Fe ar	Li ar	Na ar	Pb ar	Si ar	Th ar	U ar
Soln Std		STD-B1-1	1	1	19.6	<0.010	<0.080	<0.010	4.56	9.6	81.4	<0.020	49.3	<0.100	<0.200
blank		J02	1	2	0.194	<0.010	<0.080	<0.010	<0.004	<0.500	0.255	<0.020	<0.100	<0.100	<0.200
ARM-1		J15	1	3	10.8	<0.010	<0.080	<0.010	<0.004	8.35	22	<0.020	36.8	<0.100	<0.200
NEPHB-04	quenched	J34	1	4	11.2	<0.010	<0.080	<0.010	2.56	9.47	39.4	<0.020	53.3	<0.100	1.27
NEPHB-04ccc	ccc	J37	1	5	18.4	<0.010	<0.080	<0.010	3.45	14.2	48.7	<0.020	59.7	<0.100	1.46
NEPHB-05	quenched	J25	1	6	11.8	<0.010	<0.080	<0.010	3.32	9.08	50.5	<0.020	52.2	<0.100	1.37
NEPHB-03	quenched	J28	1	7	11.4	<0.010	<0.080	<0.010	2.71	10	31.3	<0.020	57	<0.100	1.63
NEPHB-01ccc	ccc	J26	1	8	11.5	<0.010	<0.080	<0.010	3.83	10.5	21.6	<0.020	62.4	<0.100	3.32
Soln Std		STD-B1-2	1	9	19.9	<0.010	<0.080	<0.010	4.59	9.44	82	<0.020	48.2	<0.100	<0.200
EA		J16	1	10	35.4	<0.010	<0.080	<0.010	<0.004	10.4	97.7	<0.020	50.3	<0.100	<0.200
NEPHB-03ccc	ccc	J29	1	11	10.8	<0.010	<0.080	<0.010	2.71	9.56	29.6	<0.020	55.2	<0.100	1.54
NEPHB-02ccc	ccc	J04	1	12	10.9	<0.010	<0.080	<0.010	3	9.78	23.9	<0.020	58.9	<0.100	1.96
NEPHB-01	quenched	J24	1	13	11.6	<0.010	<0.080	<0.010	4.32	11.3	19.1	<0.020	64.6	<0.100	3.68
NEPHB-02	quenched	J30	1	14	11.1	<0.010	<0.080	<0.010	3.13	10.4	23.9	<0.020	59.6	<0.100	2.12
NEPHB-05ccc	ccc	J12	1	15	9.87	<0.010	<0.080	<0.010	1.64	8.86	42.2	<0.020	46.5	<0.100	1.06
Soln Std		STD-B1-3	1	16	19.8	<0.010	<0.080	<0.010	4.47	9.29	82.6	<0.020	47.5	<0.100	<0.200
Soln Std		STD-B2-1	2	1	19.5	<0.010	<0.080	<0.010	4.47	9.71	81.5	<0.020	49.3	<0.100	<0.200
NEPHB-03ccc	ccc	J11	2	2	10.8	<0.010	<0.080	<0.010	2.75	9.63	30.7	<0.020	55.1	<0.100	1.47
NEPHB-01ccc	ccc	J36	2	3	11.5	<0.010	<0.080	<0.010	3.72	10.6	20.3	<0.020	62.8	<0.100	3.27
EA		J31	2	4	30.6	<0.010	<0.080	<0.010	<0.004	9.07	84.2	<0.020	44.9	<0.100	<0.200
NEPHB-05ccc	ccc	J06	2	5	11	<0.010	<0.080	<0.010	2.19	9.34	46.1	<0.020	50.5	<0.100	1.22
ARM-1		J14	2	6	11.6	<0.010	<0.080	<0.010	<0.004	8.42	23.1	<0.020	36.9	<0.100	<0.200
NEPHB-03	quenched	J35	2	7	11	<0.010	<0.080	<0.010	2.82	9.65	31	<0.020	54.4	<0.100	1.43
NEPHB-02	quenched	J32	2	8	11.2	<0.010	<0.080	<0.010	3.02	10.4	24.6	<0.020	59.1	<0.100	2.08
Soln Std		STD-B2-2	2	9	19.9	<0.010	<0.080	<0.010	4.64	9.48	83.9	<0.020	48.4	<0.100	<0.200
NEPHB-05	quenched	J10	2	10	11.6	<0.010	<0.080	<0.010	3.22	9.17	52	<0.020	51.1	<0.100	1.31
NEPHB-04ccc	ccc	J08	2	11	18.4	<0.010	<0.080	<0.010	3.08	14.1	50.7	<0.020	59.6	<0.100	1.47
NEPHB-04	quenched	J38	2	12	11.3	<0.010	<0.080	<0.010	2.61	9.38	40.7	<0.020	52	<0.100	1.26
NEPHB-02ccc	ccc	J13	2	13	10.9	<0.010	<0.080	<0.010	2.94	9.79	25.6	<0.020	58.3	<0.100	1.95
NEPHB-01	quenched	J05	2	14	11.4	<0.010	<0.080	<0.010	4.25	11.1	19.7	<0.020	63.8	<0.100	3.59
Soln Std		STD-B2-3	2	15	19.8	<0.010	<0.080	<0.010	4.5	9.6	84	<0.020	48.7	<0.100	<0.200
Soln Std		STD-B3-1	3	1	19.6	<0.010	<0.080	<0.010	4.5	9.72	80.4	<0.020	51	<0.100	<0.200

Table E1. Laboratory Measurements of the PCT Solutions for the Frit 503/SB4 Study Glasses in ppm (continued)

Glass ID	Heat Treatment	Laboratory ID	Block	Seq	B ar	Ba ar	Cd ar	Cr ar	Fe ar	Li ar	Na ar	Pb ar	Si ar	Th ar	U ar
NEPHB-01ccc	ccc	J17	3	2	11.4	<0.010	<0.080	<0.010	3.66	10.7	19.3	<0.020	64.2	<0.100	3.17
NEPHB-02ccc	ccc	J22	3	3	10.9	<0.010	<0.080	<0.010	2.94	10	24.4	<0.020	61.2	<0.100	1.87
NEPHB-05	quenched	J20	3	4	11.2	<0.010	<0.080	<0.010	3.14	8.98	49.1	<0.020	51.9	<0.100	1.3
NEPHB-02	quenched	J07	3	5	10.9	<0.010	<0.080	<0.010	3.03	10.4	23.4	<0.020	60.3	<0.100	1.89
ARM-1		J19	3	6	11.6	<0.010	<0.080	<0.010	<0.004	8.62	23.2	<0.020	37.8	<0.100	<0.200
NEPHB-05ccc	ccc	J27	3	7	10.7	<0.010	<0.080	<0.010	2.21	9.65	47.6	<0.020	53.5	<0.100	1.2
NEPHB-03ccc	ccc	J03	3	8	11	<0.010	<0.080	<0.010	2.75	9.7	30	<0.020	57.9	<0.100	1.29
Soln Std		STD-B3-2	3	9	20.1	<0.010	<0.080	<0.010	4.66	9.54	81.8	<0.020	49.5	<0.100	<0.200
NEPHB-04	quenched	J33	3	10	11.1	<0.010	<0.080	<0.010	2.39	9.39	39.4	<0.020	54.1	<0.100	1.31
blank		J21	3	11	0.116	<0.010	<0.080	<0.010	<0.004	<0.500	<0.100	<0.020	<0.100	<0.100	<0.200
NEPHB-04ccc	ccc	J09	3	12	18.3	<0.010	<0.080	<0.010	3.18	14.3	49.3	<0.020	62.5	<0.100	1.47
EA		J23	3	13	20.9	<0.010	<0.080	<0.010	<0.004	6.48	61.1	<0.020	35.3	<0.100	<0.200
NEPHB-01	quenched	J18	3	14	11.9	<0.010	<0.080	<0.010	4.37	11.4	20.1	<0.020	67.3	<0.100	3.49
NEPHB-03	quenched	J01	3	15	11.1	<0.010	<0.080	<0.010	3.01	9.77	29.7	<0.020	57.7	<0.100	1.5
Soln Std		STD-B3-3	3	16	20.1	<0.010	<0.080	<0.010	4.62	9.6	81	<0.020	50.7	<0.100	<0.200

Table E2. PSAL Measurements of the PCT Solutions for the Study Glasses After Appropriate Adjustments

Glass ID	Heat Treatment	Laboratory ID	Block	Seq	B (ppm)	Ba (ppm)	Cd (ppm)	Cr (ppm)	Fe (ppm)	Li (ppm)	Na (ppm)	Pb (ppm)	Si (ppm)	Th (ppm)	U (ppm)
Soln Std		STD-B1-1	1	1	19.600	0.005	0.040	0.005	4.560	9.600	81.400	0.010	49.300	0.050	0.100
blank		J02	1	2	0.323	0.008	0.067	0.008	0.003	0.417	0.425	0.017	0.083	0.083	0.167
ARM-1		J15	1	3	18.000	0.008	0.067	0.008	0.003	13.917	36.667	0.017	61.335	0.083	0.167
NEPHB-04	quenched	J34	1	4	18.667	0.008	0.067	0.008	4.267	15.784	65.668	0.017	88.835	0.083	2.117
NEPHB-04ccc	ccc	J37	1	5	30.667	0.008	0.067	0.008	5.750	23.667	81.168	0.017	99.502	0.083	2.433
NEPHB-05	quenched	J25	1	6	19.667	0.008	0.067	0.008	5.533	15.134	84.168	0.017	87.002	0.083	2.283
NEPHB-03	quenched	J28	1	7	19.000	0.008	0.067	0.008	4.517	16.667	52.168	0.017	95.002	0.083	2.717
NEPHB-01ccc	ccc	J26	1	8	19.167	0.008	0.067	0.008	6.383	17.500	36.001	0.017	104.002	0.083	5.533
Soln Std		STD-B1-2	1	9	19.900	0.005	0.040	0.005	4.590	9.440	82.000	0.010	48.200	0.050	0.100
EA		J16	1	10	590.001	0.083	0.667	0.083	0.033	173.334	1628.337	0.167	838.335	0.833	1.667
NEPHB-03ccc	ccc	J29	1	11	18.000	0.008	0.067	0.008	4.517	15.934	49.334	0.017	92.002	0.083	2.567
NEPHB-02ccc	ccc	J04	1	12	18.167	0.008	0.067	0.008	5.000	16.300	39.834	0.017	98.169	0.083	3.267
NEPHB-01	quenched	J24	1	13	19.334	0.008	0.067	0.008	7.200	18.834	31.834	0.017	107.669	0.083	6.133
NEPHB-02	quenched	J30	1	14	18.500	0.008	0.067	0.008	5.217	17.334	39.834	0.017	99.335	0.083	3.533
NEPHB-05ccc	ccc	J12	1	15	16.450	0.008	0.067	0.008	2.733	14.767	70.335	0.017	77.502	0.083	1.767
Soln Std		STD-B1-3	1	16	19.800	0.005	0.040	0.005	4.470	9.290	82.600	0.010	47.500	0.050	0.100
Soln Std		STD-B2-1	2	1	19.500	0.005	0.040	0.005	4.470	9.710	81.500	0.010	49.300	0.050	0.100
NEPHB-03ccc	ccc	J11	2	2	18.000	0.008	0.067	0.008	4.583	16.050	51.168	0.017	91.835	0.083	2.450
NEPHB-01ccc	ccc	J36	2	3	19.167	0.008	0.067	0.008	6.200	17.667	33.834	0.017	104.669	0.083	5.450
EA		J31	2	4	510.001	0.083	0.667	0.083	0.033	151.167	1403.336	0.167	748.335	0.833	1.667
NEPHB-05ccc	ccc	J06	2	5	18.334	0.008	0.067	0.008	3.650	15.567	76.835	0.017	84.168	0.083	2.033
ARM-1		J14	2	6	19.334	0.008	0.067	0.008	0.003	14.034	38.501	0.017	61.501	0.083	0.167
NEPHB-03	quenched	J35	2	7	18.334	0.008	0.067	0.008	4.700	16.084	51.668	0.017	90.668	0.083	2.383
NEPHB-02	quenched	J32	2	8	18.667	0.008	0.067	0.008	5.033	17.334	41.001	0.017	98.502	0.083	3.467
Soln Std		STD-B2-2	2	9	19.900	0.005	0.040	0.005	4.640	9.480	83.900	0.010	48.400	0.050	0.100
NEPHB-05	quenched	J10	2	10	19.334	0.008	0.067	0.008	5.367	15.284	86.668	0.017	85.168	0.083	2.183
NEPHB-04ccc	ccc	J08	2	11	30.667	0.008	0.067	0.008	5.133	23.500	84.502	0.017	99.335	0.083	2.450
NEPHB-04	quenched	J38	2	12	18.834	0.008	0.067	0.008	4.350	15.634	67.835	0.017	86.668	0.083	2.100
NEPHB-02ccc	ccc	J13	2	13	18.167	0.008	0.067	0.008	4.900	16.317	42.668	0.017	97.169	0.083	3.250
NEPHB-01	quenched	J05	2	14	19.000	0.008	0.067	0.008	7.083	18.500	32.834	0.017	106.335	0.083	5.983
Soln Std		STD-B2-3	2	15	19.800	0.005	0.040	0.005	4.500	9.600	84.000	0.010	48.700	0.050	0.100
Soln Std		STD-B3-1	3	1	19.600	0.005	0.040	0.005	4.500	9.720	80.400	0.010	51.000	0.050	0.100

Table E2. PSAL Measurements of the PCT Solutions for the Study Glasses After Appropriate Adjustments (continued)

Glass ID	Heat Treatment	Laboratory ID	Block	Seq	B (ppm)	Ba (ppm)	Cd (ppm)	Cr (ppm)	Fe (ppm)	Li (ppm)	Na (ppm)	Pb (ppm)	Si (ppm)	Th (ppm)	U (ppm)
NEPHB-01ccc	ccc	J17	3	2	19.000	0.008	0.067	0.008	6.100	17.834	32.167	0.017	107.002	0.083	5.283
NEPHB-02ccc	ccc	J22	3	3	18.167	0.008	0.067	0.008	4.900	16.667	40.667	0.017	102.002	0.083	3.117
NEPHB-05	quenched	J20	3	4	18.667	0.008	0.067	0.008	5.233	14.967	81.835	0.017	86.502	0.083	2.167
NEPHB-02	quenched	J07	3	5	18.167	0.008	0.067	0.008	5.050	17.334	39.001	0.017	100.502	0.083	3.150
ARM-1		J19	3	6	19.334	0.008	0.067	0.008	0.003	14.367	38.667	0.017	63.001	0.083	0.167
NEPHB-05ccc	ccc	J27	3	7	17.834	0.008	0.067	0.008	3.683	16.084	79.335	0.017	89.168	0.083	2.000
NEPHB-03ccc	ccc	J03	3	8	18.334	0.008	0.067	0.008	4.583	16.167	50.001	0.017	96.502	0.083	2.150
Soln Std		STD-B3-2	3	9	20.100	0.005	0.040	0.005	4.660	9.540	81.800	0.010	49.500	0.050	0.100
NEPHB-04	quenched	J33	3	10	18.500	0.008	0.067	0.008	3.983	15.650	65.668	0.017	90.168	0.083	2.183
blank		J21	3	11	0.193	0.008	0.067	0.008	0.003	0.417	0.083	0.017	0.083	0.083	0.167
NEPHB-04ccc	ccc	J09	3	12	30.501	0.008	0.067	0.008	5.300	23.834	82.168	0.017	104.169	0.083	2.450
EA		J23	3	13	348.334	0.083	0.667	0.083	0.033	108.000	1018.335	0.167	588.335	0.833	1.667
NEPHB-01	quenched	J18	3	14	19.834	0.008	0.067	0.008	7.283	19.000	33.501	0.017	112.169	0.083	5.817
NEPHB-03	quenched	J01	3	15	18.500	0.008	0.067	0.008	5.017	16.284	49.501	0.017	96.169	0.083	2.500
Soln Std		STD-B3-3	3	16	20.100	0.005	0.040	0.005	4.620	9.600	81.000	0.010	50.700	0.050	0.100

Table E3. Laboratory Measurements As-Received of the PCT Solutions for the Re-Tested ccc Glasses

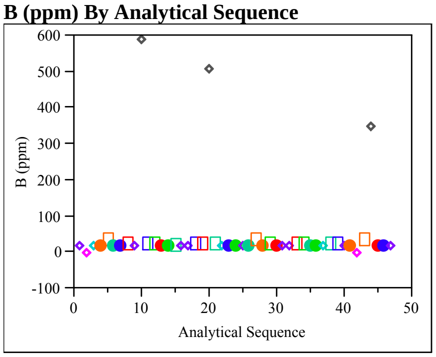
Glass ID	Heat Treatment	Laboratory ID	Block	Seq	B ar	Ba ar	Cd ar	Cr ar	Fe ar	Li ar	Na ar	Pb ar	Si ar	Th ar	U ar
Soln Std		STD-B1-1	4	1	21.2	<0.010	<0.080	<0.010	4.04	10.0	79.6	<0.020	52.6	<0.100	<0.200
EA		K05	4	2	22.8	<0.010	<0.080	<0.010	<0.040	8.01	62.1	<0.020	38.1	<0.100	<0.200
NEPHB-01ccc	ccc	K17	4	3	11.9	<0.010	<0.080	<0.010	4.04	11.1	19.2	<0.020	62.5	<0.100	3.35
NEPHB-02ccc	ccc	K22	4	4	11.5	<0.010	<0.080	<0.010	3.72	10.4	23.2	<0.020	58.5	<0.100	1.95
ARM-1		K18	4	5	11.3	<0.010	<0.080	<0.010	<0.040	9.51	23.7	<0.020	39.5	<0.100	<0.200
Soln Std		STD-B1-2	4	6	20.9	<0.010	<0.080	<0.010	4.15	10.7	78.6	<0.020	53.4	<0.100	<0.200
blank		K08	4	7	<0.100	<0.010	<0.080	<0.010	<0.040	<0.500	<0.100	<0.020	<0.100	<0.100	<0.200
NEPHB-04ccc	ccc	K21	4	8	22.2	<0.010	<0.080	<0.010	3.94	18.6	50.7	<0.020	61.8	<0.100	1.50
NEPHB-03ccc	ccc	K23	4	9	11.2	<0.010	<0.080	<0.010	3.00	9.92	28.1	<0.020	54.4	<0.100	1.52
NEPHB-05ccc	ccc	K13	4	10	10.8	<0.010	<0.080	<0.010	1.89	9.81	42.6	<0.020	49.7	<0.100	1.12
Soln Std		STD-B1-3	4	11	20.9	<0.010	<0.080	<0.010	4.38	10.4	78.1	<0.020	51.9	<0.100	<0.200
Soln Std		STD-B2-1	5	1	21.9	<0.010	<0.080	<0.010	4.47	9.86	77.4	<0.020	52.2	<0.100	<0.200
NEPHB-03ccc	ccc	K16	5	2	11.9	<0.010	<0.080	<0.010	3.36	9.88	29.9	<0.020	53.6	<0.100	1.44
ARM-1		K02	5	3	13.5	<0.010	<0.080	<0.010	<0.040	9.48	23.8	<0.020	38.5	<0.100	<0.200
NEPHB-01ccc	ccc	K01	5	4	11.8	<0.010	<0.080	<0.010	4.10	10.7	18.7	<0.020	59.2	<0.100	3.09
NEPHB-02ccc	ccc	K06	5	5	11.3	<0.010	<0.080	<0.010	3.63	10.2	23.7	<0.020	56.1	<0.100	1.80
Soln Std		STD-B2-2	5	6	20.9	<0.010	<0.080	<0.010	4.49	9.73	80.5	<0.020	51.4	<0.100	<0.200
NEPHB-04ccc	ccc	K09	5	7	22.4	<0.010	<0.080	<0.010	4.07	17.7	51.8	<0.020	58.5	<0.100	1.46
EA		K15	5	8	22.8	<0.010	<0.080	<0.010	<0.040	7.73	62.4	<0.020	36.6	<0.100	<0.200
NEPHB-05ccc	ccc	K12	5	9	10.7	<0.010	<0.080	<0.010	2.18	9.40	45.6	<0.020	46.7	<0.100	1.08
Soln Std		STD-B2-3	5	10	20.7	<0.010	<0.080	<0.010	4.30	9.55	82.2	<0.020	49.8	<0.100	<0.200
Soln Std		STD-B3-1	6	1	21.4	<0.010	<0.080	<0.010	4.18	9.79	83.0	<0.020	52.4	<0.100	<0.200
NEPHB-04ccc	ccc	K03	6	2	23.8	<0.010	<0.080	<0.010	6.32	18.2	53.9	<0.020	60.8	<0.100	1.64
NEPHB-02ccc	ccc	K10	6	3	11.7	<0.010	<0.080	<0.010	3.43	10.3	23.2	<0.020	56.8	<0.100	1.81
EA		K20	6	4	36.2	<0.010	<0.080	<0.010	<0.040	11.3	94.9	<0.020	48.6	<0.100	<0.200
NEPHB-03ccc	ccc	K11	6	5	11.1	<0.010	<0.080	<0.010	3.07	9.94	29.9	<0.020	53.8	<0.100	1.37
Soln Std		STD-B-3-2	6	6	21.2	<0.010	<0.080	<0.010	4.49	9.87	81.4	<0.020	53.0	<0.100	<0.200
ARM-1		K04	6	7	14.0	<0.010	<0.080	<0.010	<0.040	10.0	27.1	<0.020	40.3	<0.100	<0.200
blank		K19	6	8	<0.100	<0.010	<0.080	<0.010	<0.040	<0.500	<0.100	<0.020	<0.100	<0.100	<0.200
NEPHB-05ccc	ccc	K07	6	9	10.2	<0.010	<0.080	<0.010	2.32	9.56	44.8	<0.020	48.6	<0.100	1.15
NEPHB-01ccc	ccc	K14	6	10	11.3	<0.010	<0.080	<0.010	4.41	11.4	19.8	<0.020	62.8	<0.100	3.18
Soln Std		STD-B3-3	6	11	21.0	<0.010	<0.080	<0.010	4.47	9.85	82.2	<0.020	52.5	<0.100	<0.200

Table E4. Laboratory Measurements after Adjustments of the PCT Solutions for the Re-Tested ccc Glasses

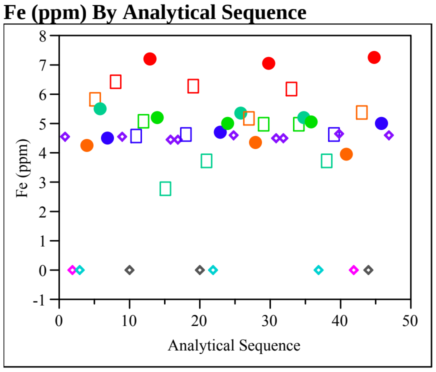
Glass ID	Heat Treatment	Laboratory ID	Block	Seq	B (ppm)	Ba (ppm)	Cd (ppm)	Cr (ppm)	Fe (ppm)	Li (ppm)	Na (ppm)	Pb (ppm)	Si (ppm)	Th (ppm)	U (ppm)
Soln Std		STD-B1-1	4	1	21.200	0.005	0.040	0.005	4.040	10.000	79.600	0.010	52.600	0.050	0.100
EA		K05	4	2	380.001	0.083	0.667	0.083	0.333	133.500	1035.002	0.167	635.001	0.833	1.667
NEPHB-01ccc	ccc	K17	4	3	19.834	0.008	0.067	0.008	6.733	18.500	32.001	0.017	104.169	0.083	5.583
NEPHB-02ccc	ccc	K22	4	4	19.167	0.008	0.067	0.008	6.200	17.334	38.667	0.017	97.502	0.083	3.250
ARM-1		K18	4	5	18.834	0.008	0.067	0.008	0.033	15.850	39.501	0.017	65.835	0.083	0.167
Soln Std		STD-B1-2	4	6	20.900	0.005	0.040	0.005	4.150	10.700	78.600	0.010	53.400	0.050	0.100
blank		K08	4	7	0.083	0.008	0.067	0.008	0.033	0.417	0.083	0.017	0.083	0.083	0.167
NEPHB-04ccc	ccc	K21	4	8	37.001	0.008	0.067	0.008	6.567	31.001	84.502	0.017	103.002	0.083	2.500
NEPHB-03ccc	ccc	K23	4	9	18.667	0.008	0.067	0.008	5.000	16.534	46.834	0.017	90.668	0.083	2.533
NEPHB-05ccc	ccc	K13	4	10	18.000	0.008	0.067	0.008	3.150	16.350	71.001	0.017	82.835	0.083	1.867
Soln Std		STD-B1-3	4	11	20.900	0.005	0.040	0.005	4.380	10.400	78.100	0.010	51.900	0.050	0.100
Soln Std		STD-B2-1	5	1	21.900	0.005	0.040	0.005	4.470	9.860	77.400	0.010	52.200	0.050	0.100
NEPHB-03ccc	ccc	K16	5	2	19.834	0.008	0.067	0.008	5.600	16.467	49.834	0.017	89.335	0.083	2.400
ARM-1		K02	5	3	22.500	0.008	0.067	0.008	0.033	15.800	39.667	0.017	64.168	0.083	0.167
NEPHB-01ccc	ccc	K01	5	4	19.667	0.008	0.067	0.008	6.833	17.834	31.167	0.017	98.669	0.083	5.150
NEPHB-02ccc	ccc	K06	5	5	18.834	0.008	0.067	0.008	6.050	17.000	39.501	0.017	93.502	0.083	3.000
Soln Std		STD-B2-2	5	6	20.900	0.005	0.040	0.005	4.490	9.730	80.500	0.010	51.400	0.050	0.100
NEPHB-04ccc	ccc	K09	5	7	37.334	0.008	0.067	0.008	6.783	29.501	86.335	0.017	97.502	0.083	2.433
EA		K15	5	8	380.001	0.083	0.667	0.083	0.333	128.834	1040.002	0.167	610.001	0.833	1.667
NEPHB-05ccc	ccc	K12	5	9	17.834	0.008	0.067	0.008	3.633	15.667	76.002	0.017	77.835	0.083	1.800
Soln Std		STD-B2-3	5	10	20.700	0.005	0.040	0.005	4.300	9.550	82.200	0.010	49.800	0.050	0.100
Soln Std		STD-B3-1	6	1	21.400	0.005	0.040	0.005	4.180	9.790	83.000	0.010	52.400	0.050	0.100
NEPHB-04ccc	ccc	K03	6	2	39.667	0.008	0.067	0.008	10.534	30.334	89.835	0.017	101.335	0.083	2.733
NEPHB-02ccc	ccc	K10	6	3	19.500	0.008	0.067	0.008	5.717	17.167	38.667	0.017	94.669	0.083	3.017
EA		K20	6	4	603.335	0.083	0.667	0.083	0.333	188.334	1581.670	0.167	810.002	0.833	1.667
NEPHB-03ccc	ccc	K11	6	5	18.500	0.008	0.067	0.008	5.117	16.567	49.834	0.017	89.668	0.083	2.283
Soln Std		STD-B-3-2	6	6	21.200	0.005	0.040	0.005	4.490	9.870	81.400	0.010	53.000	0.050	0.100
ARM-1		K04	6	7	23.334	0.008	0.067	0.008	0.033	16.667	45.168	0.017	67.168	0.083	0.167
blank		K19	6	8	0.083	0.008	0.067	0.008	0.033	0.417	0.083	0.017	0.083	0.083	0.167
NEPHB-05ccc	ccc	K07	6	9	17.000	0.008	0.067	0.008	3.867	15.934	74.668	0.017	81.002	0.083	1.917
NEPHB-01ccc	ccc	K14	6	10	18.834	0.008	0.067	0.008	7.350	19.000	33.001	0.017	104.669	0.083	5.300
Soln Std		STD-B3-3	6	11	21.000	0.005	0.040	0.005	4.470	9.850	82.200	0.010	52.500	0.050	0.100

Exhibit E1. Laboratory PCT Measurements in Analytical Sequence for Study Glasses, EA, ARM, Blanks, and Solution Standards

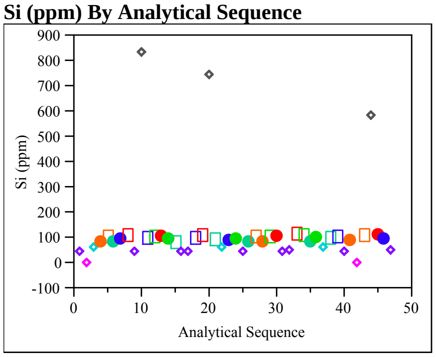
B (ppm) By Analytical Sequence



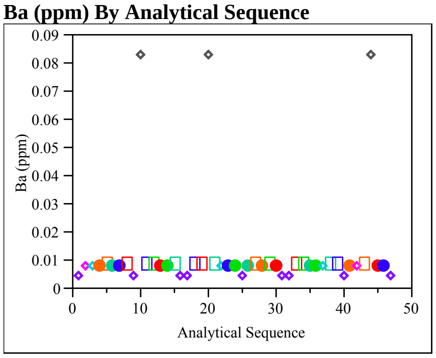
Fe (ppm) By Analytical Sequence



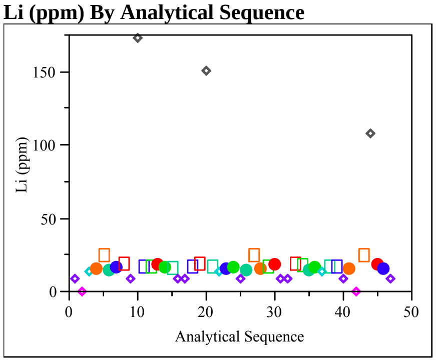
Si (ppm) By Analytical Sequence



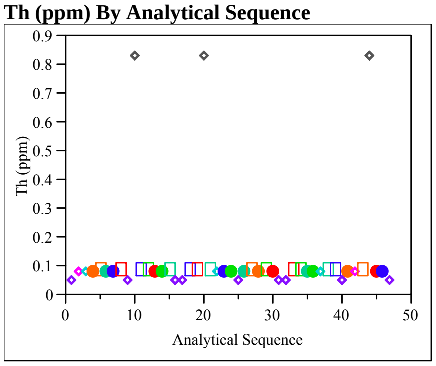
Ba (ppm) By Analytical Sequence



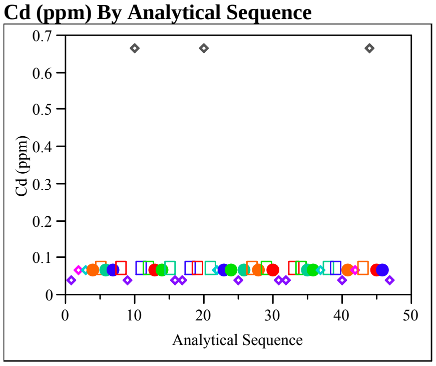
Li (ppm) By Analytical Sequence



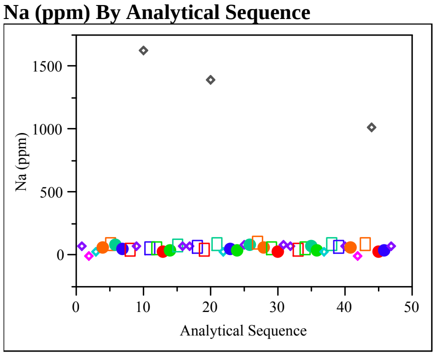
Th (ppm) By Analytical Sequence



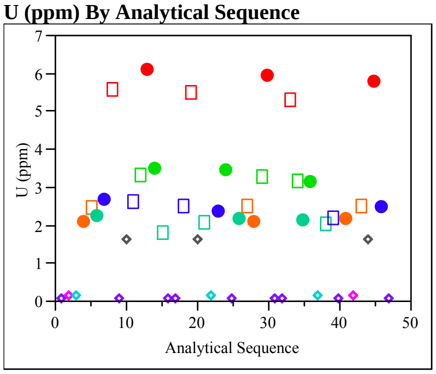
Cd (ppm) By Analytical Sequence



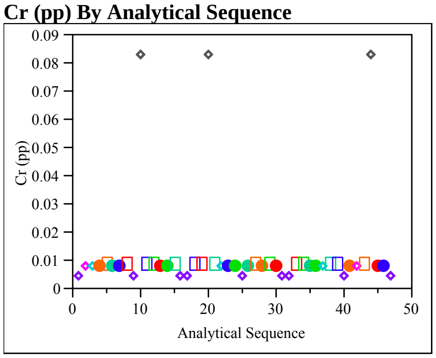
Na (ppm) By Analytical Sequence



U (ppm) By Analytical Sequence



Cr (pp) By Analytical Sequence



Pb (ppm) By Analytical Sequence

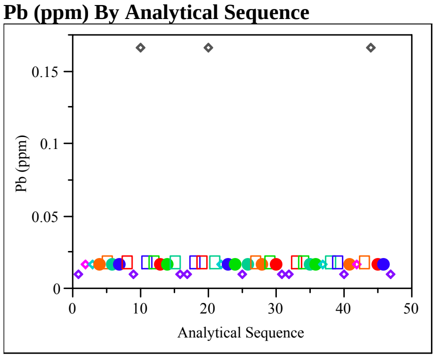
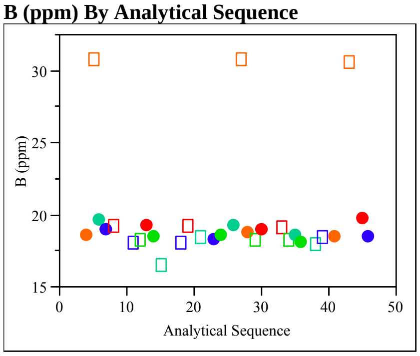
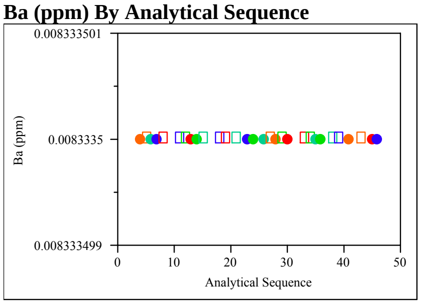


Exhibit E2. Laboratory PCT Measurements in Analytical Sequence for Study Glasses

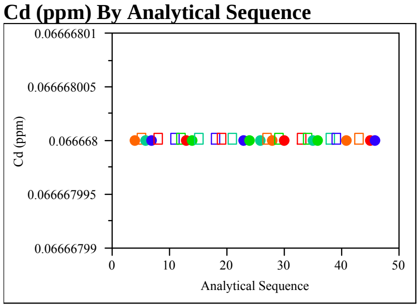
B (ppm) By Analytical Sequence



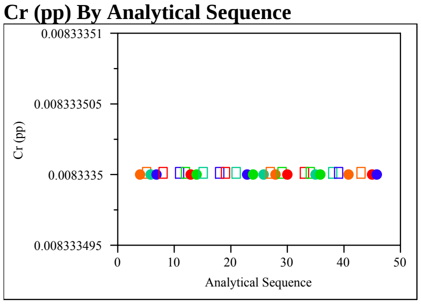
Ba (ppm) By Analytical Sequence



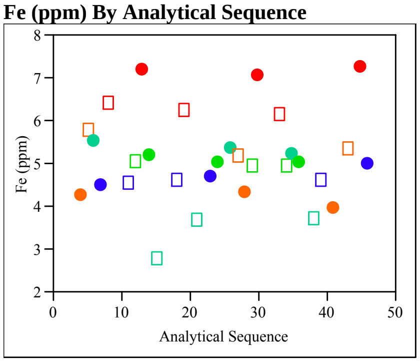
Cd (ppm) By Analytical Sequence



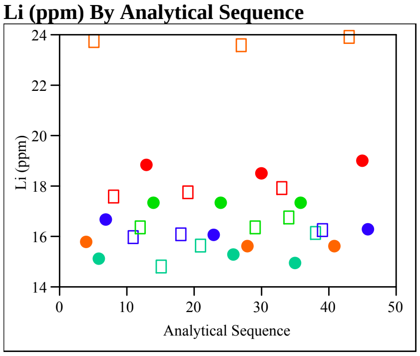
Cr (pp) By Analytical Sequence



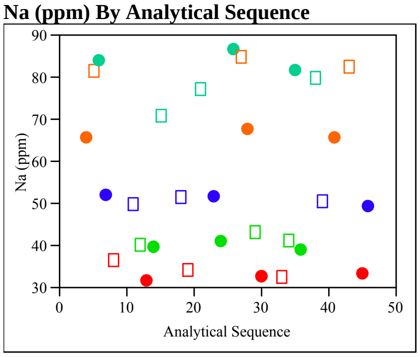
Fe (ppm) By Analytical Sequence



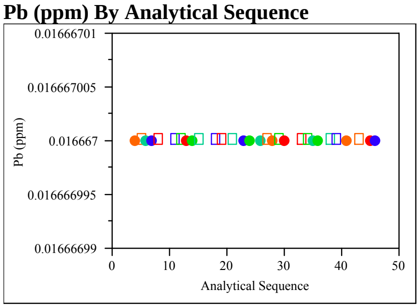
Li (ppm) By Analytical Sequence



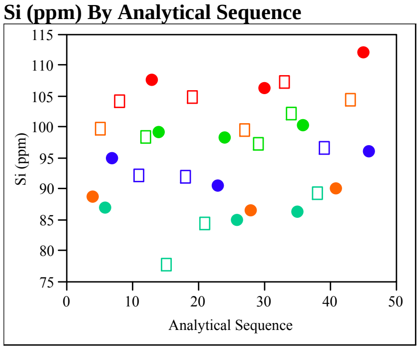
Na (ppm) By Analytical Sequence



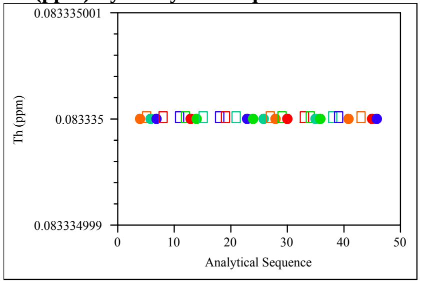
Pb (ppm) By Analytical Sequence



Si (ppm) By Analytical Sequence



Th (ppm) By Analytical Sequence



U (ppm) By Analytical Sequence

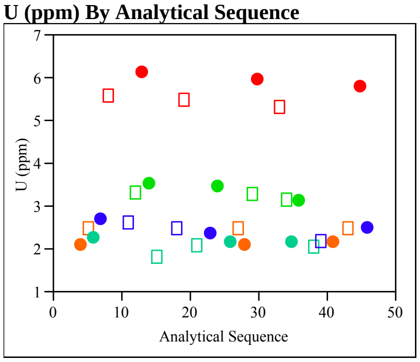
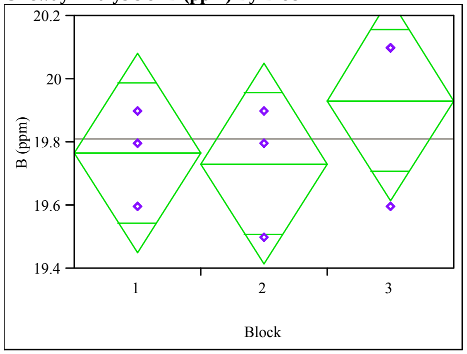


Exhibit E3. Measurements of the Multi-Element Solution Standard by ICP-AES Block

Oneway Analysis of B (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0.186747
Adj Rsquare	-0.08434
Root Mean Square Error	0.223607
Mean of Response	19.81111
Observations (or Sum Wgts)	9

Analysis of Variance

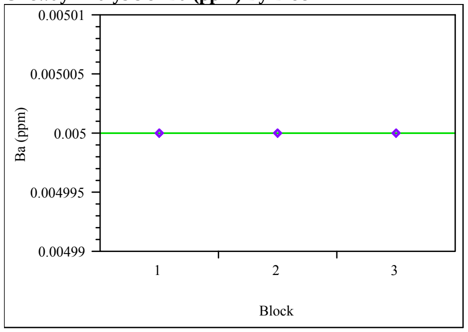
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0.06888889	0.034444	0.6889	0.5379
Error	6	0.30000000	0.050000		
C. Total	8	0.36888889			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	19.7667	0.12910	19.451	20.083
2	3	19.7333	0.12910	19.417	20.049
3	3	19.9333	0.12910	19.617	20.249

Std Error uses a pooled estimate of error variance

Oneway Analysis of Ba (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.005
Observations (or Sum Wgts)	9

Analysis of Variance

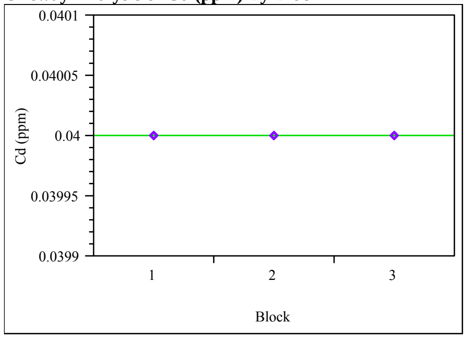
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0	0	.	.
Error	6	0	0		
C. Total	8	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.005000	0	0.00500	0.00500
2	3	0.005000	0	0.00500	0.00500
3	3	0.005000	0	0.00500	0.00500

Std Error uses a pooled estimate of error variance

Oneway Analysis of Cd (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.04
Observations (or Sum Wgts)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0	0	.	.
Error	6	0	0		
C. Total	8	0			

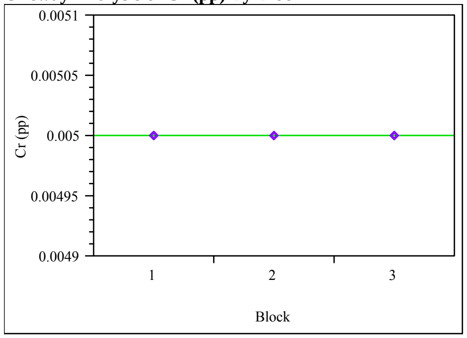
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.040000	0	0.04000	0.04000
2	3	0.040000	0	0.04000	0.04000
3	3	0.040000	0	0.04000	0.04000

Std Error uses a pooled estimate of error variance

Exhibit E3. Measurements of the Multi-Element Solution Standard by ICP-AES Block (continued)

Oneway Analysis of Cr (pp) By Block



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.005
Observations (or Sum Wgts)	9

Analysis of Variance

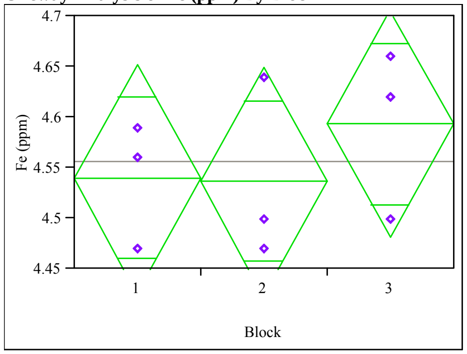
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0	0	.	.
Error	6	0	0		
C. Total	8	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.005000	0	0.00500	0.00500
2	3	0.005000	0	0.00500	0.00500
3	3	0.005000	0	0.00500	0.00500

Std Error uses a pooled estimate of error variance

Oneway Analysis of Fe (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0.137255
Adj Rsquare	-0.15033
Root Mean Square Error	0.079722
Mean of Response	4.556667
Observations (or Sum Wgts)	9

Analysis of Variance

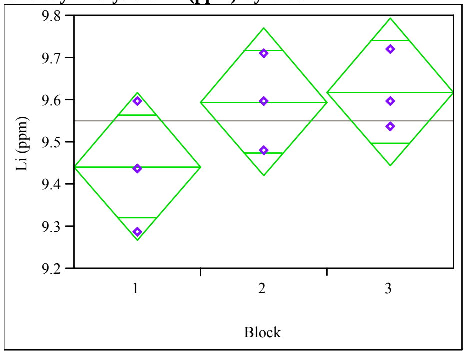
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0.00606667	0.003033	0.4773	0.6422
Error	6	0.03813333	0.006356		
C. Total	8	0.04420000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	4.54000	0.04603	4.4274	4.6526
2	3	4.53667	0.04603	4.4240	4.6493
3	3	4.59333	0.04603	4.4807	4.7060

Std Error uses a pooled estimate of error variance

Oneway Analysis of Li (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0.37699
Adj Rsquare	0.169319
Root Mean Square Error	0.123378
Mean of Response	9.553333
Observations (or Sum Wgts)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0.05526667	0.027633	1.8153	0.2418
Error	6	0.09133333	0.015222		
C. Total	8	0.14660000			

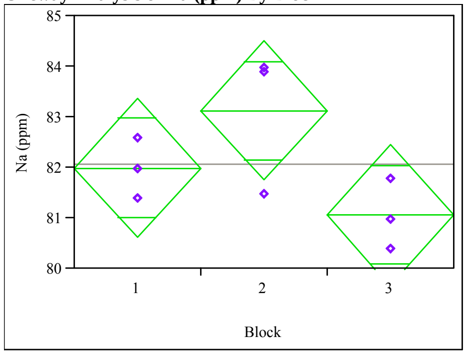
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	9.44333	0.07123	9.2690	9.6176
2	3	9.59667	0.07123	9.4224	9.7710
3	3	9.62000	0.07123	9.4457	9.7943

Std Error uses a pooled estimate of error variance

Exhibit E3. Measurements of the Multi-Element Solution Standard by ICP-AES Block (continued)

Oneway Analysis of Na (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0.529379
Adj Rsquare	0.372506
Root Mean Square Error	0.975819
Mean of Response	82.06667
Observations (or Sum Wgts)	9

Analysis of Variance

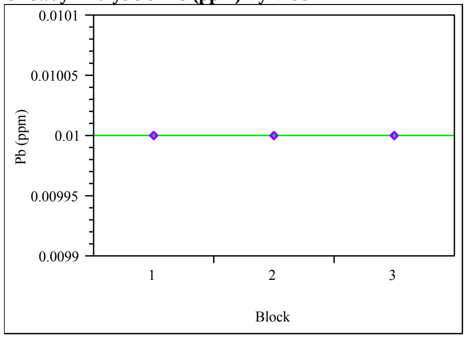
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	6.426667	3.21333	3.3746	0.1042
Error	6	5.713333	0.95222		
C. Total	8	12.140000			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	82.0000	0.56339	80.621	83.379
2	3	83.1333	0.56339	81.755	84.512
3	3	81.0667	0.56339	79.688	82.445

Std Error uses a pooled estimate of error variance

Oneway Analysis of Pb (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	.
Adj Rsquare	.
Root Mean Square Error	0
Mean of Response	0.01
Observations (or Sum Wgts)	9

Analysis of Variance

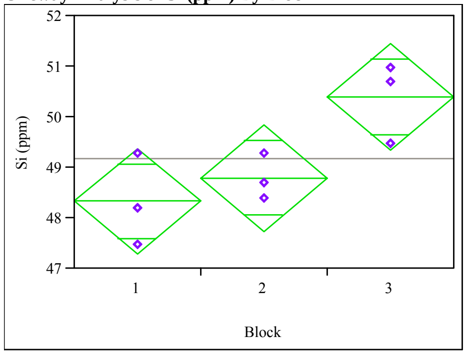
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0	0	.	.
Error	6	0	0		
C. Total	8	0			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.010000	0	0.01000	0.01000
2	3	0.010000	0	0.01000	0.01000
3	3	0.010000	0	0.01000	0.01000

Std Error uses a pooled estimate of error variance

Oneway Analysis of Si (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0.679375
Adj Rsquare	0.572499
Root Mean Square Error	0.74461
Mean of Response	49.17778
Observations (or Sum Wgts)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	7.048889	3.52444	6.3567	0.0330
Error	6	3.326667	0.55444		
C. Total	8	10.375556			

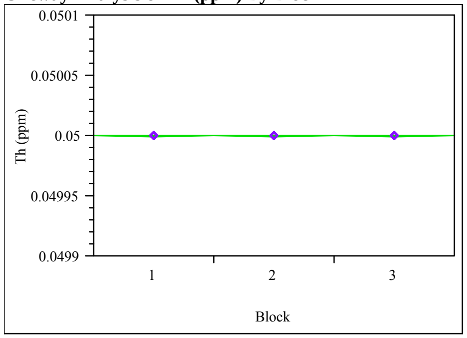
Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	48.3333	0.42990	47.281	49.385
2	3	48.8000	0.42990	47.748	49.852
3	3	50.4000	0.42990	49.348	51.452

Std Error uses a pooled estimate of error variance

Exhibit E3. Measurements of the Multi-Element Solution Standard by ICP-AES Block (continued)

Oneway Analysis of Th (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	-0.33333
Root Mean Square Error	8.5e-18
Mean of Response	0.05
Observations (or Sum Wgts)	9

Analysis of Variance

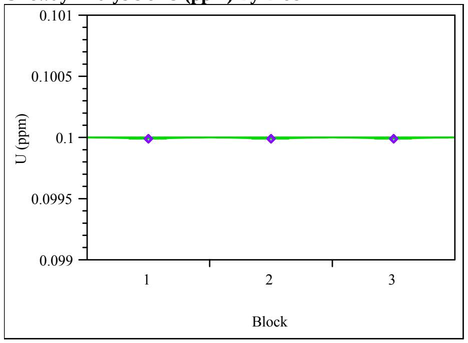
Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0	0	0.0000	1.0000
Error	6	4.3333e-34	7.222e-35		
C. Total	8	4.3333e-34			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.050000	4.907e-18	0.05000	0.05000
2	3	0.050000	4.907e-18	0.05000	0.05000
3	3	0.050000	4.907e-18	0.05000	0.05000

Std Error uses a pooled estimate of error variance

Oneway Analysis of U (ppm) By Block



Oneway Anova
Summary of Fit

Rsquare	0
Adj Rsquare	-0.33333
Root Mean Square Error	1.7e-17
Mean of Response	0.1
Observations (or Sum Wgts)	9

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Block	2	0	0	0.0000	1.0000
Error	6	1.7333e-33	2.889e-34		
C. Total	8	1.7333e-33			

Means for Oneway Anova

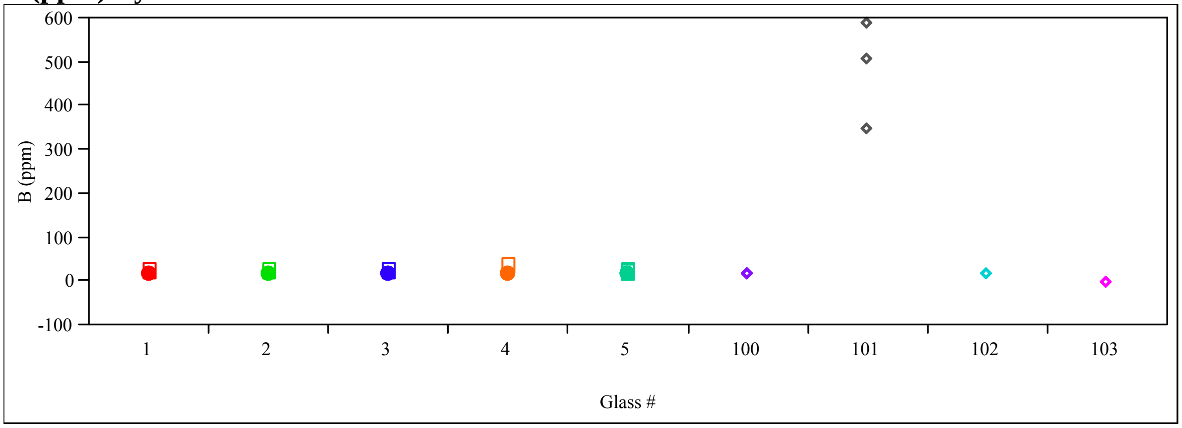
Level	Number	Mean	Std Error	Lower 95%	Upper 95%
1	3	0.100000	9.813e-18	0.10000	0.10000
2	3	0.100000	9.813e-18	0.10000	0.10000
3	3	0.100000	9.813e-18	0.10000	0.10000

Std Error uses a pooled estimate of error variance

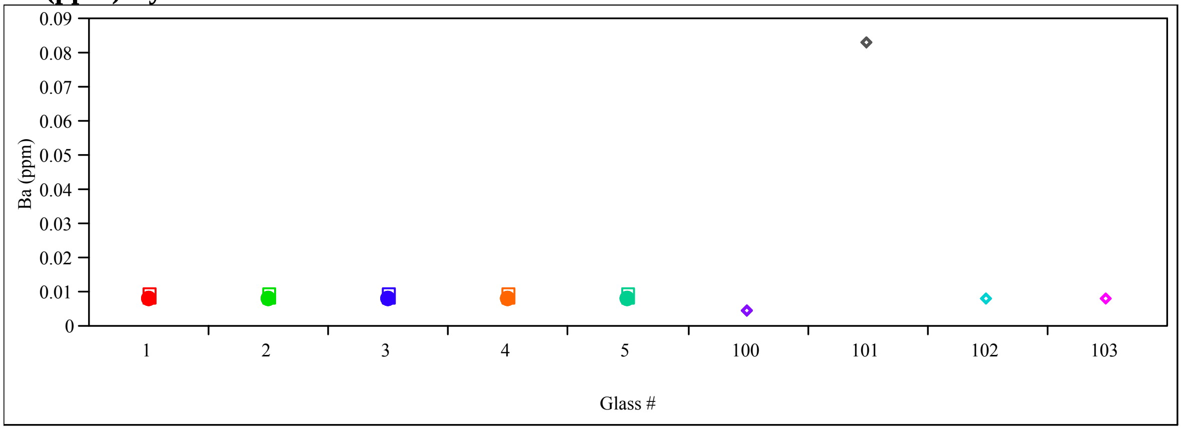
Exhibit E4. Laboratory PCT Measurements by Glass Number for Study Glasses and Standards

(100 – Solution Standard; 101 – EA; 102 – ARM; 103 – Blanks)

B (ppm) By Glass



Ba (ppm) By Glass



Cd (ppm) By Glass

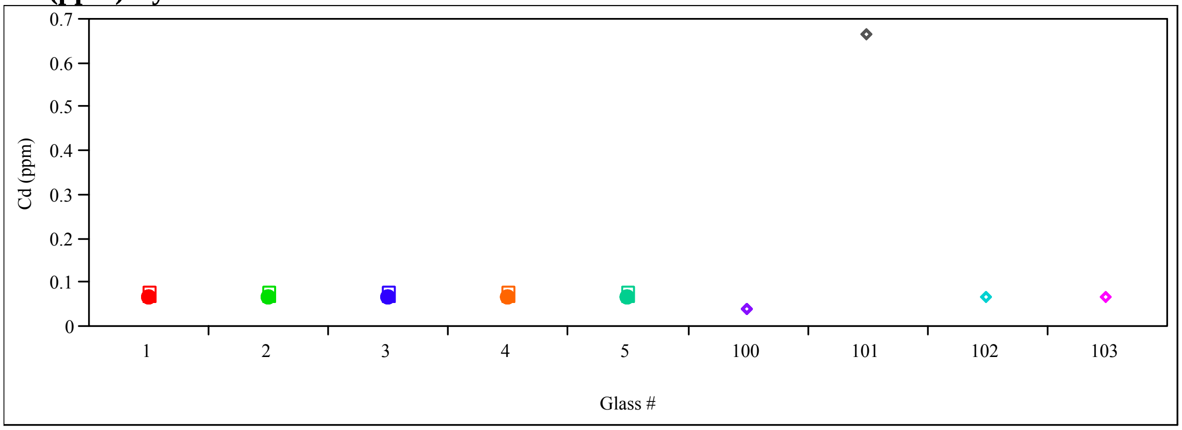
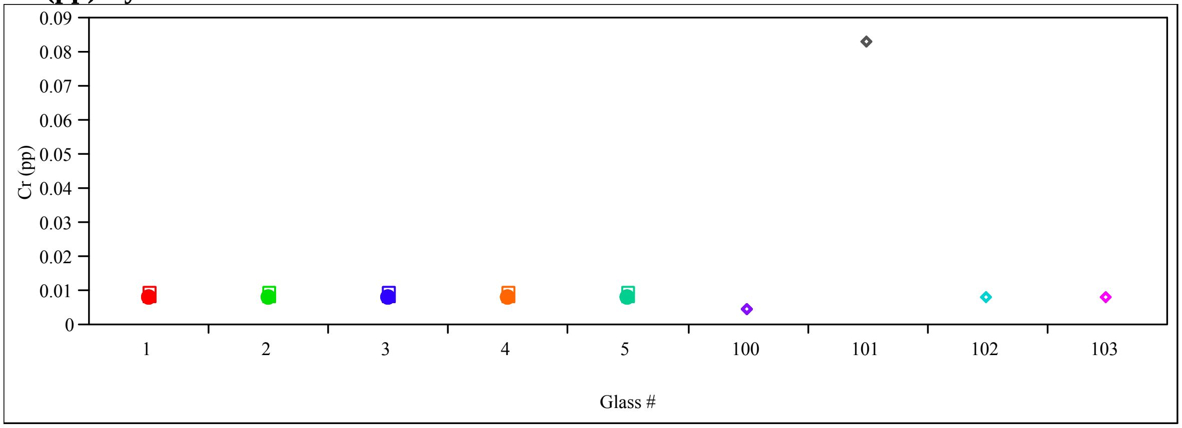


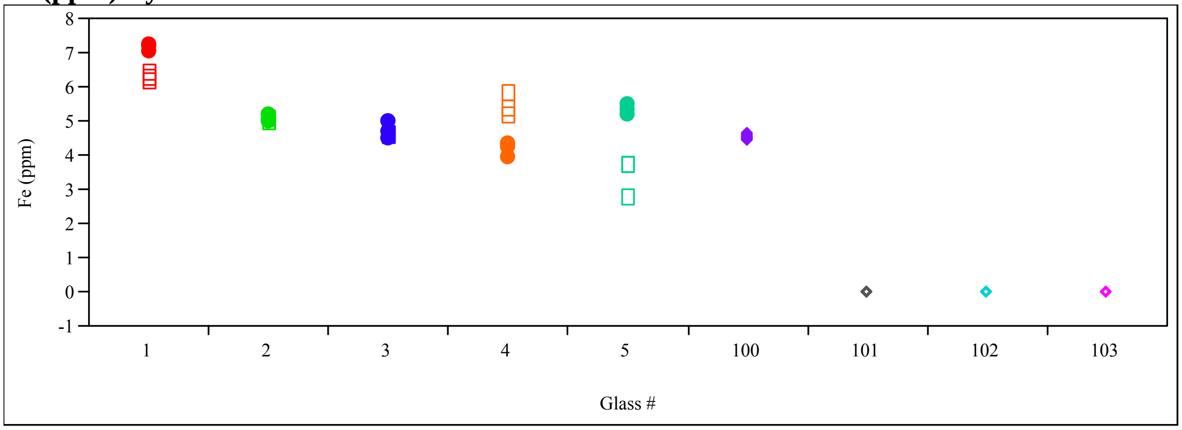
Exhibit E4. Laboratory PCT Measurements by Glass Number for Study Glasses and Standards (continued)

(100 – Solution Standard; 101 – EA; 102 – ARM; 103 – Blanks)

Cr (pp) By Glass



Fe (ppm) By Glass



Li (ppm) By Glass

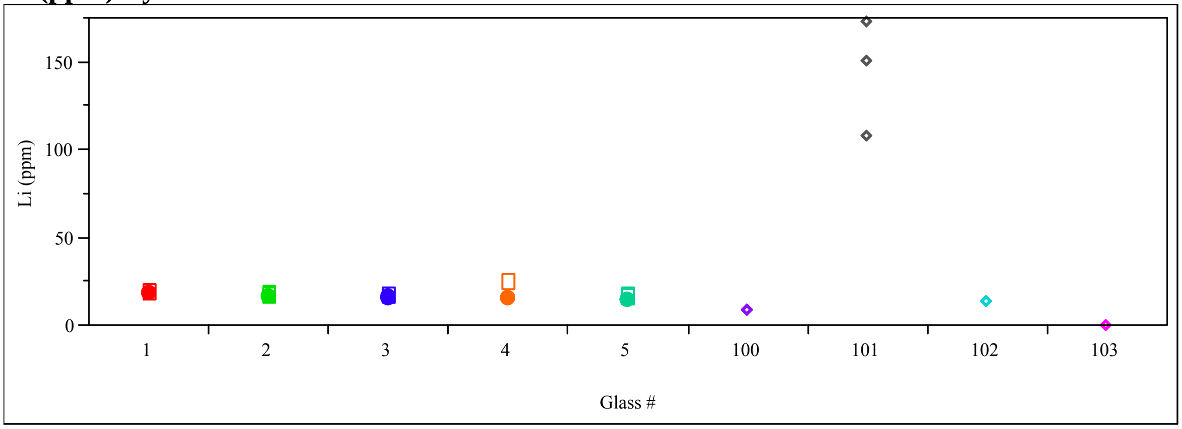
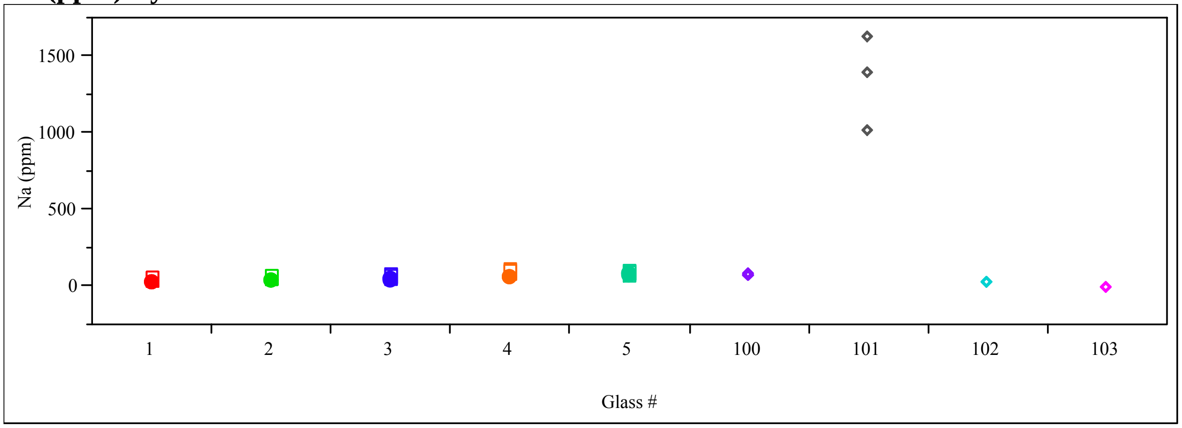


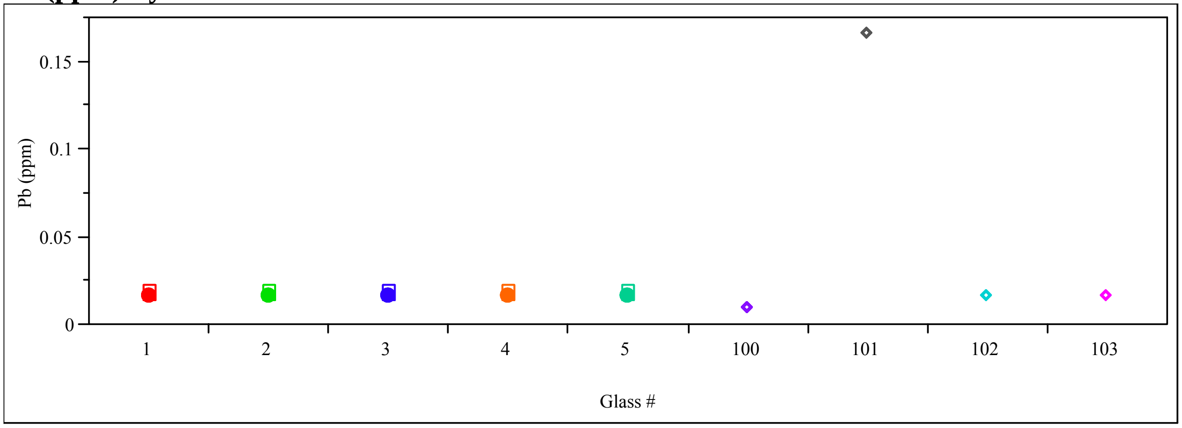
Exhibit E4. Laboratory PCT Measurements by Glass Number for Study Glasses and Standards (continued)

(100 – Solution Standard; 101 – EA; 102 – ARM; 103 – Blanks)

Na (ppm) By Glass



Pb (ppm) By Glass



Si (ppm) By Glass

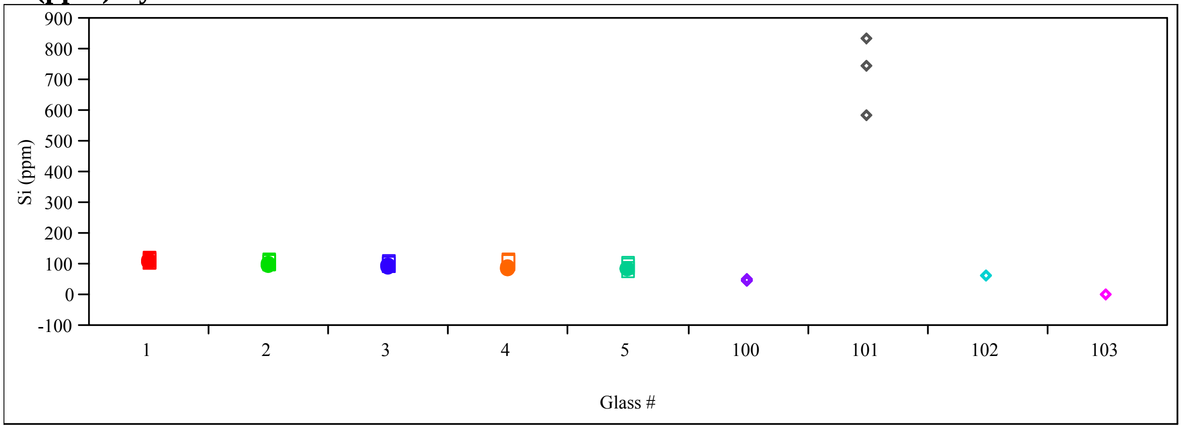
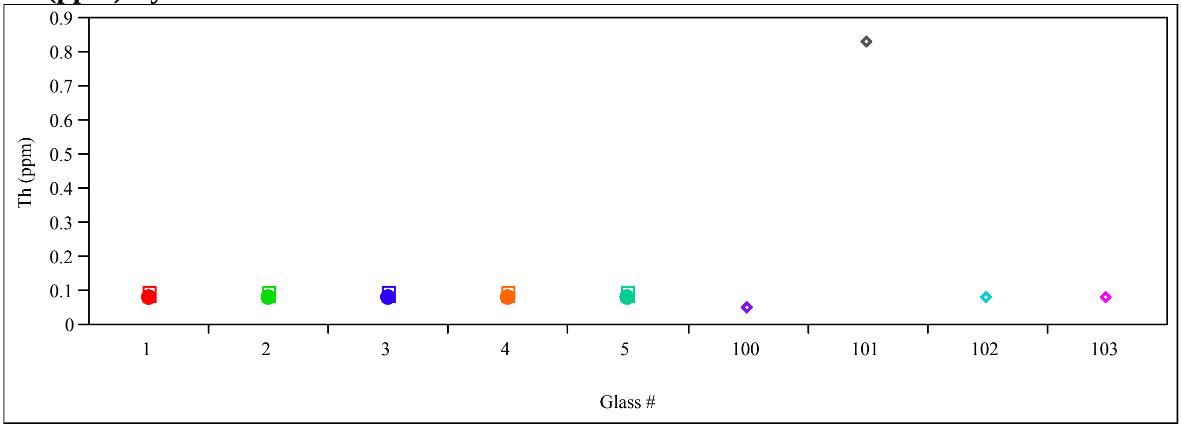


Exhibit E4. Laboratory PCT Measurements by Glass Number for Study Glasses and Standards (continued)

(100 – Solution Standard; 101 – EA; 102 – ARM; 103 – Blanks)

Th (ppm) By Glass



U (ppm) By Glass

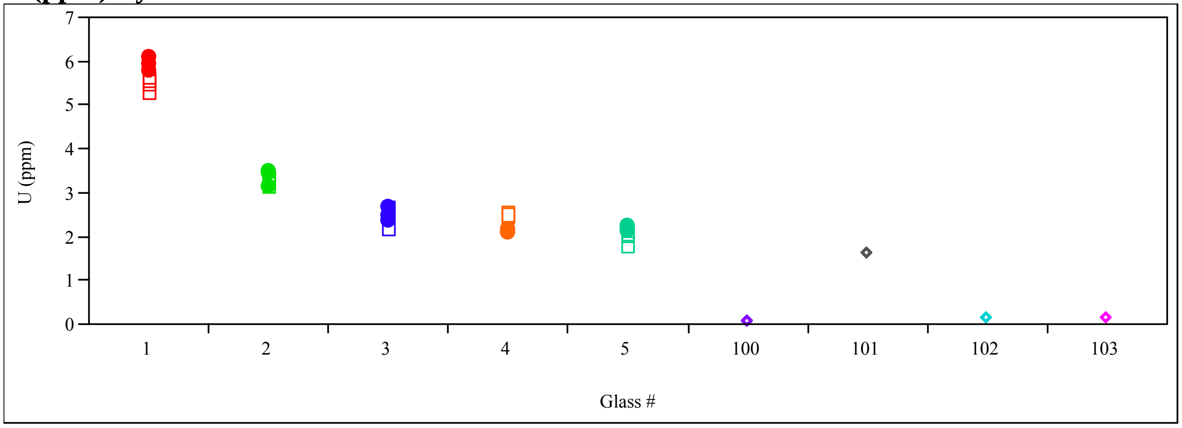
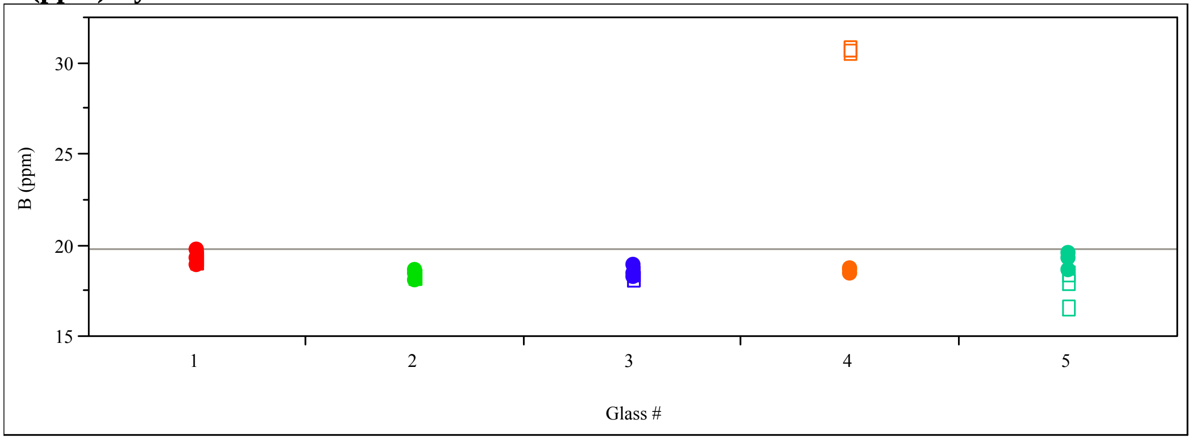
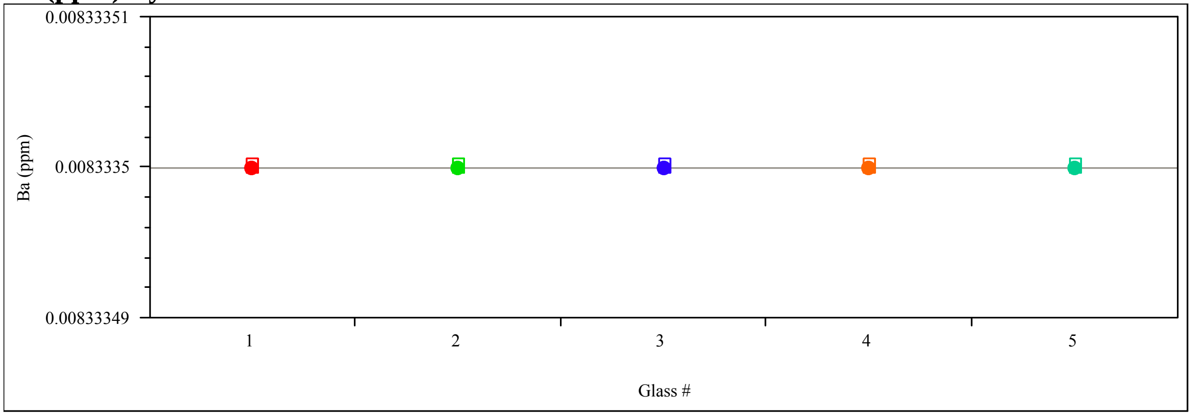


Exhibit E5. Laboratory PCT Measurements by Glass Number for Study Glasses

B (ppm) By Glass



Ba (ppm) By Glass



Cd (ppm) By Glass

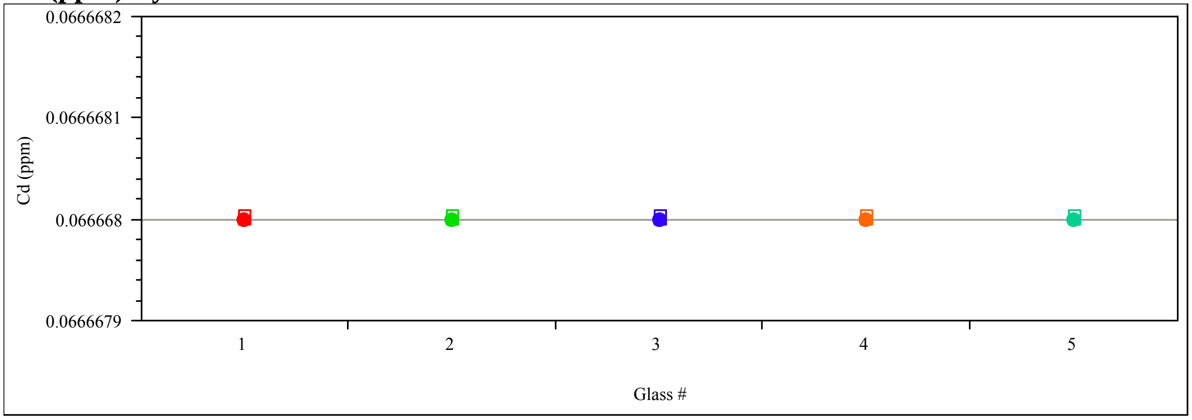
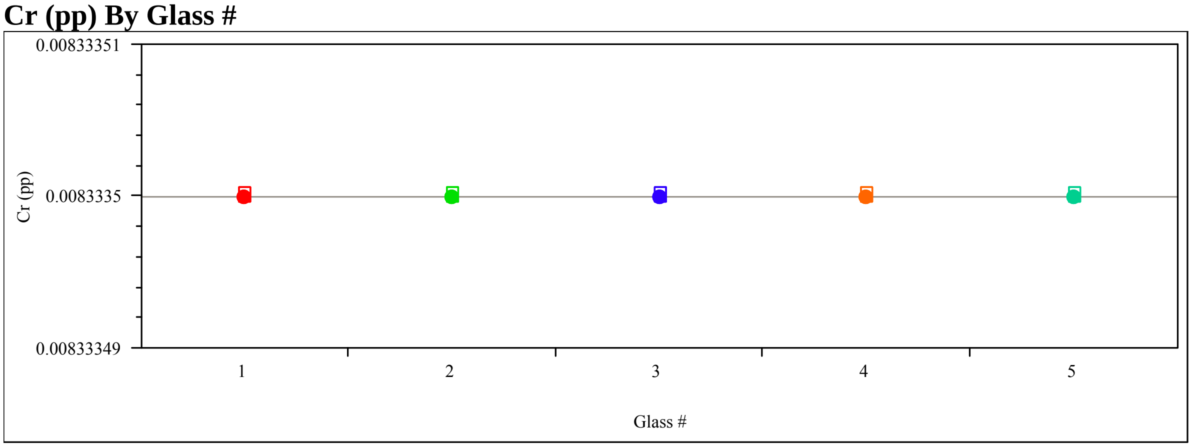
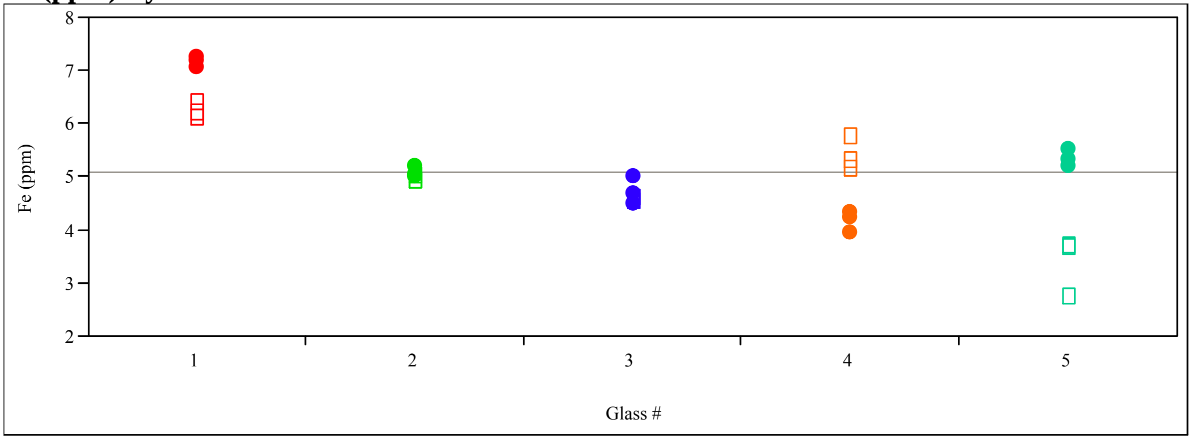


Exhibit E5. Laboratory PCT Measurements by Glass Number for Study Glasses (continued)

Cr (pp) By Glass



Fe (ppm) By Glass



Li (ppm) By Glass

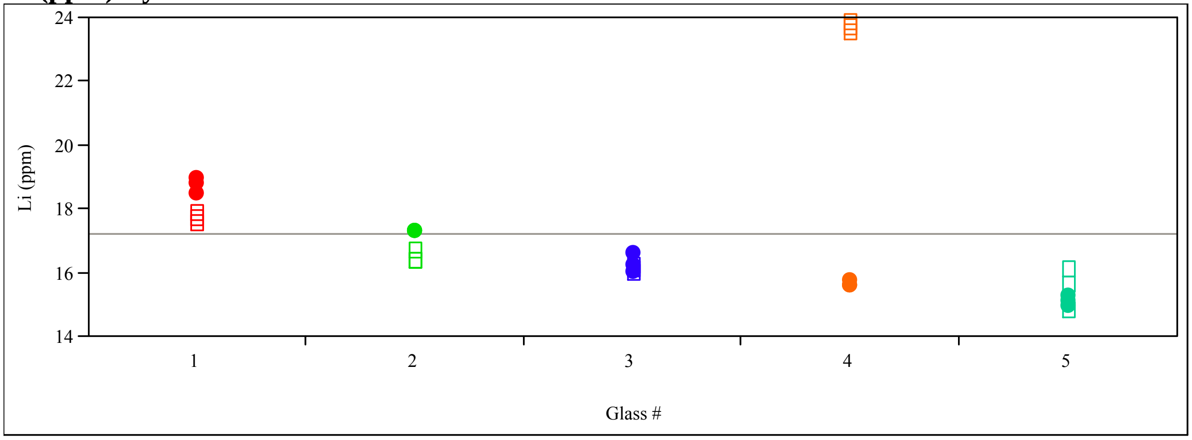
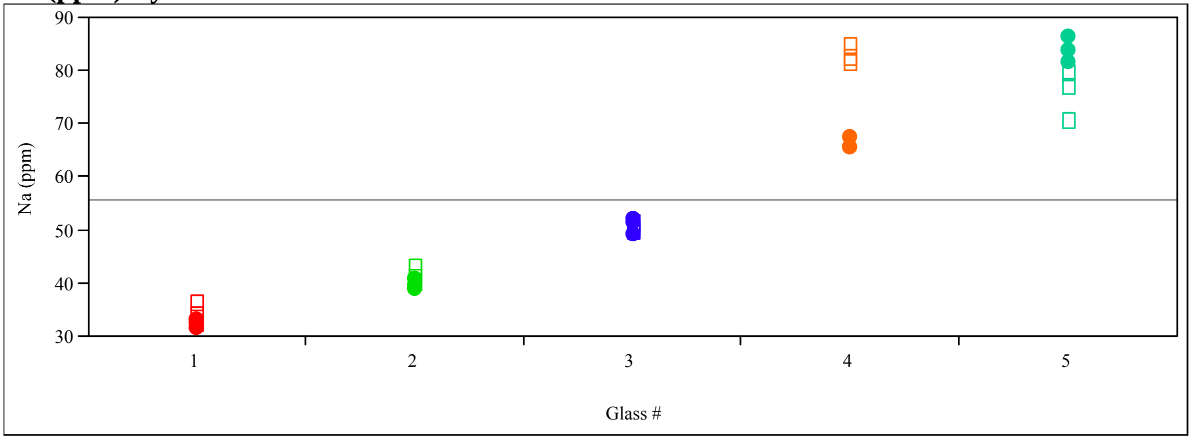
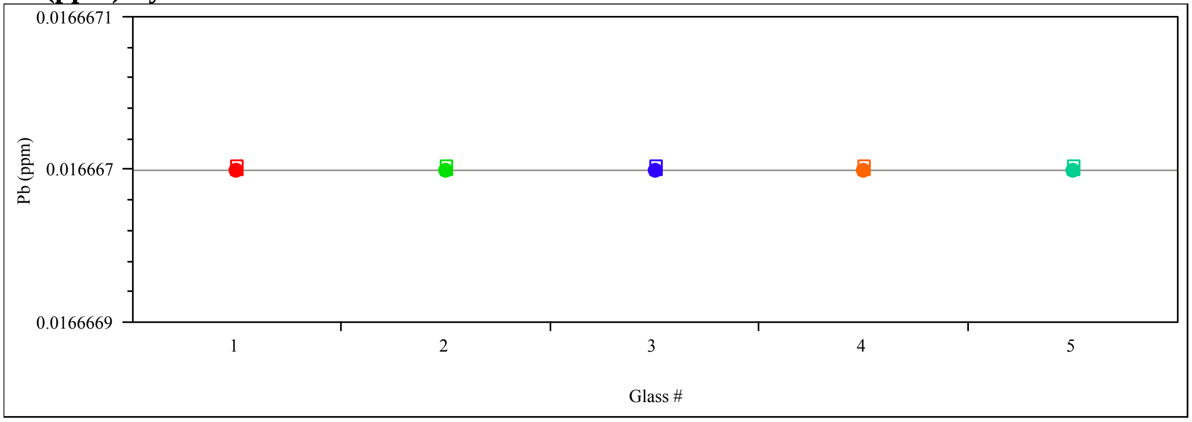


Exhibit E5. Laboratory PCT Measurements by Glass Number for Study Glasses (continued)

Na (ppm) By Glass



Pb (ppm) By Glass



Si (ppm) By Glass

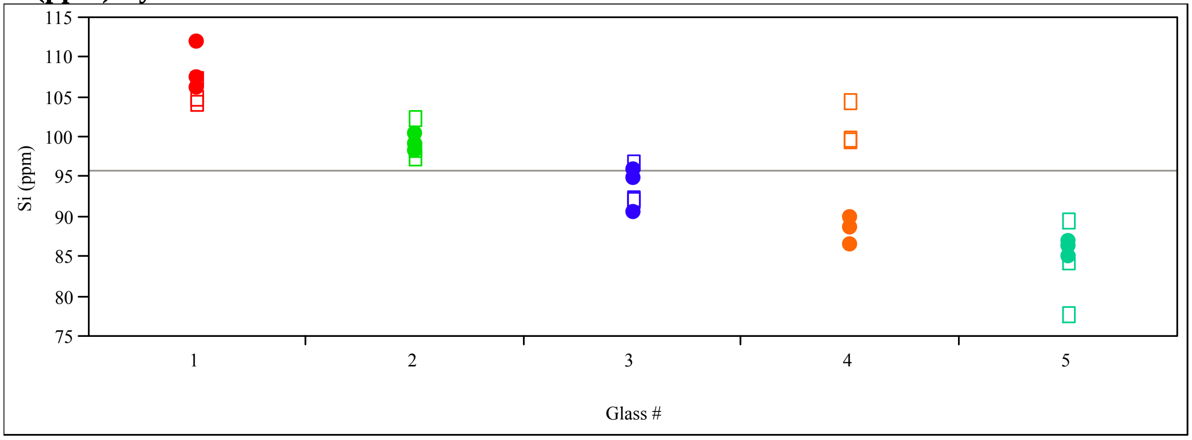
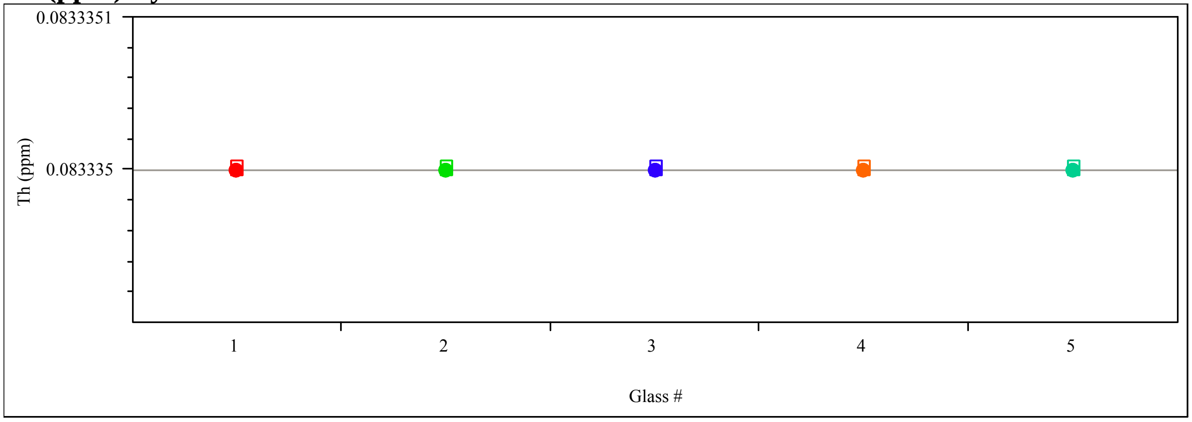


Exhibit E5. Laboratory PCT Measurements by Glass Number for Study Glasses (continued)

Th (ppm) By Glass



U (ppm) By Glass

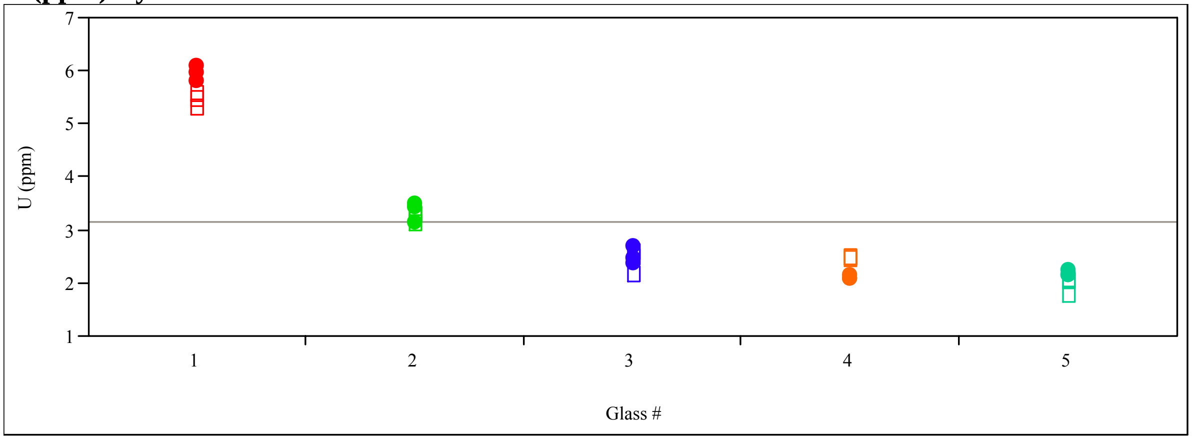


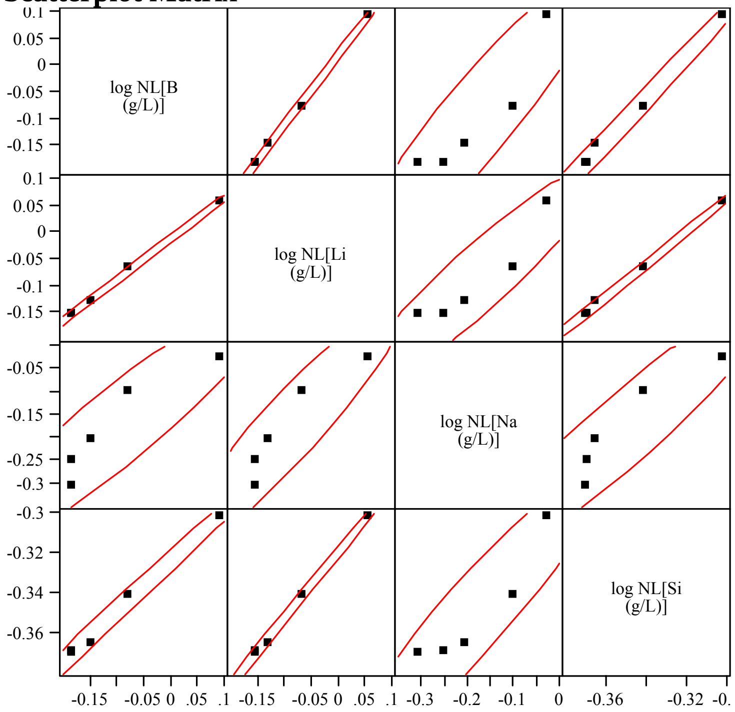
Exhibit E6. Correlations and Scatter Plots of Normalized PCTs Over All Compositional Views and Heat Treatments

Comp/HT=measured bc-ccc

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9990	0.9299	0.9959
log NL[Li (g/L)]	0.9990	1.0000	0.9378	0.9987
log NL[Na (g/L)]	0.9299	0.9378	1.0000	0.9369
log NL[Si (g/L)]	0.9959	0.9987	0.9369	1.0000

Scatterplot Matrix



Comp/HT=measured bc-quenched

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9497	0.9354	0.8839
log NL[Li (g/L)]	0.9497	1.0000	0.7925	0.9668
log NL[Na (g/L)]	0.9354	0.7925	1.0000	0.6810
log NL[Si (g/L)]	0.8839	0.9668	0.6810	1.0000

Scatterplot Matrix

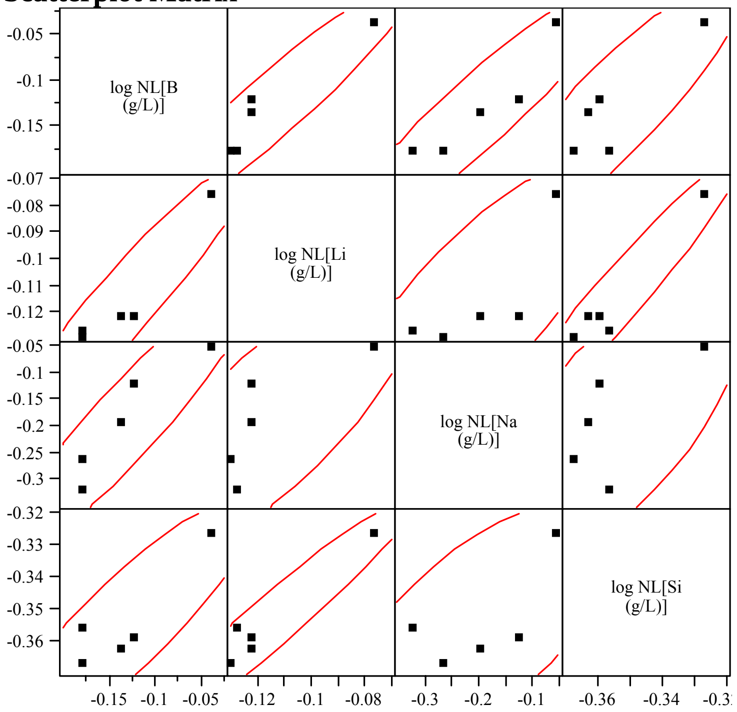


Exhibit E6. Correlations and Scatter Plots of Normalized PCTs Over All Compositional Views and Heat Treatments (continued)

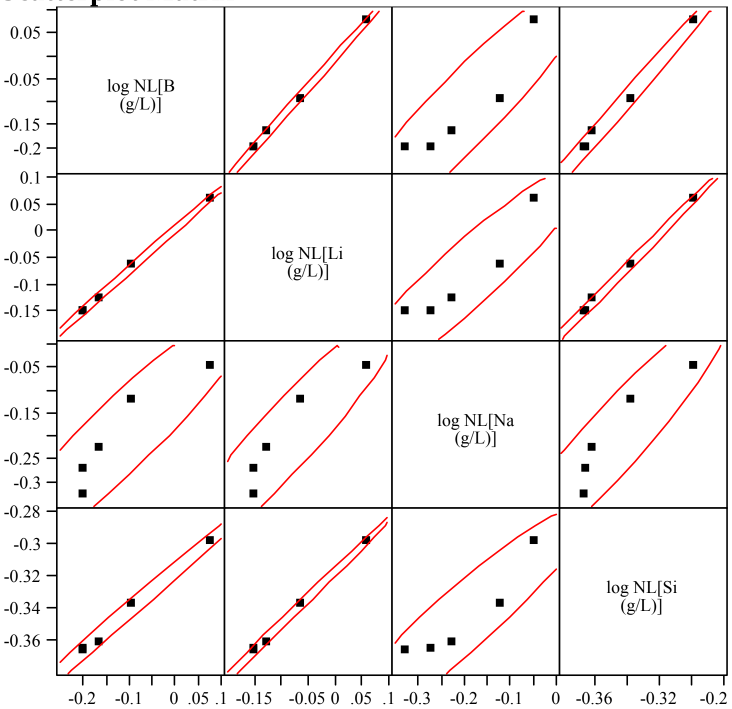
Comp/HT=measured-ccc

Multivariate

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9990	0.9300	0.9959
log NL[Li (g/L)]	0.9990	1.0000	0.9378	0.9986
log NL[Na (g/L)]	0.9300	0.9378	1.0000	0.9368
log NL[Si (g/L)]	0.9959	0.9986	0.9368	1.0000

Scatterplot Matrix



Comp/HT=measured-quenched

Multivariate

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9498	0.9353	0.8840
log NL[Li (g/L)]	0.9498	1.0000	0.7924	0.9666
log NL[Na (g/L)]	0.9353	0.7924	1.0000	0.6810
log NL[Si (g/L)]	0.8840	0.9666	0.6810	1.0000

Scatterplot Matrix

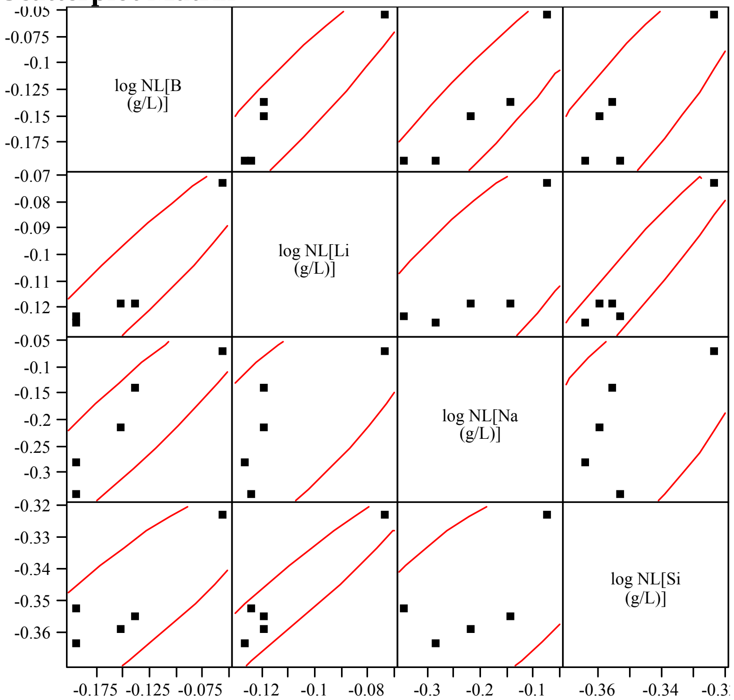


Exhibit E6. Correlations and Scatter Plots of Normalized PCTs Over All Compositional Views and Heat Treatments (continued)

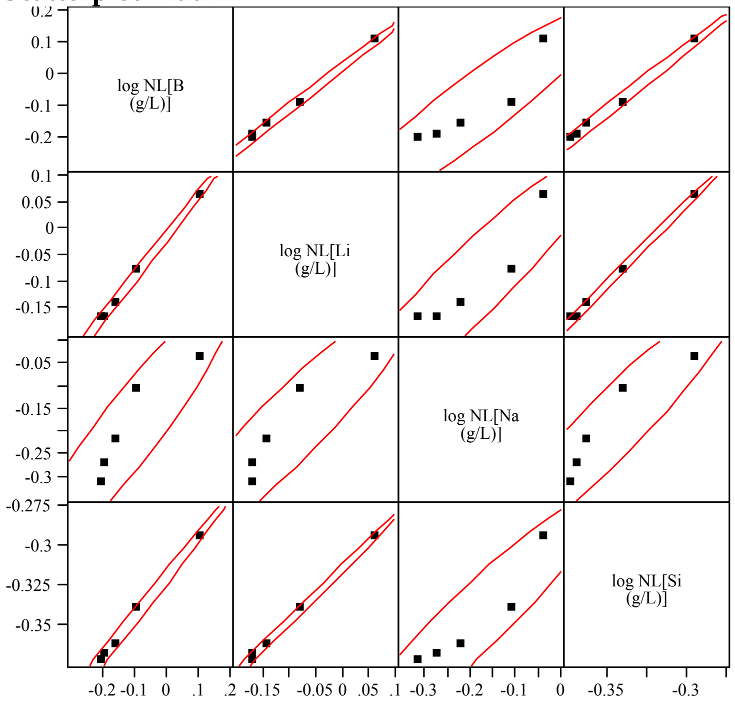
Comp/HT=targeted-ccc

Multivariate

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9984	0.9221	0.9972
log NL[Li (g/L)]	0.9984	1.0000	0.9314	0.9990
log NL[Na (g/L)]	0.9221	0.9314	1.0000	0.9418
log NL[Si (g/L)]	0.9972	0.9990	0.9418	1.0000

Scatterplot Matrix



Comp/HT=targeted-quenched

Multivariate

Correlations

	log NL[B (g/L)]	log NL[Li (g/L)]	log NL[Na (g/L)]	log NL[Si (g/L)]
log NL[B (g/L)]	1.0000	0.9787	0.9908	0.9011
log NL[Li (g/L)]	0.9787	1.0000	0.9469	0.9620
log NL[Na (g/L)]	0.9908	0.9469	1.0000	0.8350
log NL[Si (g/L)]	0.9011	0.9620	0.8350	1.0000

Scatterplot Matrix

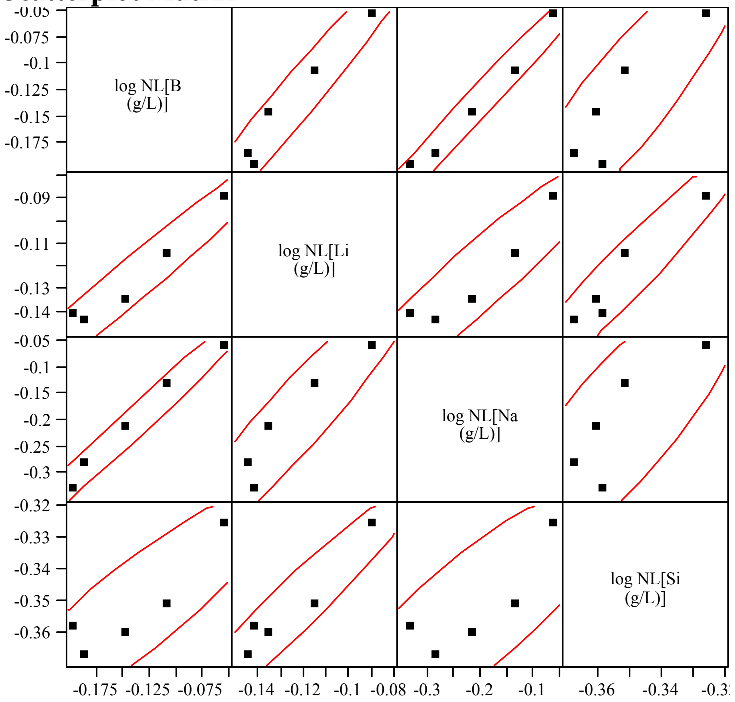
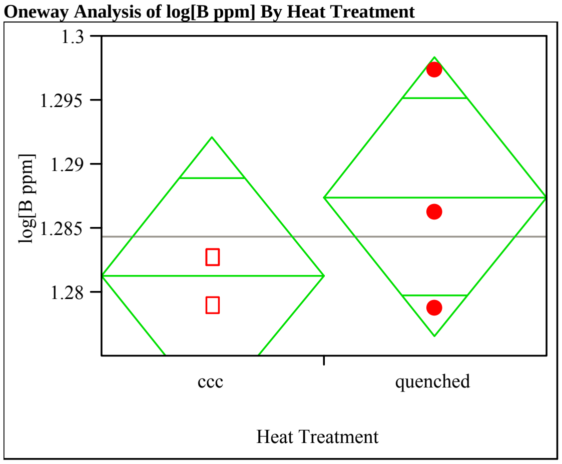


Exhibit E7. Effects of Heat Treatment on PCT ppm-Response of Study Glasses

Glass #=1

Oneway Analysis of log[B ppm] By Heat Treatment



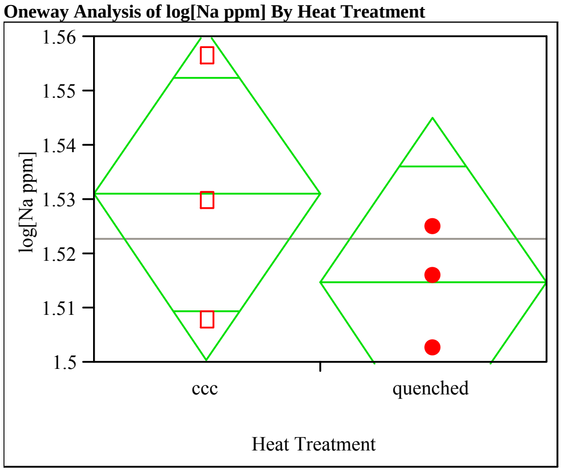
t Test

ccc-quenched

Assuming equal variances

Difference	-0.00620	t Ratio	-1.11579
Std Err Dif	0.00556	DF	4
Upper CL Dif	0.00923	Prob > |t|	0.3270
Lower CL Dif	-0.02164	Prob > t	0.8365
Confidence	0.95	Prob < t	0.1635

Oneway Analysis of log[Na ppm] By Heat Treatment



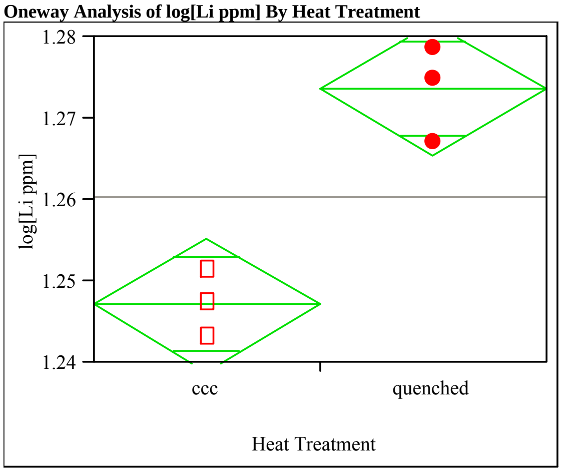
t Test

ccc-quenched

Assuming equal variances

Difference	0.01627	t Ratio	1.047022
Std Err Dif	0.01554	DF	4
Upper CL Dif	0.05942	Prob > |t|	0.3542
Lower CL Dif	-0.02687	Prob > t	0.1771
Confidence	0.95	Prob < t	0.8229

Oneway Analysis of log[Li ppm] By Heat Treatment



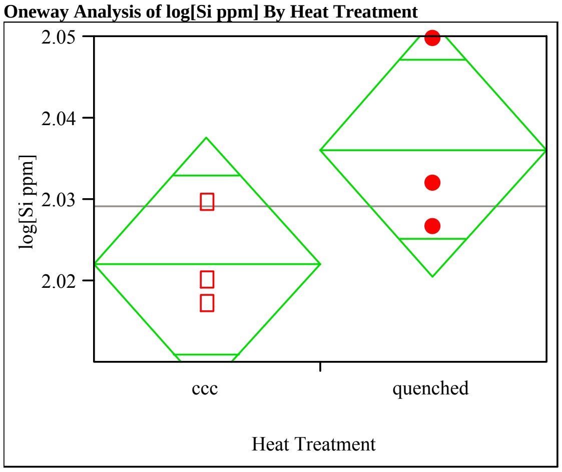
t Test

ccc-quenched

Assuming equal variances

Difference	-0.02648	t Ratio	-6.38333
Std Err Dif	0.00415	DF	4
Upper CL Dif	-0.01496	Prob > |t|	0.0031
Lower CL Dif	-0.03799	Prob > t	0.9985
Confidence	0.95	Prob < t	0.0015

Oneway Analysis of log[Si ppm] By Heat Treatment



t Test

ccc-quenched

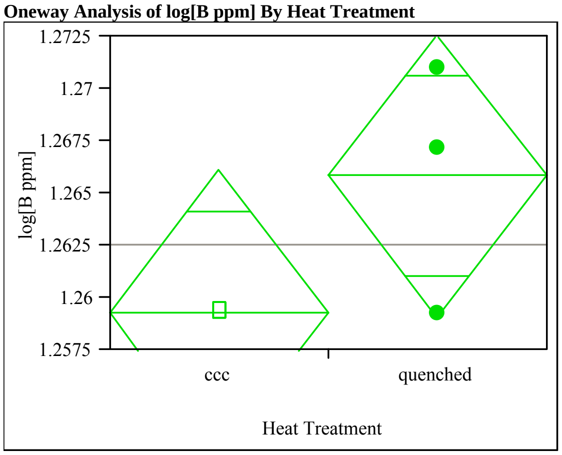
Assuming equal variances

Difference	-0.01413	t Ratio	-1.77907
Std Err Dif	0.00794	DF	4
Upper CL Dif	0.00792	Prob > |t|	0.1498
Lower CL Dif	-0.03618	Prob > t	0.9251
Confidence	0.95	Prob < t	0.0749

Exhibit E7. Effects of Heat Treatment on PCT ppm-Response of Study Glasses (continued)

Glass #=2

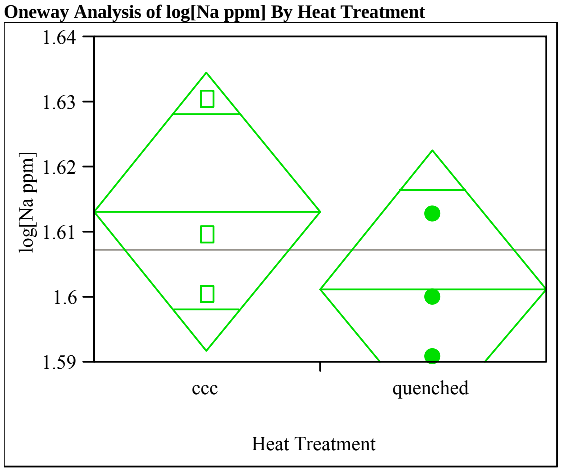
Oneway Analysis of log[B ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	-0.00656	t Ratio	-1.892
Std Err Dif	0.00347	DF	4
Upper CL Dif	0.00307	Prob > |t|	0.1314
Lower CL Dif	-0.01619	Prob > t	0.9343
Confidence	0.95	Prob < t	0.0657

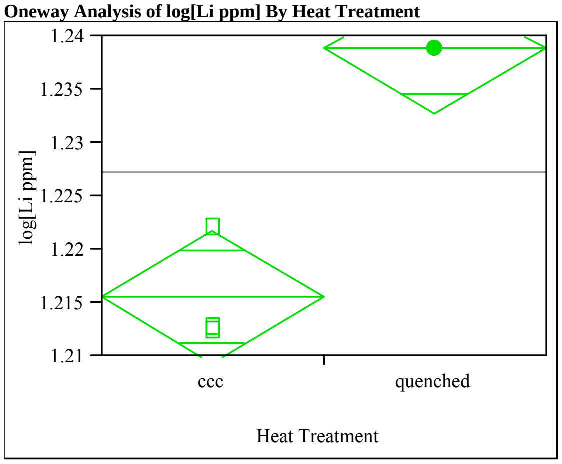
Oneway Analysis of log[Na ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	0.01183	t Ratio	1.089891
Std Err Dif	0.01085	DF	4
Upper CL Dif	0.04195	Prob > |t|	0.3370
Lower CL Dif	-0.01830	Prob > t	0.1685
Confidence	0.95	Prob < t	0.8315

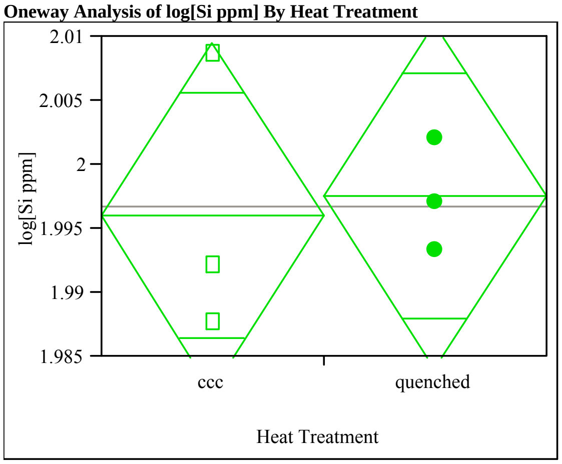
Oneway Analysis of log[Li ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	-0.02333	t Ratio	-7.40744
Std Err Dif	0.00315	DF	4
Upper CL Dif	-0.01458	Prob > |t|	0.0018
Lower CL Dif	-0.03207	Prob > t	0.9991
Confidence	0.95	Prob < t	0.0009

Oneway Analysis of log[Si ppm] By Heat Treatment



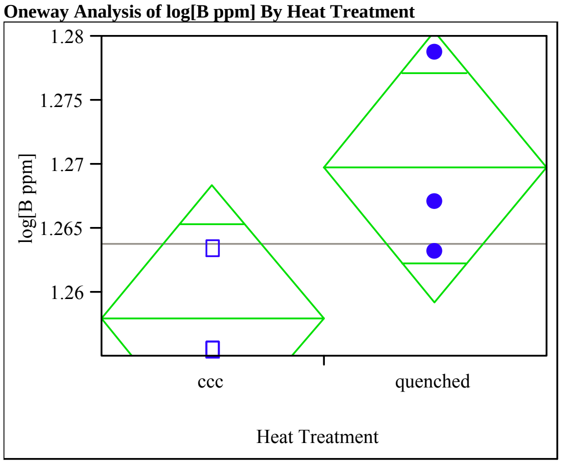
t Test
ccc-quenched
Assuming equal variances

Difference	-0.00154	t Ratio	-0.22307
Std Err Dif	0.00690	DF	4
Upper CL Dif	0.01761	Prob > |t|	0.8344
Lower CL Dif	-0.02069	Prob > t	0.5828
Confidence	0.95	Prob < t	0.4172

Exhibit E7. Effects of Heat Treatment on PCT ppm-Response of Study Glasses (continued)

Glass #=3

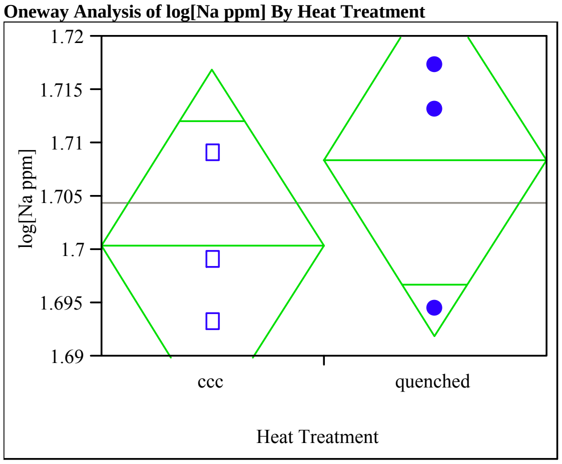
Oneway Analysis of log[B ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

```
Difference     -0.01179  t Ratio     -2.20008
Std Err Dif     0.00536  DF                 4
Upper CL Dif    0.00309  Prob > |t|    0.0926
Lower CL Dif  -0.02668  Prob > t       0.9537
Confidence         0.95  Prob < t      0.0463
```

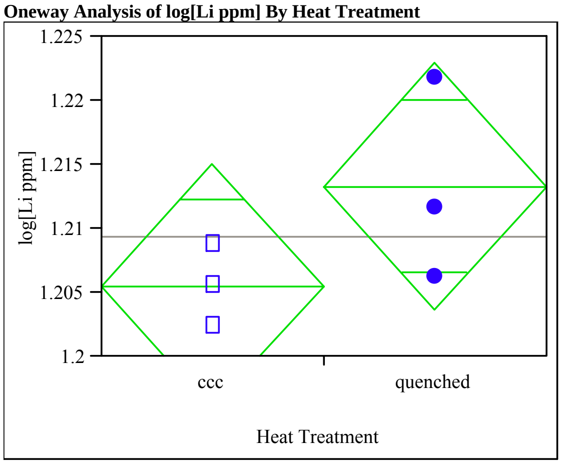
Oneway Analysis of log[Na ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

```
Difference     -0.00804  t Ratio     -0.95743
Std Err Dif     0.00839  DF                 4
Upper CL Dif    0.01527  Prob > |t|    0.3926
Lower CL Dif  -0.03134  Prob > t       0.8037
Confidence         0.95  Prob < t      0.1963
```

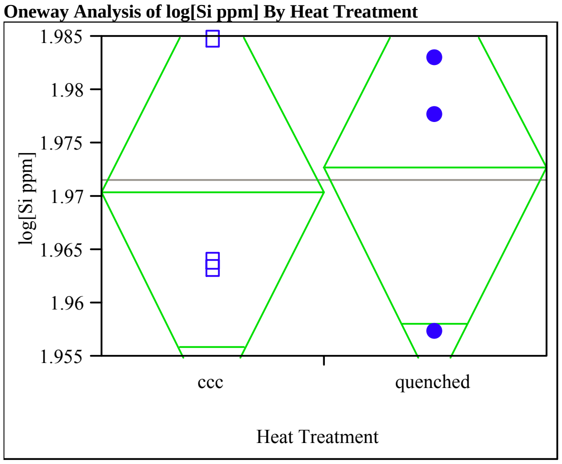
Oneway Analysis of log[Li ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

```
Difference     -0.00786  t Ratio     -1.60695
Std Err Dif     0.00489  DF                 4
Upper CL Dif    0.00572  Prob > |t|    0.1833
Lower CL Dif  -0.02143  Prob > t       0.9083
Confidence         0.95  Prob < t      0.0917
```

Oneway Analysis of log[Si ppm] By Heat Treatment



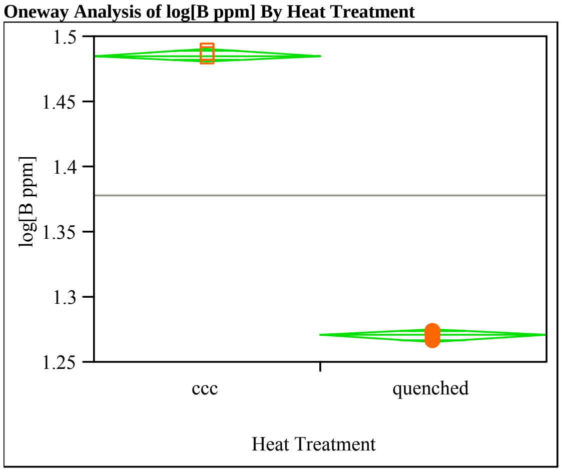
t Test
ccc-quenched
Assuming equal variances

```
Difference     -0.00229  t Ratio     -0.21826
Std Err Dif     0.01051  DF                 4
Upper CL Dif    0.02688  Prob > |t|    0.8379
Lower CL Dif  -0.03147  Prob > t       0.5810
Confidence         0.95  Prob < t      0.4190
```

Exhibit E7. Effects of Heat Treatment on PCT ppm-Response of Study Glasses (continued)

Glass #=4

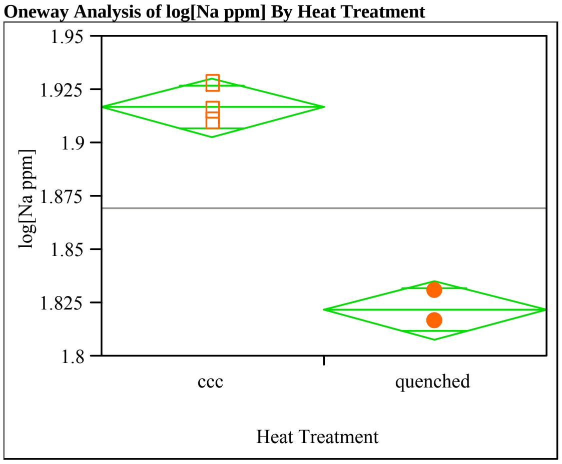
Oneway Analysis of log[B ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	0.214822	t Ratio	90.49923
Std Err Dif	0.002374	DF	4
Upper CL Dif	0.221413	Prob > |t|	<.0001
Lower CL Dif	0.208232	Prob > t	<.0001
Confidence	0.95	Prob < t	1.0000

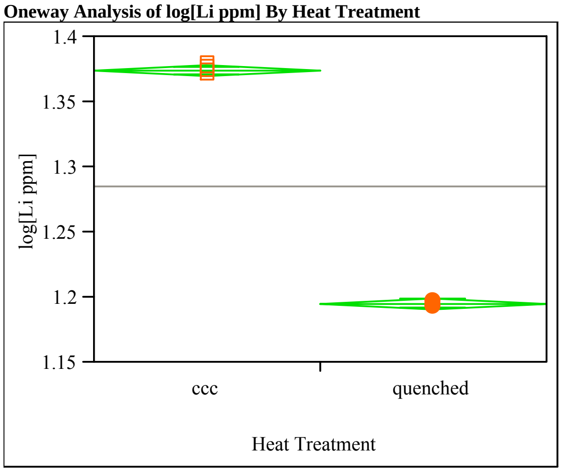
Oneway Analysis of log[Na ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	0.094932	t Ratio	13.5833
Std Err Dif	0.006989	DF	4
Upper CL Dif	0.114337	Prob > |t|	0.0002
Lower CL Dif	0.075528	Prob > t	<.0001
Confidence	0.95	Prob < t	0.9999

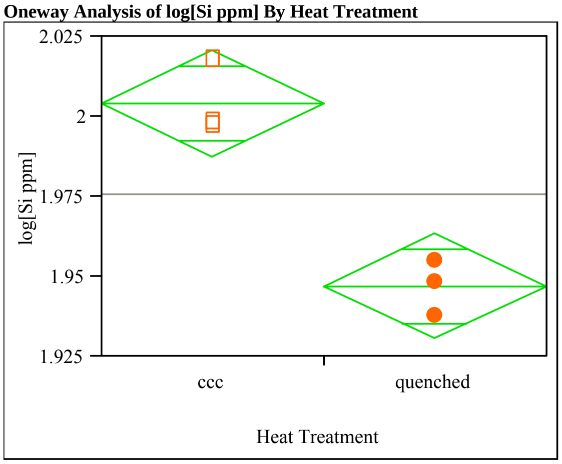
Oneway Analysis of log[Li ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	0.178542	t Ratio	81.15857
Std Err Dif	0.002200	DF	4
Upper CL Dif	0.184650	Prob > |t|	<.0001
Lower CL Dif	0.172434	Prob > t	<.0001
Confidence	0.95	Prob < t	1.0000

Oneway Analysis of log[Si ppm] By Heat Treatment



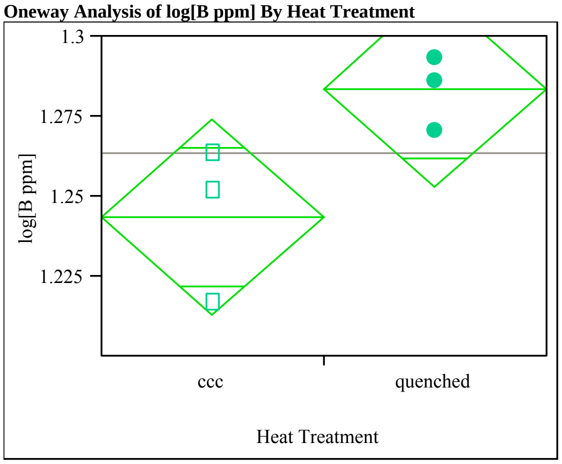
t Test
ccc-quenched
Assuming equal variances

Difference	0.057058	t Ratio	6.77942
Std Err Dif	0.008416	DF	4
Upper CL Dif	0.080425	Prob > |t|	0.0025
Lower CL Dif	0.033690	Prob > t	0.0012
Confidence	0.95	Prob < t	0.9988

Exhibit E7. Effects of Heat Treatment on PCT ppm-Response of Study Glasses (continued)

Glass #=5

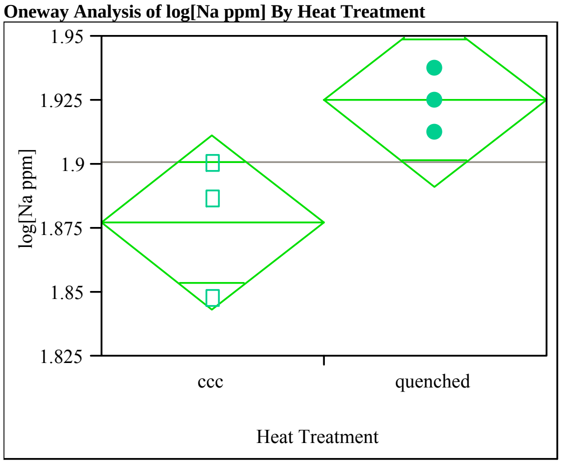
Oneway Analysis of log[B ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	-0.04015	t Ratio	-2.57094
Std Err Dif	0.01562	DF	4
Upper CL Dif	0.00321	Prob > |t|	0.0619
Lower CL Dif	-0.08352	Prob > t	0.9690
Confidence	0.95	Prob < t	0.0310

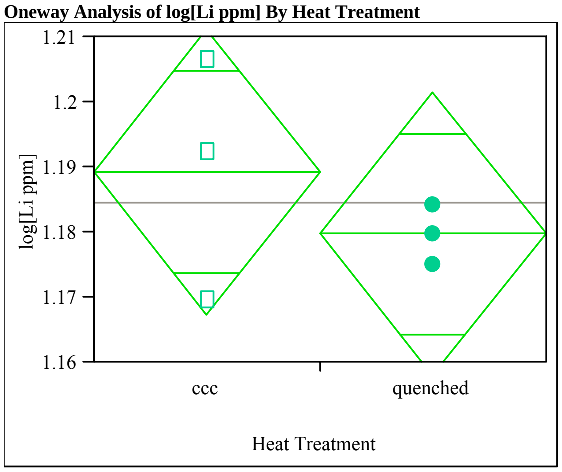
Oneway Analysis of log[Na ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	-0.04792	t Ratio	-2.78377
Std Err Dif	0.01721	DF	4
Upper CL Dif	-0.00013	Prob > |t|	0.0496
Lower CL Dif	-0.09571	Prob > t	0.9752
Confidence	0.95	Prob < t	0.0248

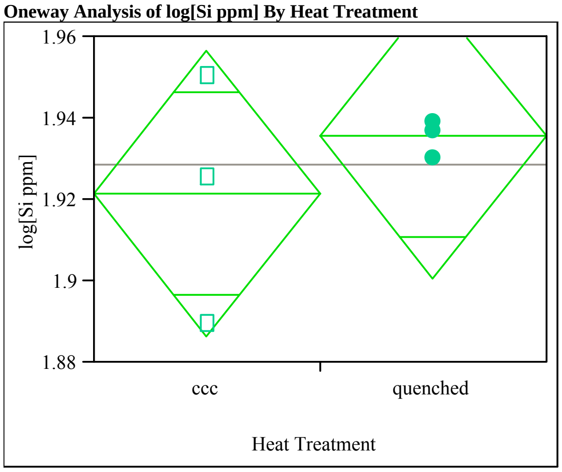
Oneway Analysis of log[Li ppm] By Heat Treatment



t Test
ccc-quenched
Assuming equal variances

Difference	0.00953	t Ratio	0.856527
Std Err Dif	0.01112	DF	4
Upper CL Dif	0.04040	Prob > |t|	0.4400
Lower CL Dif	-0.02135	Prob > t	0.2200
Confidence	0.95	Prob < t	0.7800

Oneway Analysis of log[Si ppm] By Heat Treatment



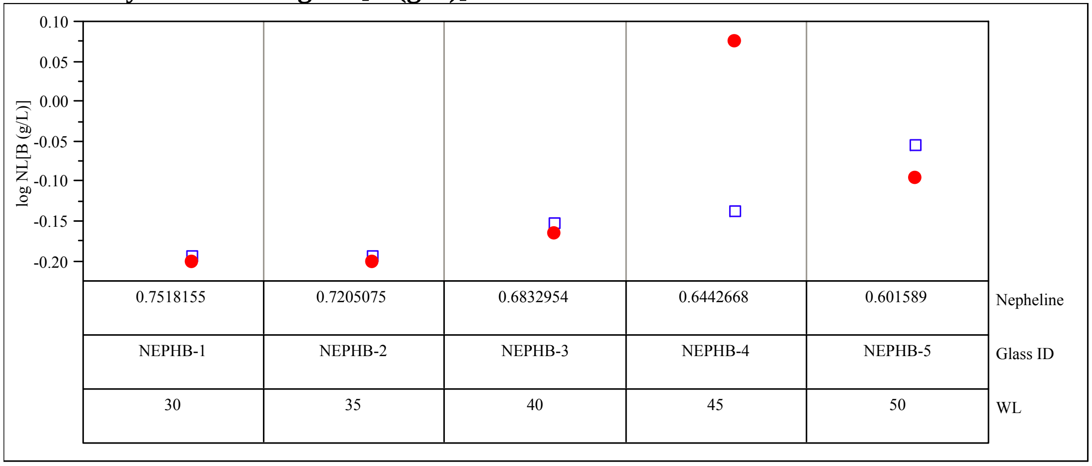
t Test
ccc-quenched
Assuming equal variances

Difference	-0.01405	t Ratio	-0.7857
Std Err Dif	0.01789	DF	4
Upper CL Dif	0.03561	Prob > |t|	0.4760
Lower CL Dif	-0.06371	Prob > t	0.7620
Confidence	0.95	Prob < t	0.2380

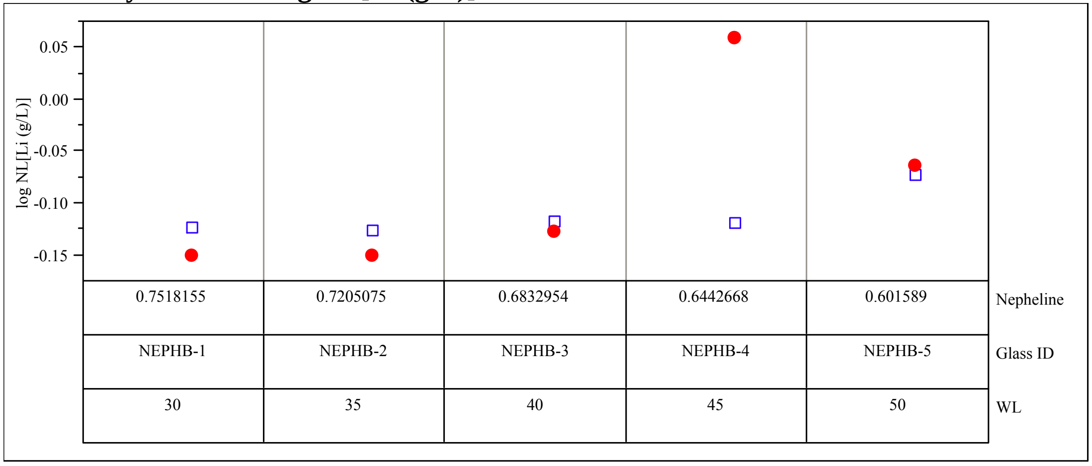
Exhibit E8. Effects of Heat Treatment for Study Glasses by Compositional View

Composition=measured

Variability Chart for log NL[B (g/L)]



Variability Chart for log NL[Li (g/L)]



Variability Chart for log NL[Na (g/L)]

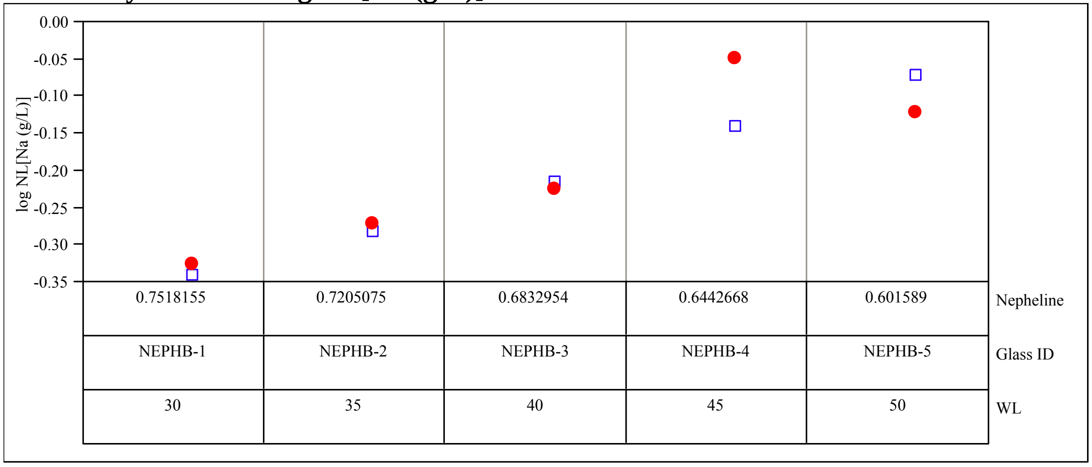
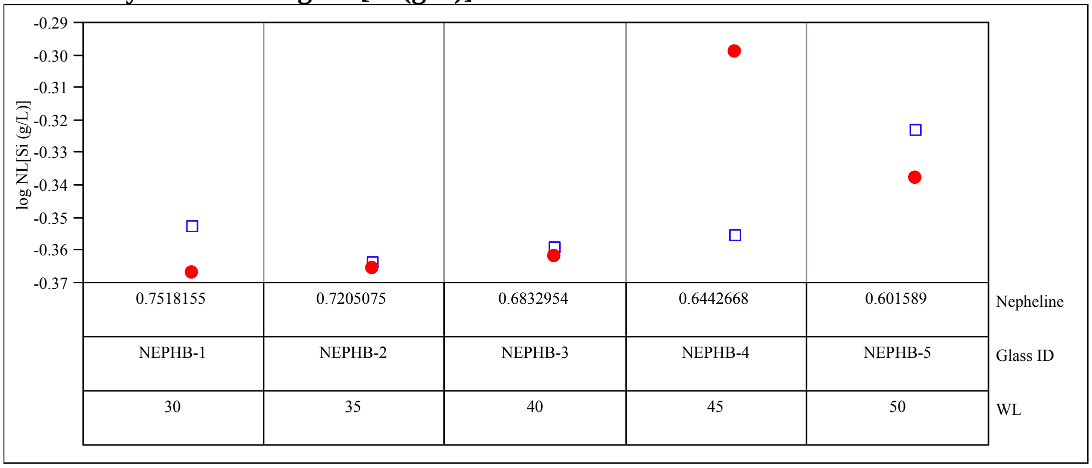


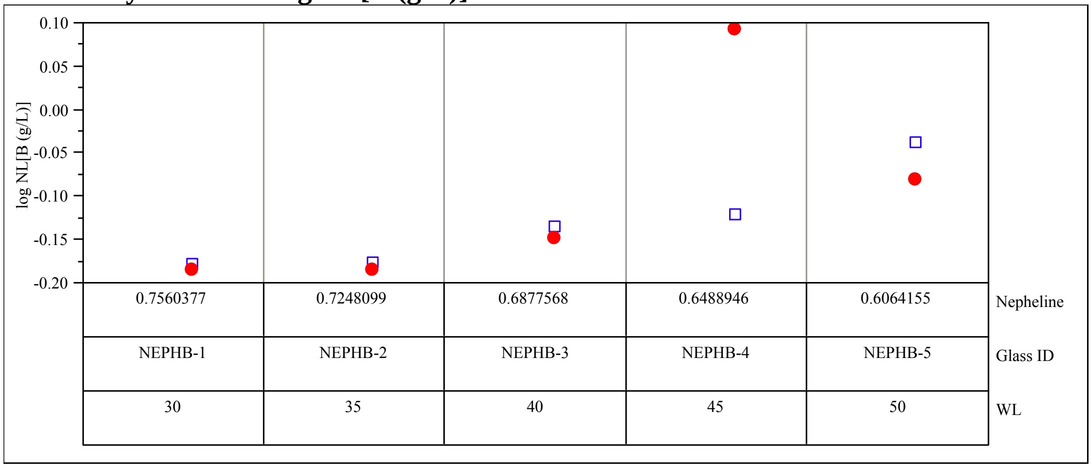
Exhibit E8. Effects of Heat Treatment for Study Glasses by Compositional View (continued)

Variability Chart for log NL[Si (g/L)]



Composition=measured bc

Variability Chart for log NL[B (g/L)]



Variability Chart for log NL[Li (g/L)]

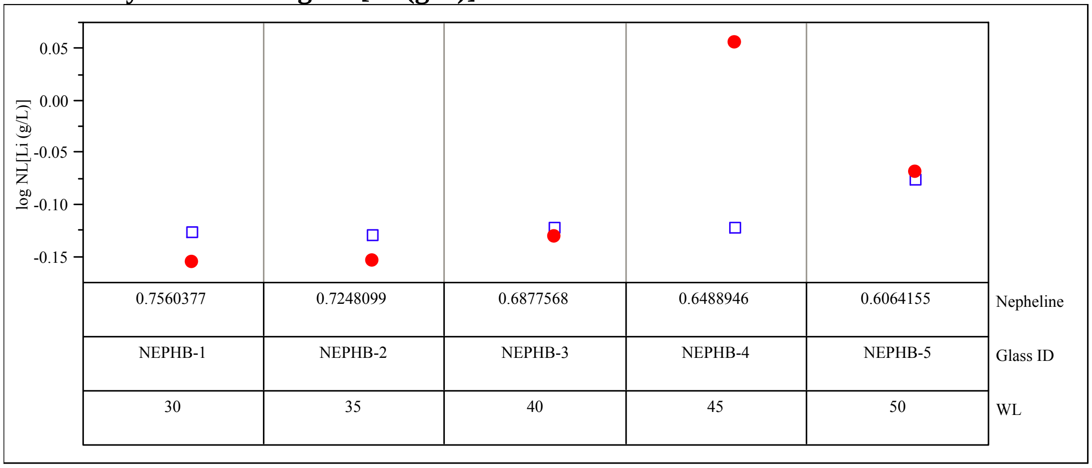
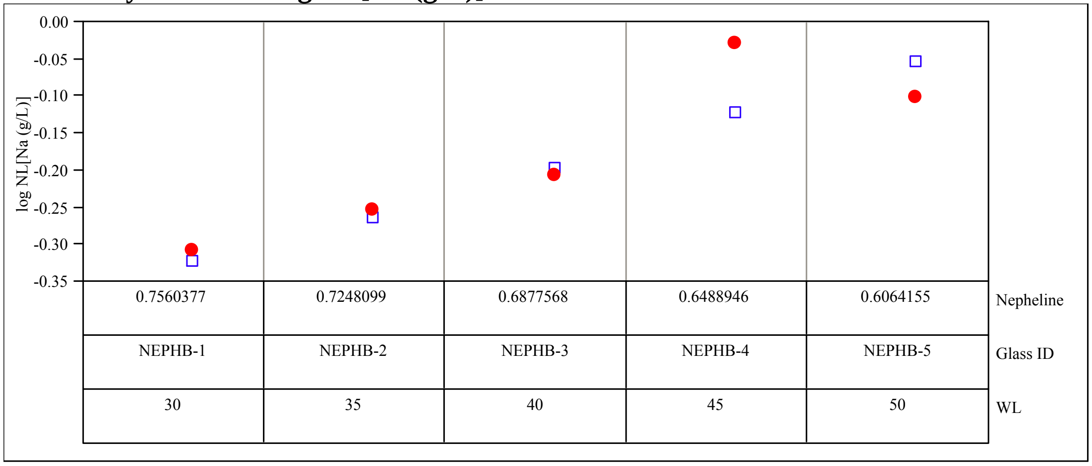
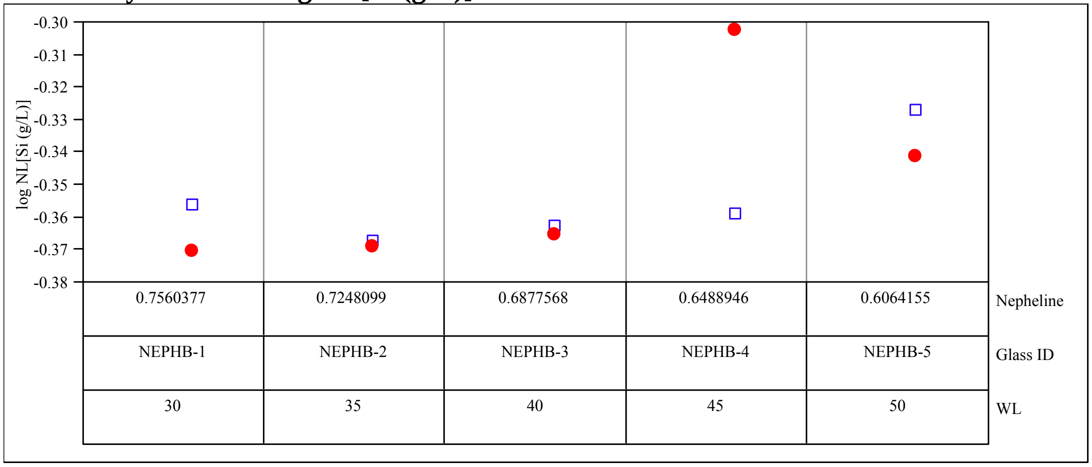


Exhibit E8. Effects of Heat Treatment for Study Glasses by Compositional View (continued)

Variability Chart for log NL[Na (g/L)]



Variability Chart for log NL[Si (g/L)]



Composition=targeted

Variability Chart for log NL[B (g/L)]

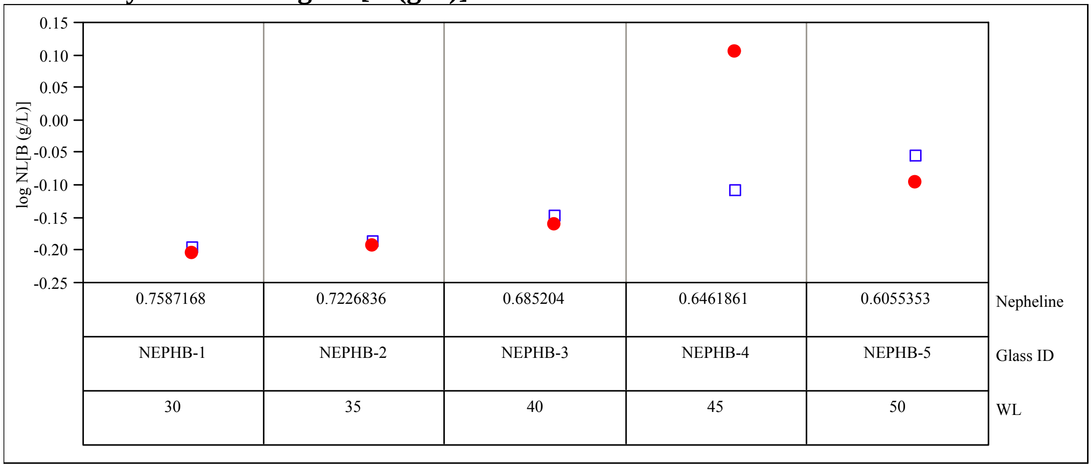
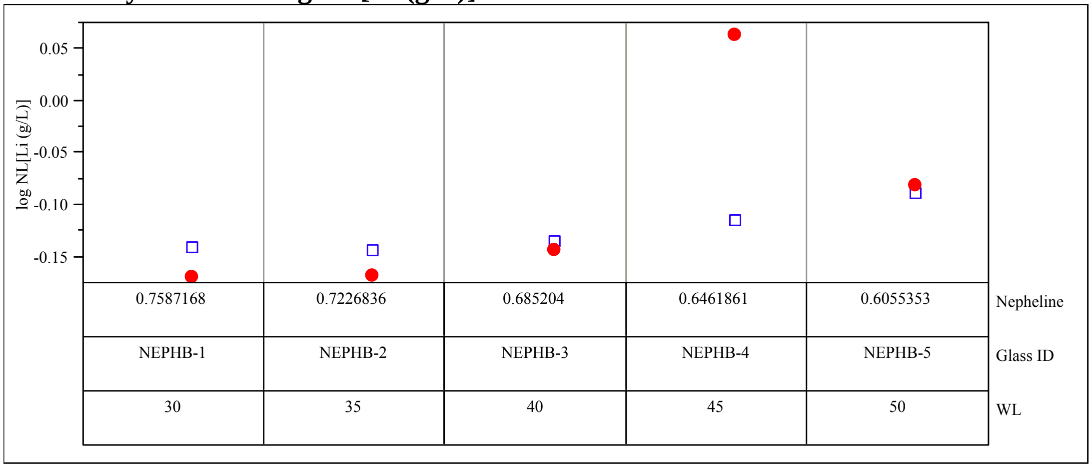
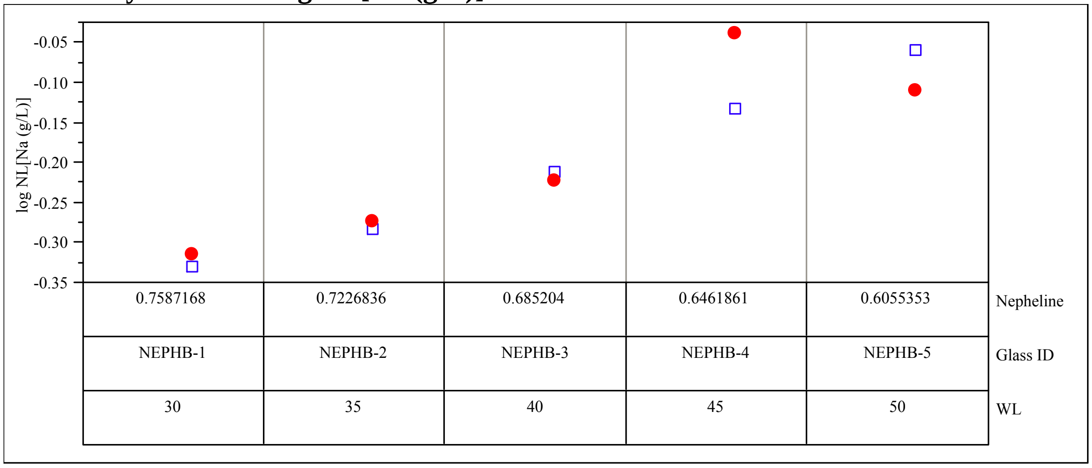


Exhibit E8. Effects of Heat Treatment for Study Glasses by Compositional View (continued)

Variability Chart for log NL[Li (g/L)]



Variability Chart for log NL[Na (g/L)]



Variability Chart for log NL[Si (g/L)]

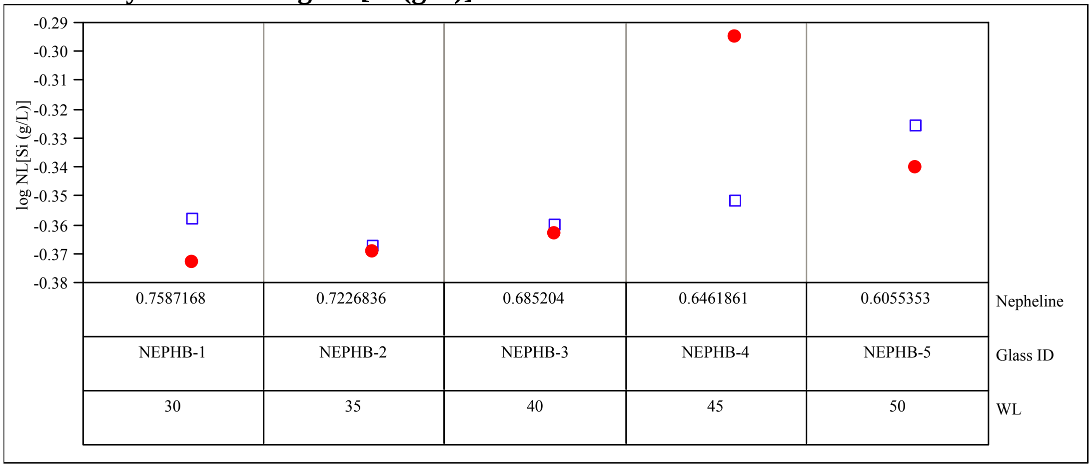
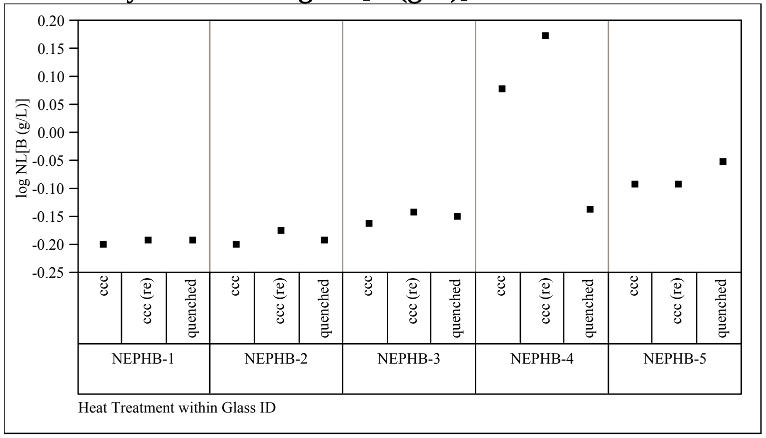


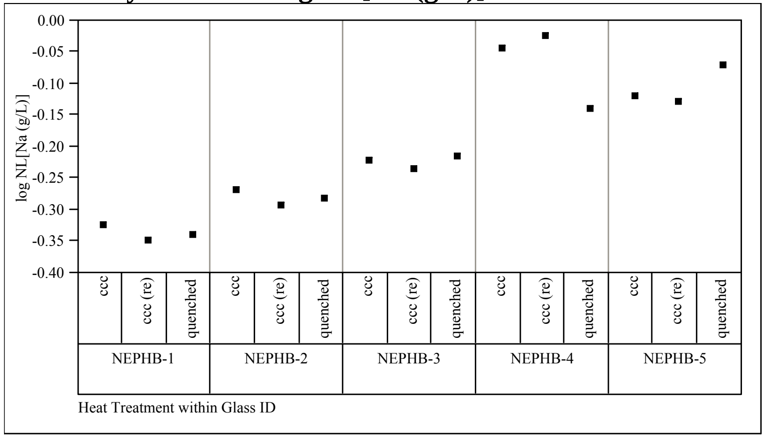
Exhibit E9. Effects of Heat Treatment for Study Glasses by Compositional View, Including Re-tested Values for ccc Glasses

Comp View=measured

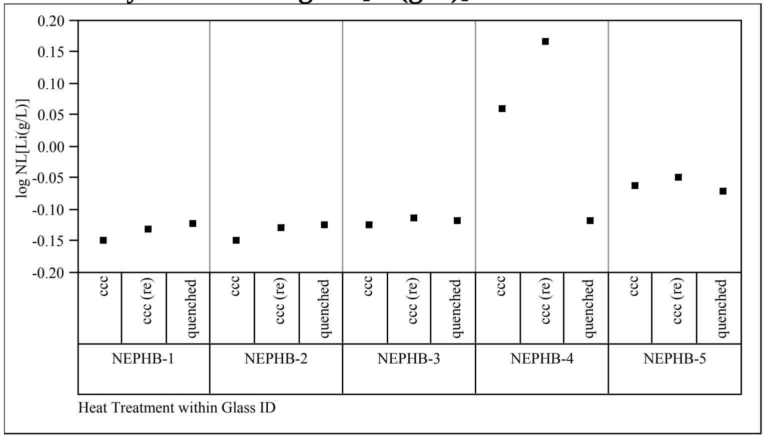
Variability Chart for log NL[B (g/L)]



Variability Chart for log NL[Na (g/L)]



Variability Chart for log NL[Li(g/L)]



Variability Chart for log NL[Si (g/L)]

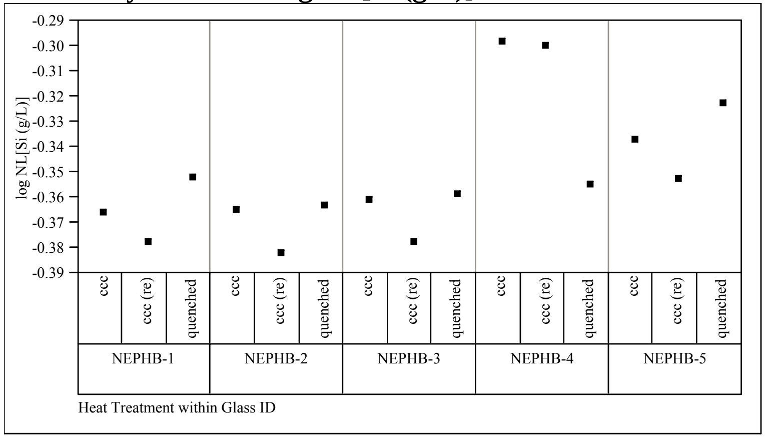
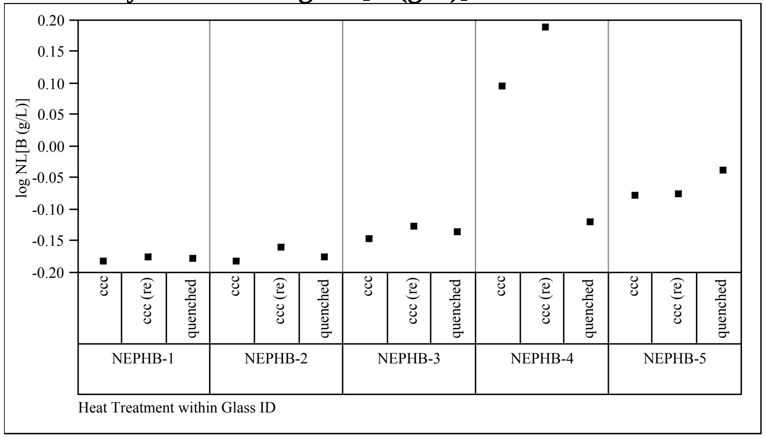


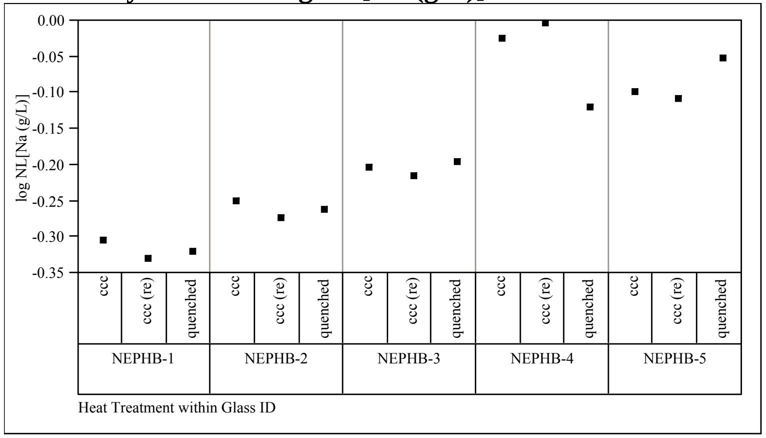
Exhibit E9. Effects of Heat Treatment for Study Glasses by Compositional View, Including Re-tested Values for ccc Glasses (continued)

Comp View=measured bc

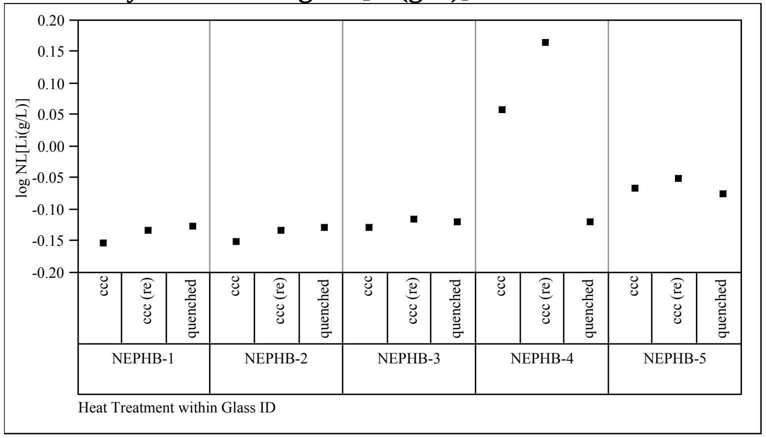
Variability Chart for log NL[B (g/L)]



Variability Chart for log NL[Na (g/L)]



Variability Chart for log NL[Li(g/L)]



Variability Chart for log NL[Si (g/L)]

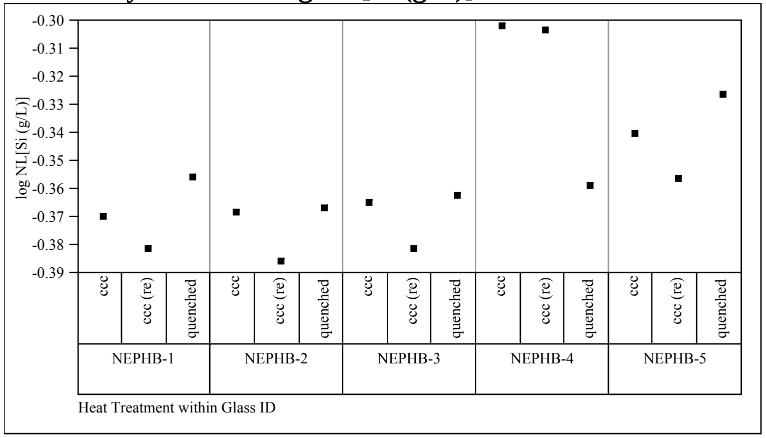
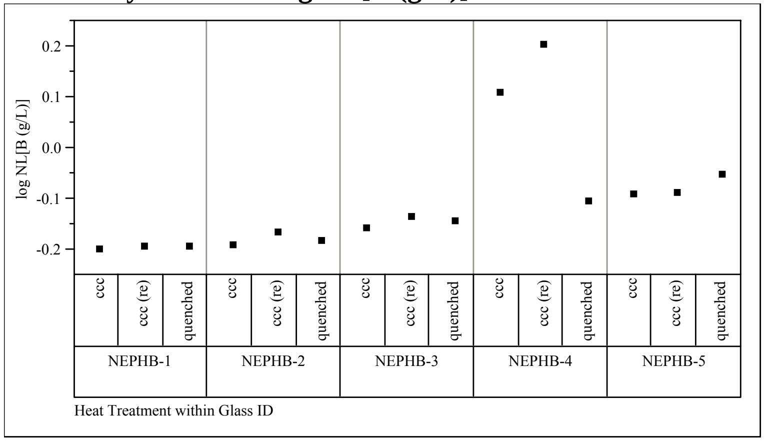


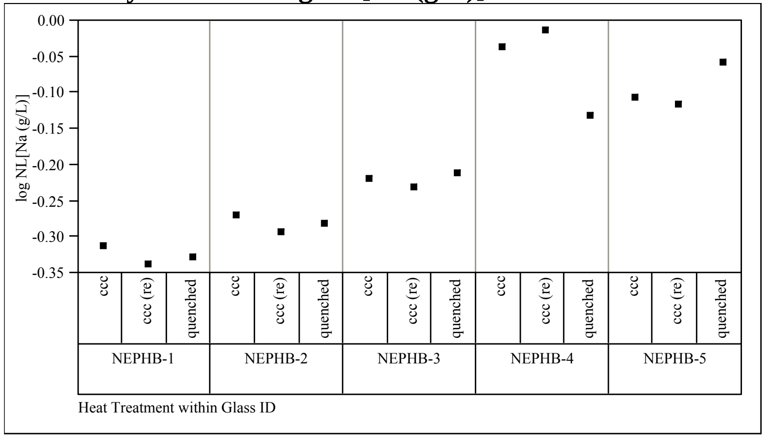
Exhibit E9. Effects of Heat Treatment for Study Glasses by Compositional View, Including Re-tested Values for ccc Glasses (continued)

Comp View=targeted

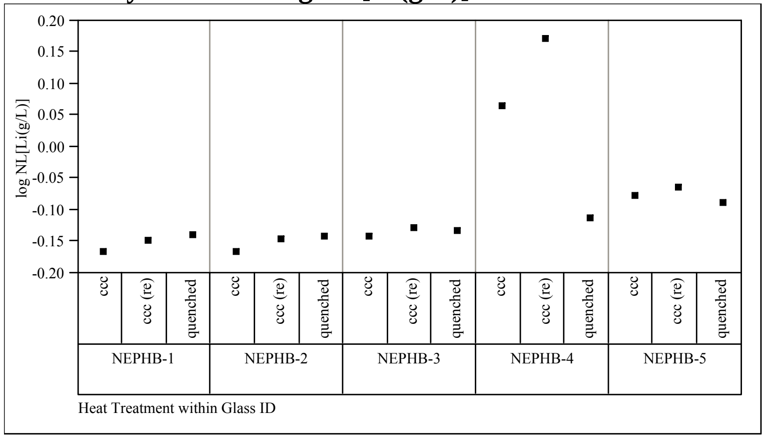
Variability Chart for log NL[B (g/L)]



Variability Chart for log NL[Na (g/L)]



Variability Chart for log NL[Li(g/L)]



Variability Chart for log NL[Si (g/L)]

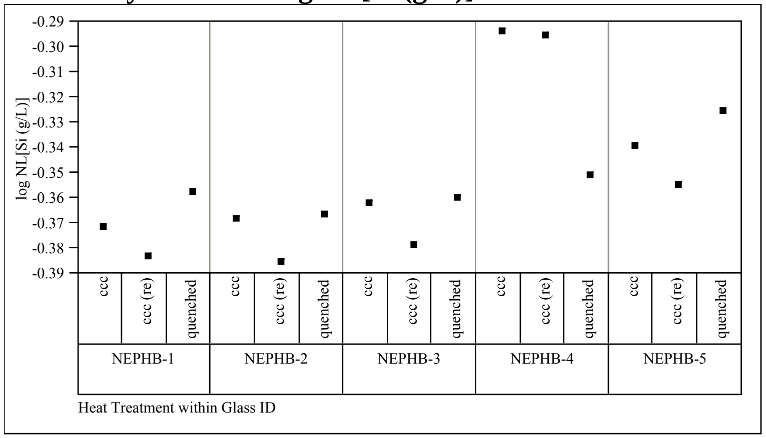
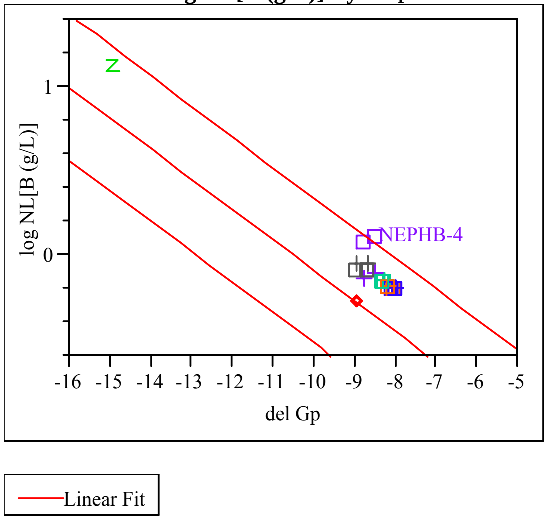
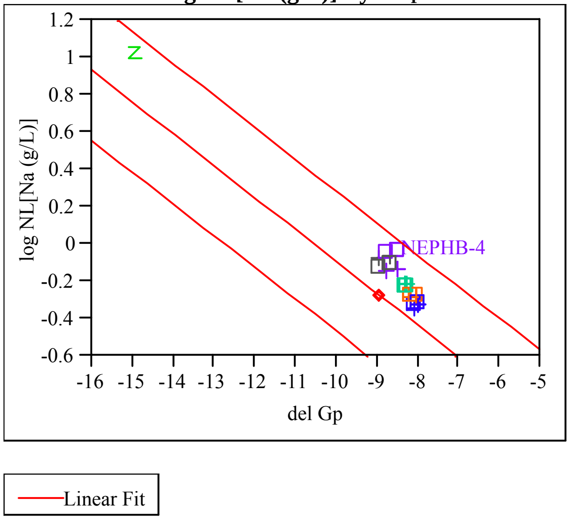
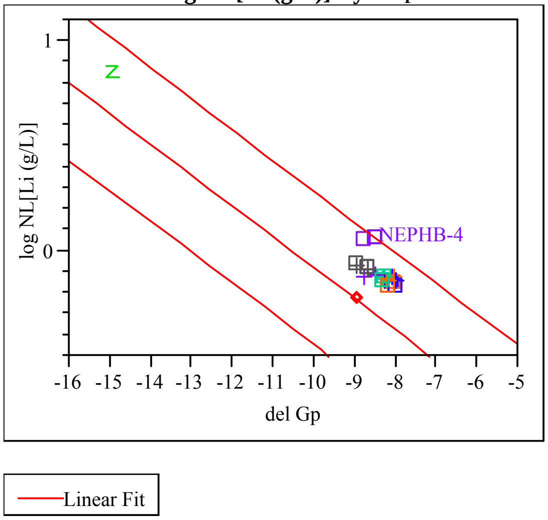


Exhibit E10. ΔG_P Predictions versus Common Logarithm Normalized Leachate (log NL[.]) for B, Li, Na, and Si Over All Compositional Views and Heat Treatments


Bivariate Fit of log NL[B (g/L)] By ΔG_P


Bivariate Fit of log NL[Na (g/L)] By ΔG_P


Bivariate Fit of log NL[Li (g/L)] By ΔG_P

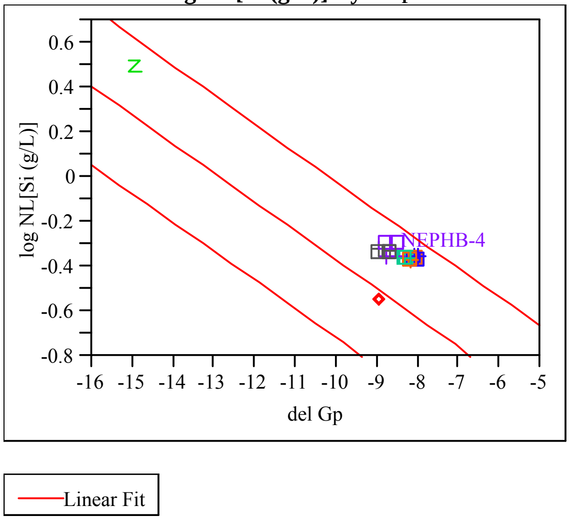
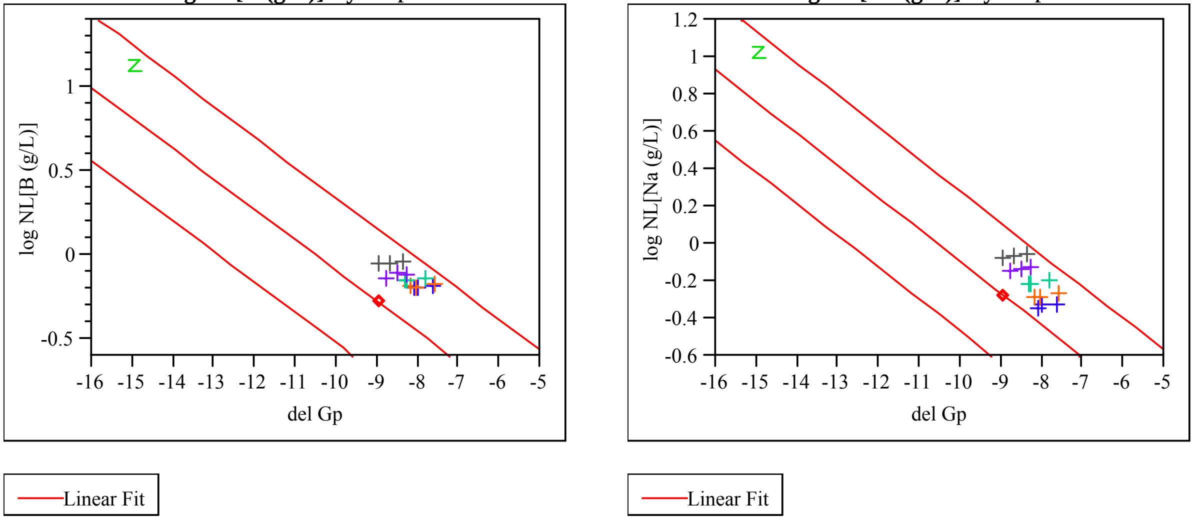
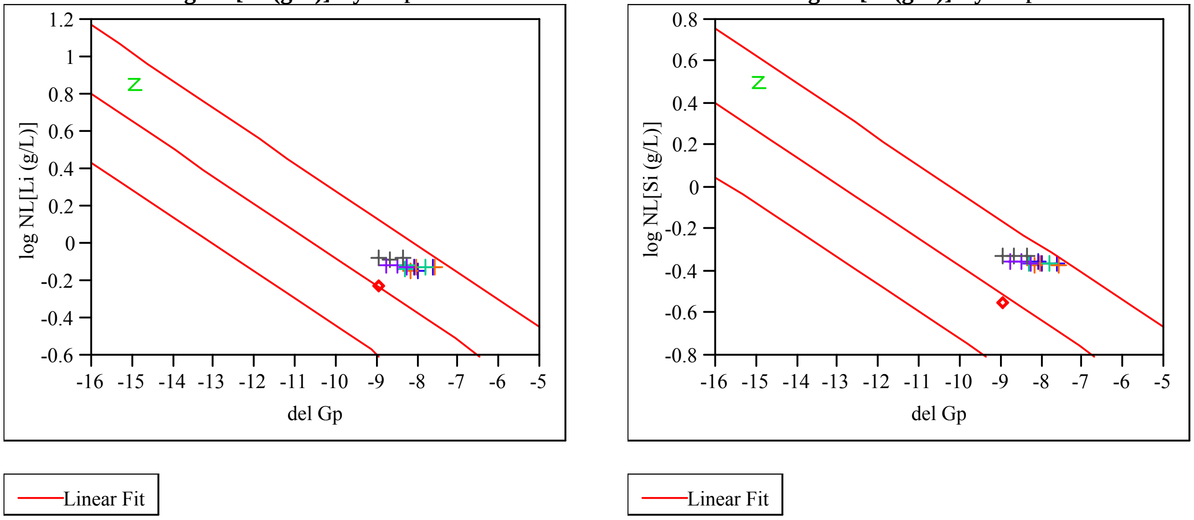

Bivariate Fit of log NL[Si (g/L)] By ΔG_P

Exhibit E11. ΔG_P Predictions versus Common Logarithm Normalized Leachate (log NL[.]) for B, Li, Na, and Si Over All Compositional Views for Quenched Glasses

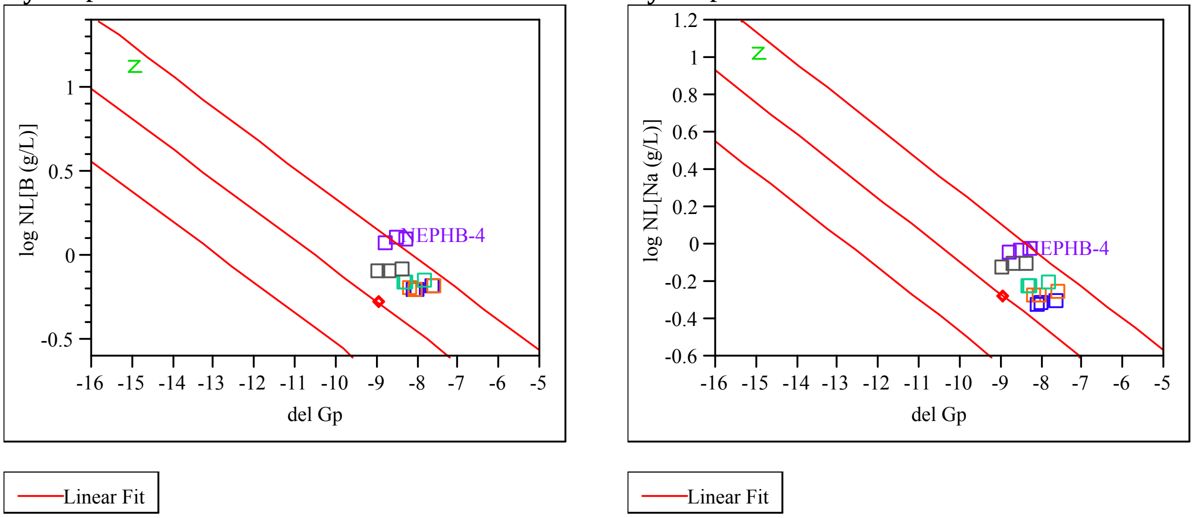

Bivariate Fit of log NL[B (g/L)] By ΔG_P

Bivariate Fit of log NL[Na (g/L)] By ΔG_P


Bivariate Fit of log NL[Li (g/L)] By ΔG_P

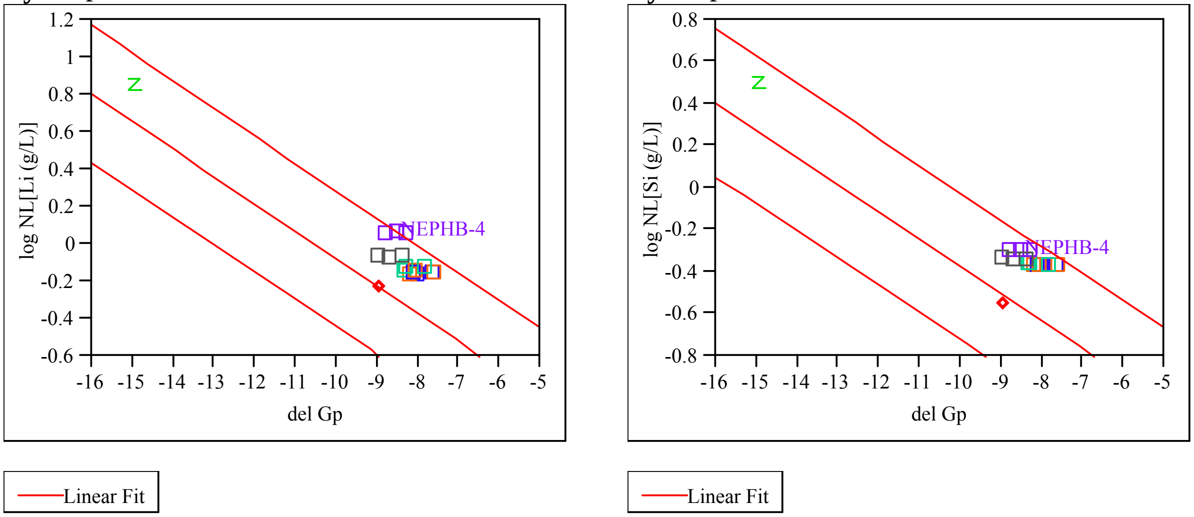
Bivariate Fit of log NL[Si (g/L)] By ΔG_P

Exhibit E12. ΔG_P Predictions versus Common Logarithm Normalized Leachate (log NL[.]) for B, Li, Na, and Si Over All Compositional Views for ccc Glasses



Bivariate Fit of log NL[B (g/L)] By ΔG_P

Bivariate Fit of log NL[Na (g/L)] By ΔG_P



Bivariate Fit of log NL[Li (g/L)] By ΔG_P

Bivariate Fit of log NL[Si (g/L)] By ΔG_P