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Hanford Enhanced Waste Glass Characterization: Influence of Composition on Chemical Durability

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EXECUTIVE SUMMARY

This report provides a review of the complete high-level waste (HLW) and low-activity waste (LAW) data sets for the glasses recently fabricated at Pacific Northwest National Laboratory and characterized at Savannah River National Laboratory (SRNL). The review is from the perspective of relating the chemical durability performance to the compositions of these study glasses, since the characterization work at SRNL focused on chemical analysis and ASTM Product Consistency Test (PCT) performance.

The review showed that some elements were leached congruently from the HLW glasses, while others were not. In general, boron and sodium were leached congruently from the HLW glasses, while silicon was not released congruently with boron. This may be indicative of amorphous phase separation in the glasses, the formation of crystalline phases, or formation of colloidal solids in the leachate. Further characterization is needed to understand the differences in silicon leaching, particularly since durability model development may be challenging for an inhomogeneous glass.

Leaching of silicon from the LAW glasses was better correlated with that of the other elements measured. Leaching of elements from the canister centerline cooled (CCC) LAW glasses was somewhat less congruent than that from the quenched versions of the glasses. This may indicate that the formation of secondary phases during the slow cooling resulted in partitioning of the elements among phases with varying durability responses. This should be further investigated to support the durability modeling effort.

Partitioning of the HLW and LAW glass compositions was used to provide further perspective on the influence of glass composition on chemical durability. In general, those CCC HLW glasses with higher concentrations of Na_2O and Al_2O_3, and lower concentrations of B_2O_3 had poorer chemical durability. Previous studies[1-6] have shown that this is indicative of nepheline crystallization ($NaAlSiO_4$) during the CCC heat treatment, resulting in reduced chemical durability. Further characterization should be performed to confirm the formation of crystalline phases during the CCC heat treatment. For the quenched and CCC LAW glasses, Li_2O, ZrO_2, V_2O_5, and SnO_2 concentrations were shown to impact chemical durability. Higher concentrations of Li_2O and ZrO_2 reduced the durability of the quenched glasses. Higher concentrations of V_2O_5 combined with lower concentrations of SnO_2 reduced the durability of the CCC glasses, as did higher Li_2O concentrations. These results should be leveraged for further studies given that these components are additives intended to control the properties and performance of the LAW glass.

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LIST OF ABBREVIATIONS

CCC	Canister Centerline Cooled
DOE	U.S. Department of Energy
EA	Environmental Assessment Glass
EGCR	Experimental Glass Composition Region
HLW	High-Level Waste
ILAW	Immobilized Low-Activity Waste
LAW	Low-Activity Waste
NC_B	Normalized Concentration of Boron
ORP	U.S. Department of Energy, Office of River Protection
PCT	Product Consistency Test
PNNL	Pacific Northwest National Laboratory
REML	Restricted Maximum Likelihood
SRNL	Savannah River National Laboratory
WTP	Hanford Tank Waste Treatment and Immobilization Plant

1.0 Introduction

The U.S. Department of Energy (DOE), Office of River Protection (ORP) is developing advanced waste glass formulations and models to allow for increased waste loading in glass to be produced at the Hanford Tank Waste Treatment and Immobilization Plant (WTP).[7,8] As part of this program, simulated high-level waste (HLW) and low-activity waste (LAW) glasses have been selected,[9,10] fabricated,[11-13] and characterized[14-18] in order to expand the glass composition and property data available for developing process control models for high waste loaded glasses.

The intent of this report is to provide a review of the complete HLW and LAW data sets for the glasses recently fabricated at Pacific Northwest National Laboratory (PNNL) and characterized at Savannah River National Laboratory (SRNL). The HLW glasses targeted the high alumina region of the Hanford HLW experimental glass composition region (EGCR).[7] The LAW glasses targeted the high waste loading region of the Hanford LAW EGCR.[8] This review is from the perspective of relating the chemical durability performance to the compositions of these study glasses, since the characterization work at SRNL focused on chemical analysis and ASTM Product Consistency Test (PCT)[19] performance.

2.0 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Savannah River Site Manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. Laboratory data for this study were recorded in the SRNL Electronic Laboratory Notebook system, experiments C3489-00079.

3.0 High-Level Waste Glasses

SRNL characterized a total of 50 simulated HLW glass compositions fabricated by PNNL and the results have been reported earlier, in three groups.[14-16] In most cases, both quenched versions of the glasses and versions of the glasses heat treated to represent WTP canister centerline cooling (CCC) were provided. The quenched glasses were characterized for chemical composition, and both the quenched and CCC versions of the glasses were characterized for chemical durability via the PCT. Both the targeted and the measured glass compositions were used in normalizing the reported PCT results.

The complete set of PCT results was reviewed to explore relationships among the normalized concentration values (defined as NC_i in units of $g_{waste\ form}/L_{leachant}$ for the element, *i*, of interest)[19] for the elements tracked during the PCT. This review showed that some elements were leached congruently from the glasses, while others were not. For example, Figure 3-1 presents plots of the normalized concentration of boron (NC_B) versus NC_{Na} (left) and NC_{Si} (right) for the quenched HLW glasses. The plots show that, in general, boron and sodium were leached congruently from the quenched HLW glasses, while silicon was not released congruently with boron. Figure 3-2 presents similar plots for the CCC versions of the HLW glasses. Again, boron and sodium were generally leached congruently, while silicon was not released congruently with boron. In all cases, the observations are the same regardless of whether the PCT data are normalized to the targeted or measured glass compositions. There are multiple factors that may have contributed to this response, individually or in combination:

- Dissolved silicon may have remained adhered to the ground glasses as a gel or reaction layer after the PCT, rather than having dissolved in the leachate.
- Silicon may have formed colloidal solids in the leachate. Colloids larger than 0.45 um would have been excluded from analysis when the leachate was filtered.
- Some of the glasses may have been phase separated (two or more amorphous phases), with the boron and alkali partitioning to a phase that was more easily leached, and with the silicon remaining in a more durable phase. Note however that the relatively high Al_2O_3 concentrations

of these glasses would be expected to suppress immiscibility, at least in terms of a simplified B_2O_3-R_2O-SiO_2 glass.

- The formation of crystalline phases, which were identified via Scanning Electron Microscopy in many of the quenched glasses during composition measurements,[14,15] likely altered the composition of the residual glass and may have led to further inhomogeneity.

These potential factors should be taken into account when developing a model relating PCT performance to the compositions of the glasses. In particular, the glasses should be further characterized to determine whether liquid-liquid phase separation occurred, since modeling may be challenging for an inhomogeneous glass. The known presence of crystalline phases in the glasses[14,15] may also complicate a modeling effort.

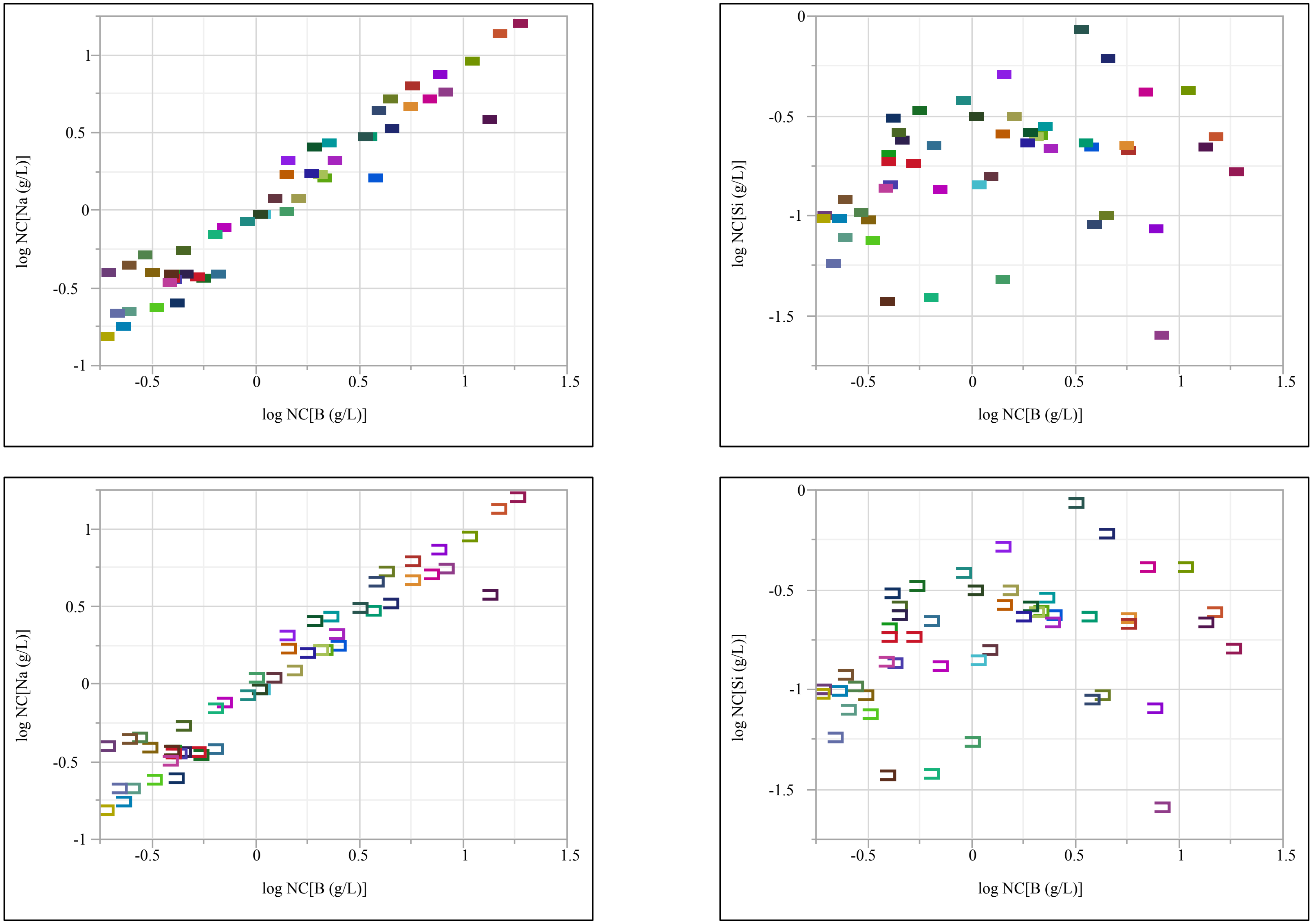

Figure 3-1. Plots of NC_{Na} versus NC_B (left) and NC_{Si} versus NC_B (right) for the quenched HLW glasses, normalized using the targeted (closed markers) and measured (open markers) compositions

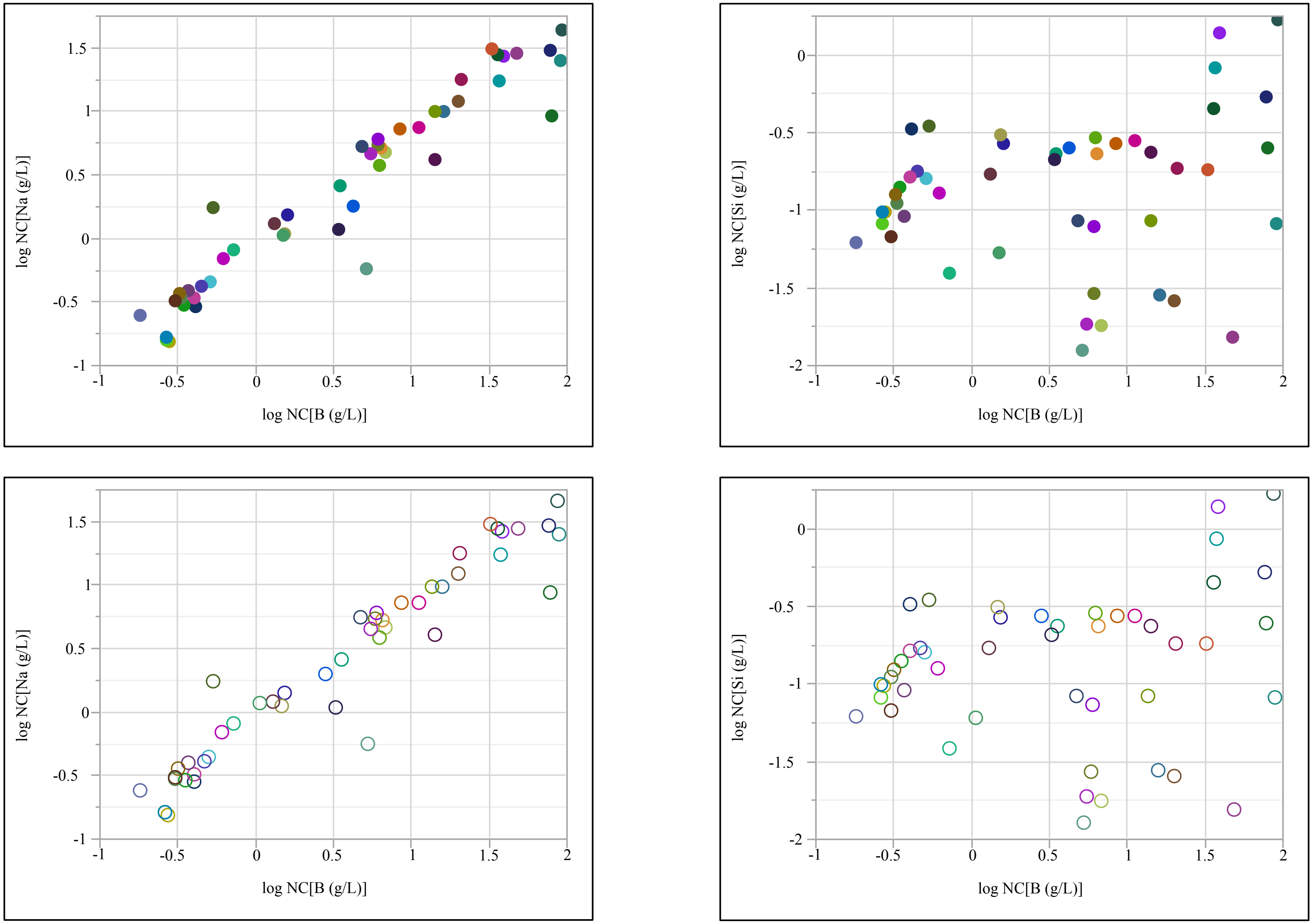

Figure 3-2. Plots of NC_{Na} versus NC_B (left) and NC_{Si} versus NC_B (right) for the CCC HLW glasses, normalized using the targeted (closed markers) and measured (open markers) compositions

A partitioning routine in JMP Pro Version 11.2.1 (SAS Institute, Inc.)[20] was used to further review the PCT results as a function of glass composition. To set up the partitioning routine, the PCT results for the HLW glasses[a] were grouped according to whether the NC_B was greater than the Environmental Assessment (EA) glass benchmark of 16.695 g/L.[21] As shown in Table 3-1, only one of the quenched glasses had an NC_B value greater than 16.695 g/L, while 11 of the CCC glasses had NC_B values greater than 16.695 g/L. Therefore, the partitioning was focused on the CCC glasses to identify the influence of composition on their PCT responses.

Table 3-1. Grouping of HLW Glasses by PCT Response Relative to that of the EA Glass

Heat Treatment	Compositional View	NC_B < EA (16.695 g/L)	Number of HLW Glasses
Quenched	measured	No	1
Quenched	measured	Yes	50
CCC	measured	No	11
CCC	measured	Yes	36

Figure 3-3 shows the results of partitioning the compositions of the CCC HLW glasses based on whether their NC_B values were greater than that of the EA benchmark glass. The routine first split the group of 47 compositions at a Na_2O concentration of 8.334 wt %. The CCC glasses that contained less than 8.334 wt % Na_2O all had NC_B values that were less than that of the EA benchmark glass. The routine next split the glasses based on B_2O_3 concentration. Eleven glasses had Na_2O concentrations greater than or equal to 8.334 wt % and B_2O_3 concentrations greater than or equal to 14.626 wt %. Of these, one glass had a NC_B value that was greater than that of the EA benchmark glass. The final split based on RuO_2 concentration is likely of little value, since the measured concentrations of RuO_2 in the glasses were typically below detection limits. The group of glasses with B_2O_3 concentrations of less than 14.626 wt % B_2O_3 was further split based on Al_2O_3 concentration of the 14 glasses, where the 8 glasses with Al_2O_3 concentrations greater than 16.585 wt % had NC_B values that were greater than that of the EA benchmark glass.

In general, the partitioning in Figure 3-3 shows that those CCC HLW glasses with higher concentrations of Na_2O and Al_2O_3, and lower concentrations of B_2O_3, had poorer chemical durability as determined by the PCT. Previous studies[1-6] have shown that these composition trends and the reduction in durability are indicative of nepheline crystallization ($NaAlSiO_4$) during the CCC heat treatment. Further characterization should be performed to confirm the formation of crystalline phases during the CCC heat treatment.

[a] The measured glass compositions were selected for normalizing the PCT data for this evaluation since compositional view (targeted or measured) had no practical influence on the outcome.

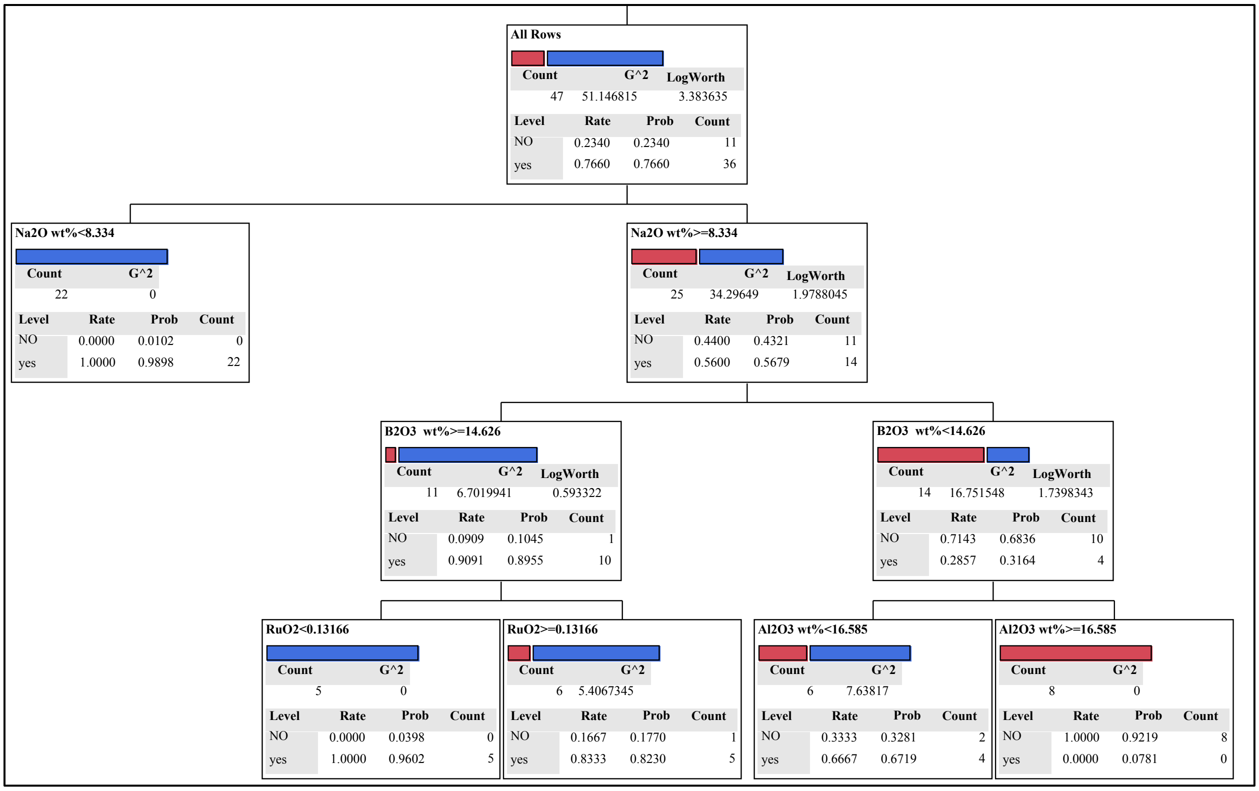

Figure 3-3. Partitioning of the HLW Glass Compositions Based on PCT Response after the CCC Heat Treatment

4.0 Low-Activity Waste Glasses

SRNL characterized a total of 36 simulated LAW glass compositions fabricated by PNNL and the results have been reported earlier, in two groups.[17,18] In most cases, both quenched versions of the glasses and versions of the glasses heat treated to represent the WTP CCC were provided. The quenched glasses were characterized for chemical composition, and both the quenched and CCC versions of the glasses were characterized for chemical durability via the PCT. Both the targeted and the measured glass compositions were used in normalizing the reported PCT results. Some of the LAW glasses were subjected to more than one type of CCC heat treatment at PNNL, with variation in the starting temperature. For the purposes of this review, all of the CCC heat treatments are treated as a single group.

The complete set of LAW glass PCT results was reviewed to explore relationships among the normalized concentration values for the elements tracked during the PCT. In contrast with the results for the HLW glasses, the leaching of all the elements, including silicon, from the LAW glasses was generally congruent. Exhibit A-1 in Appendix A presents the complete results of congruent dissolution evaluations for the quenched versions of the LAW glasses, with the PCT results normalized to both the targeted and measured glass compositions. The exhibit includes the calculated correlation values along with a graphical representation of the data. Leaching among boron, lithium, sodium, and silicon is reasonably congruent, with the smallest correlation being between the normalized concentration (NC_i) values for lithium and silicon (0.8167, where a value of 1 would represent perfect correlation), with the normalization using the measured glass compositions. Exhibit A-2 in Appendix A presents the results for the CCC versions of the LAW glasses. A review of this exhibit shows that the normalized concentrations for the four elements are somewhat less correlated than those of the quenched versions of the glasses (with the exception of boron and sodium). This may indicate that the formation of secondary phases during the slow cooling resulted in partitioning of the elements among phases with varying durability responses. The lowest correlation is again between lithium and silicon (0.6799), with the normalization using the measured glass compositions.

Of most significance is that leaching of silicon is fairly well correlated with leaching of the other elements for the LAW glasses (particularly for the quenched versions), which contrasts with the behavior of silicon in the HLW glasses. This may indicate better homogeneity of the LAW glasses. As with the HLW glasses, additional characterization to determine the homogeneity of the LAW glasses, both before and after the CCC heat treatment, is recommended to support the development of a predictive durability model. The formation of secondary phases in the glasses may add complexity to the modeling effort.

A partitioning routine in JMP Pro Version 11.2.1 (SAS Institute, Inc.)[20] was used to further review the PCT results as a function of glass composition. To set up the partitioning routine, the PCT results for the LAW glasses (normalized by the measured glass compositions) were grouped according to whether the NC_B value for each glass was greater than the WTP contractual specification for immobilized low-activity waste (ILAW) of 2.0 g/m^2 (4 g/L).[a] The results of this grouping are shown in Table 4-1. Seven of the quenched glasses and 14 of the CCC glasses had NC_B values, normalized using the measured compositions of the glasses, that were greater than 4 g/L. The partitioning routine was completed for both the quenched and CCC LAW glasses.

Table 4-1. Grouping of LAW Glasses by PCT Response Relative to the WTP ILAW PCT Specification

Heat Treatment	Compositional View	NC_B < 4 g/L	Number of LAW Glasses
Quenched	measured	No	7
Quenched	measured	Yes	29
CCC	measured	No	14
CCC	measured	Yes	28

Figure 4-1 shows the results of partitioning the compositions of the quenched LAW glasses based on whether their NC_B values (normalized by the measured compositions of the glasses) were greater than 4 g/L. The routine first split the group of 36 compositions at a Li_2O concentration of 3.251 wt %. The quenched glasses that contained less than 3.251 wt % Li_2O all had NC_B values that were less than 4 g/L. The routine split the other 17 glasses based on ZrO_2 concentration, where the five glasses with ZrO_2 concentrations greater than or equal to 4.336 wt % had NC_B values greater than 4 g/L. A final split was made based on Cr_2O_3 concentrations, where some of the glasses with Cr_2O_3 concentrations greater than 0.218 wt % had NC_B values greater than 4 g/L. The partitioning results for the quenched LAW glasses may be of interest for further studies in that Li_2O and ZrO_2, added as glass forming chemicals, may have a negative influence on chemical durability of the glass if added in larger amounts.

Figure 4-2 shows the results of partitioning the compositions of the CCC LAW glasses based on whether their NC_B values (normalized by the measured compositions of the glasses) were greater than 4 g/L. Li_2O concentration was again identified as the first factor in splitting the glasses based on PCT response, although the division was not as clear as it was for the quenched glasses. The concentrations of V_2O_5 and SnO_2 were identified as further split factors. In general, higher Li_2O concentrations, or higher V_2O_5 concentrations coupled with lower SnO_2 concentrations seemed to lead to poor chemical durability after

[a] WTP Contract DE-AC27-01RV14136, U.S. Department of Energy

the CCC heat treatment. These results may be of interest for further studies given that Li_2O, V_2O_5, and SnO_2 are all additives intended to control the properties and performance of the LAW glass.

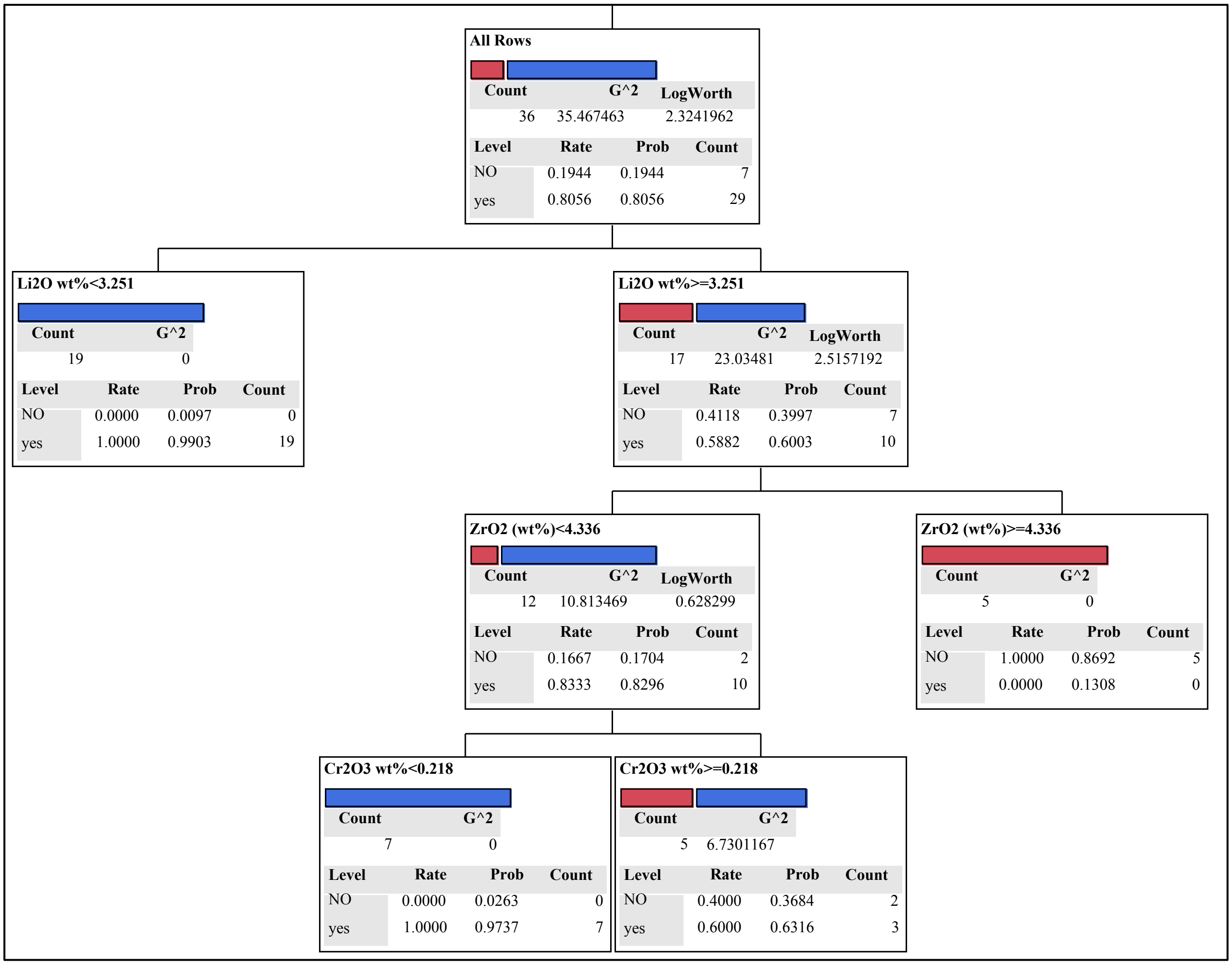

Figure 4-1. Partitioning of the Quenched LAW Glass Compositions Based on PCT Response

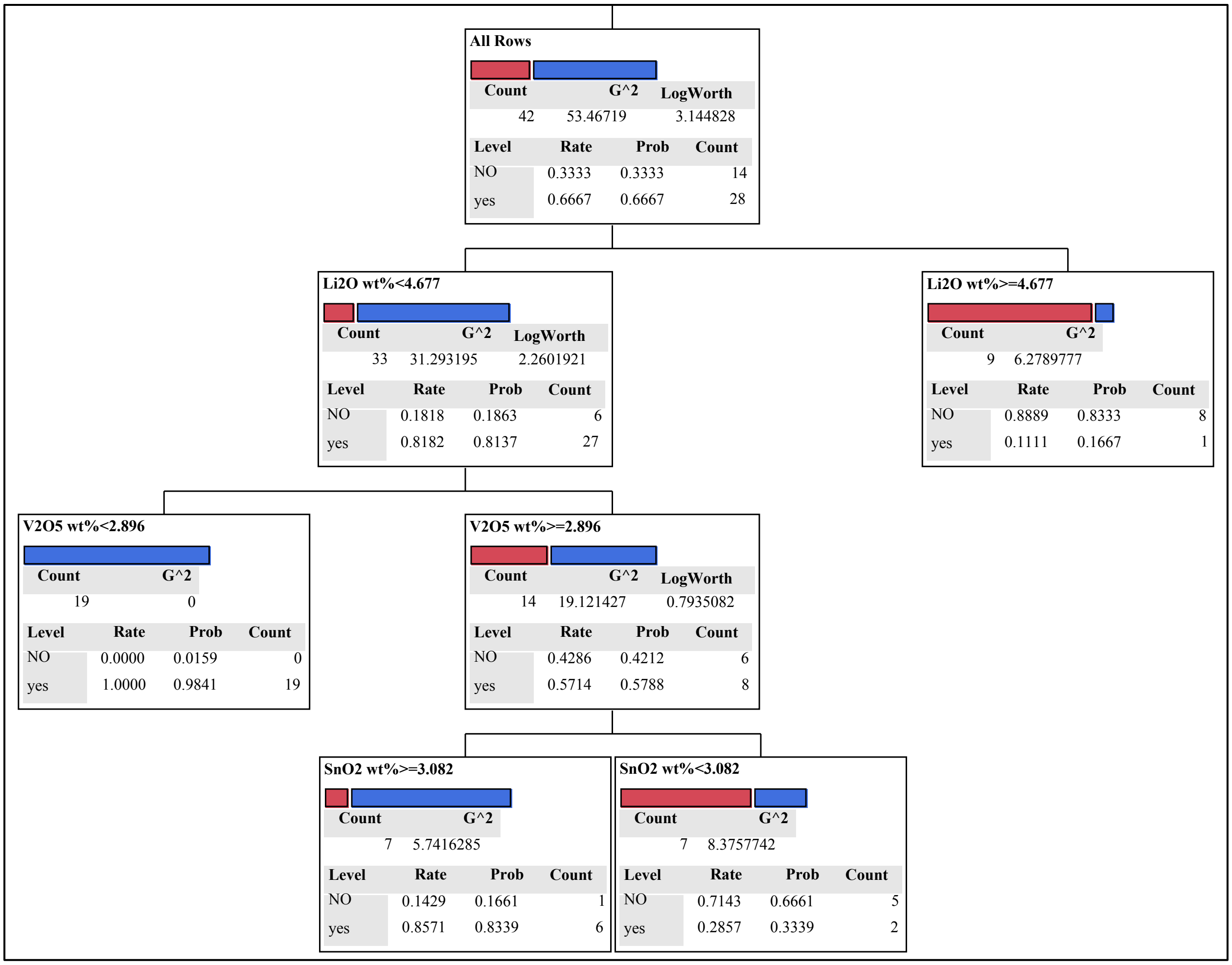

Figure 4-2. Partitioning of the LAW Glass Compositions Based on PCT Response after the CCC Heat Treatment

5.0 Summary

This report provides a review of the complete HLW and LAW data sets for the glasses recently fabricated at PNNL and characterized at SRNL. The HLW glasses targeted the high alumina region of the Hanford HLW EGCR.[7] The LAW glasses targeted the high waste loading region of the Hanford LAW EGCR.[8] The review is from the perspective of relating the chemical durability performance to the compositions of these study glasses, since the characterization work at SRNL focused on chemical analysis and PCT performance.

The review showed that some elements were leached congruently from the HLW glasses, while others were not. In general, boron and sodium were leached congruently from the HLW glasses, while silicon was not released congruently with boron. This may be indicative of amorphous phase separation in the glasses, the formation of crystalline phases, or formation of colloidal solids in the leachate. Further characterization is needed to understand the differences in silicon leaching, particularly since durability model development may be challenging for an inhomogeneous glass.

Leaching of silicon from the LAW glasses was better correlated with that of the other elements measured. Leaching of elements from the CCC LAW glasses was somewhat less congruent as compared to leaching of the quenched versions of the glasses. This may indicate that the formation of secondary phases during the slow cooling resulted in partitioning of the elements among phases with varying durability responses. This should be further investigated to support the durability modeling effort.

Partitioning of the HLW and LAW glass compositions was used to provide further perspective on the influence of glass composition on chemical durability. In general, those CCC HLW glasses with higher concentrations of Na_2O and Al_2O_3, and lower concentrations of B_2O_3 had poorer chemical durability. Previous studies have shown that these composition trends and the reduction in durability are indicative of nepheline crystallization ($NaAlSiO_4$) during the CCC heat treatment. Further characterization should be performed to confirm the formation of crystalline phases during the CCC heat treatment. For the quenched and CCC LAW glasses, Li_2O, ZrO_2, V_2O_5, and SnO_2 concentrations were shown to impact chemical durability. Higher concentrations of Li_2O and ZrO_2 reduced the durability of the quenched glasses. Higher concentrations of V_2O_5 combined with lower concentrations of SnO_2 reduced the durability of the CCC glasses, as did higher Li_2O concentrations. These results should be leveraged for further studies given that these components are additives intended to control the properties and performance of the LAW glass.

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Appendix A Exhibits Supporting Congruent Dissolution Evaluations for LAW Glasses

Exhibit A-1. Congruent Dissolution Evaluations for the Quenched LAW Glasses

Multivariate, Comp View=targeted, Heat Treatment=Quenched
Correlations:

	log NC[B (g/L)]	log NC[Li(g/L)]	log NC[Na (g/L)]	log NC[Si (g/L)]
log NC[B (g/L)]	1.0000	0.9789	0.9608	0.9113
log NC[Li(g/L)]	0.9789	1.0000	0.9769	0.8995
log NC[Na (g/L)]	0.9608	0.9769	1.0000	0.9259
log NC[Si (g/L)]	0.9113	0.8995	0.9259	1.0000

Note that there are 9 missing values in this group (those glasses with no targeted concentration of Li_2O). REML (restricted maximum likelihood) estimates for the correlations are determined by JMP™ when the data set has missing values.

Scatterplot Matrix

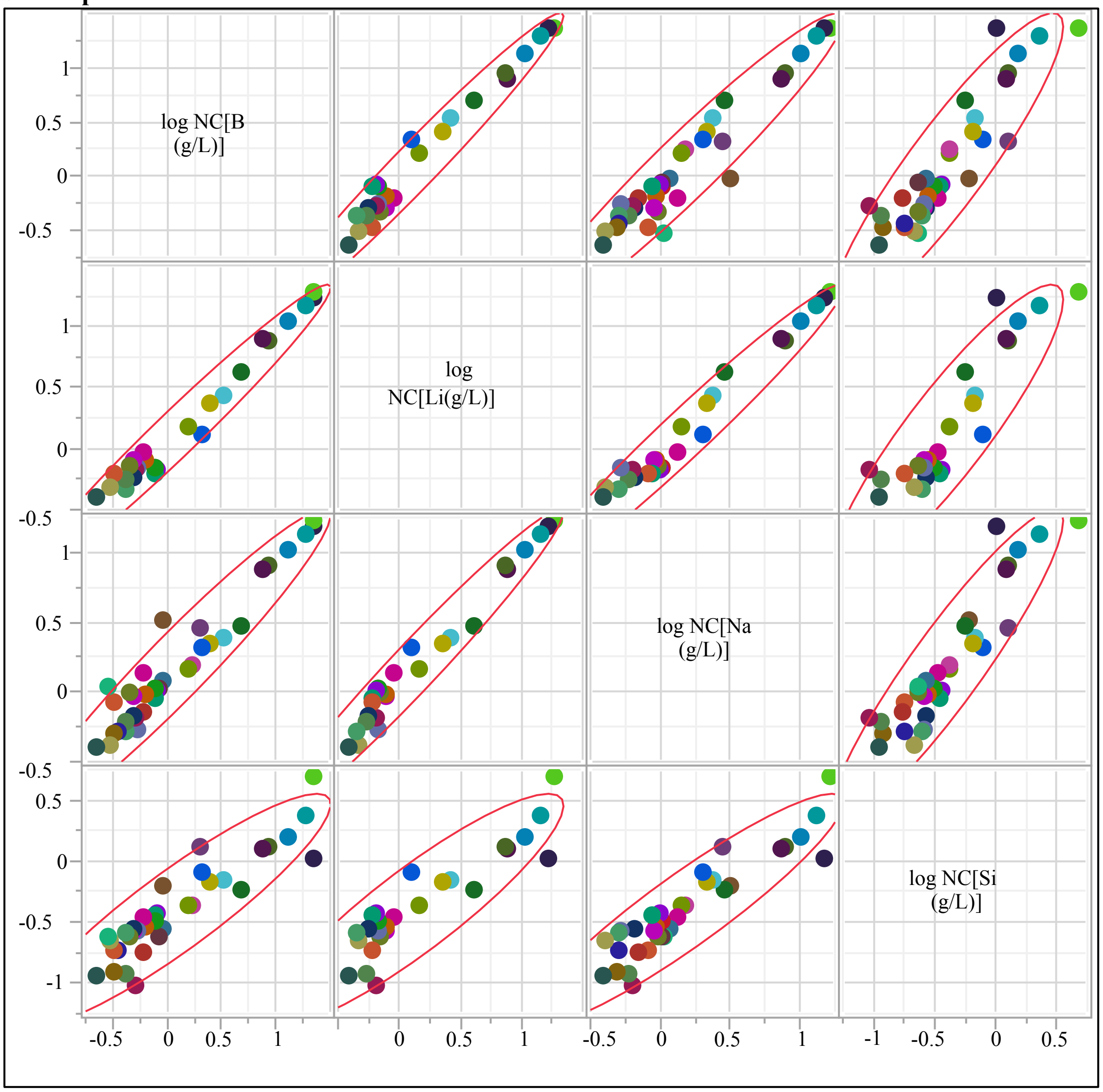


Exhibit A-1. Congruent Dissolution Evaluations for the Quenched LAW Glasses (continued)

Multivariate, Comp View=measured, Heat Treatment=quenched
Correlations:

	log NC[B (g/L)]	log NC[Li(g/L)]	log NC[Na (g/L)]	log NC[Si (g/L)]
log NC[B (g/L)]	1.0000	0.9199	0.9623	0.9108
log NC[Li(g/L)]	0.9199	1.0000	0.9316	0.8167
log NC[Na (g/L)]	0.9623	0.9316	1.0000	0.9201
log NC[Si (g/L)]	0.9108	0.8167	0.9201	1.0000

Scatterplot Matrix

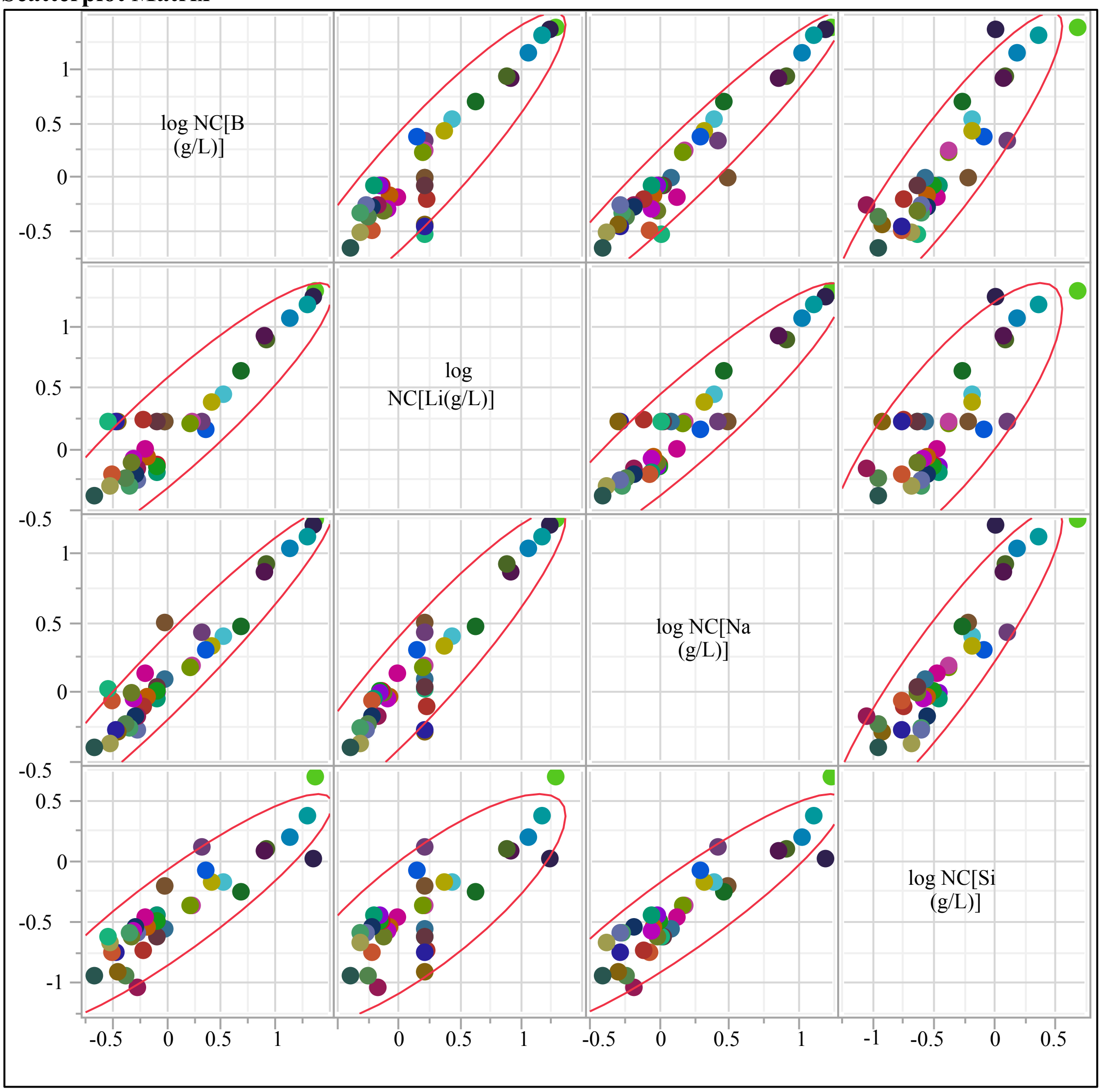


Exhibit A-2. Congruent Dissolution Evaluations for the CCC LAW Glasses

Multivariate, Comp View=targeted, Heat Treatment=CCC
Correlations:

	log NC[B (g/L)]	log NC[Li(g/L)]	log NC[Na (g/L)]	log NC[Si (g/L)]
log NC[B (g/L)]	1.0000	0.9827	0.9737	0.7900
log NC[Li(g/L)]	0.9827	1.0000	0.9713	0.8362
log NC[Na (g/L)]	0.9737	0.9713	1.0000	0.8388
log NC[Si (g/L)]	0.7900	0.8362	0.8388	1.0000

Note that there are 11 missing values in this group (those glasses with no targeted concentration of Li_2O). REML (restricted maximum likelihood) estimates for the correlations are determined by JMP™ when the data set has missing values.

Scatterplot Matrix

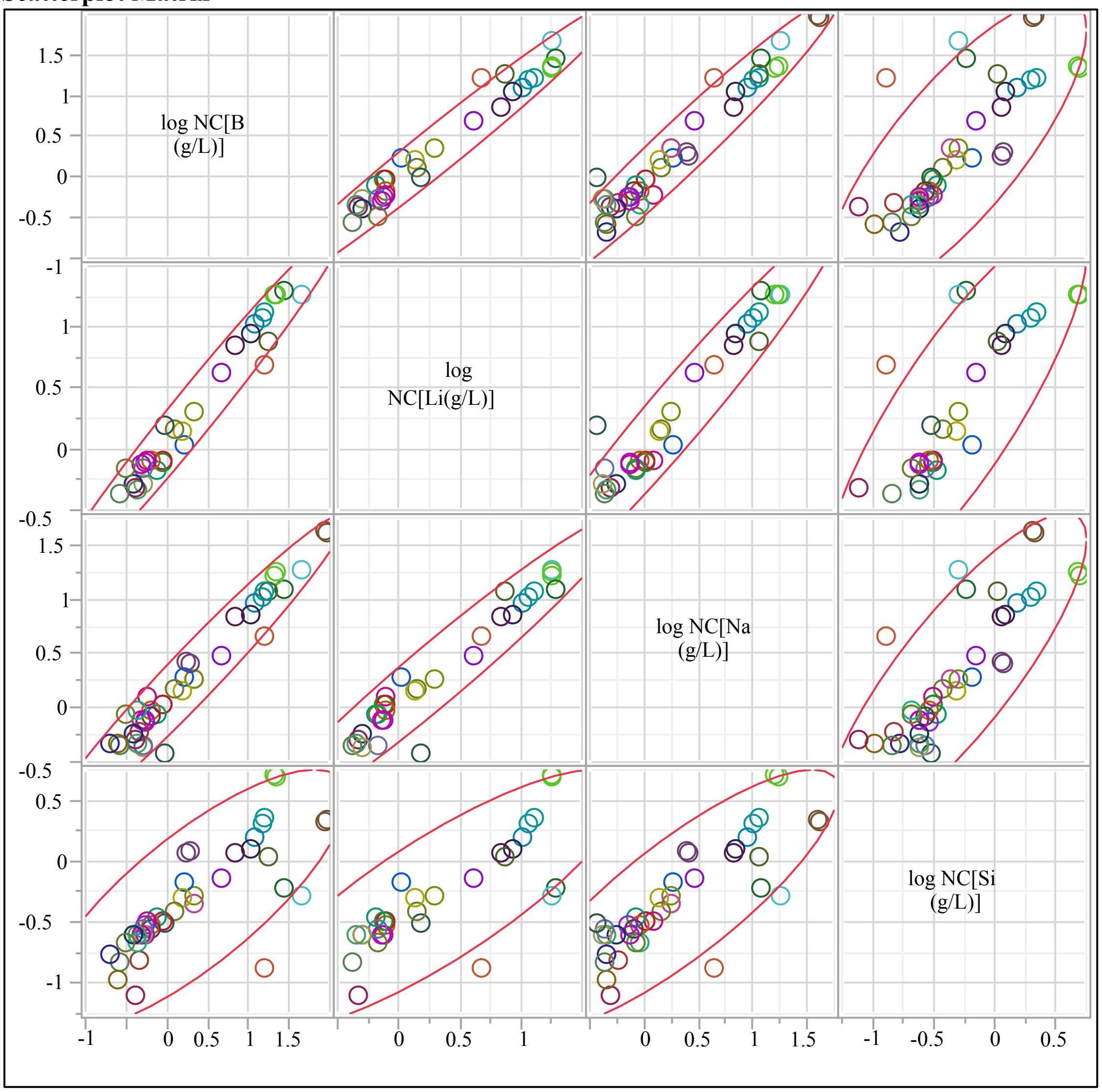
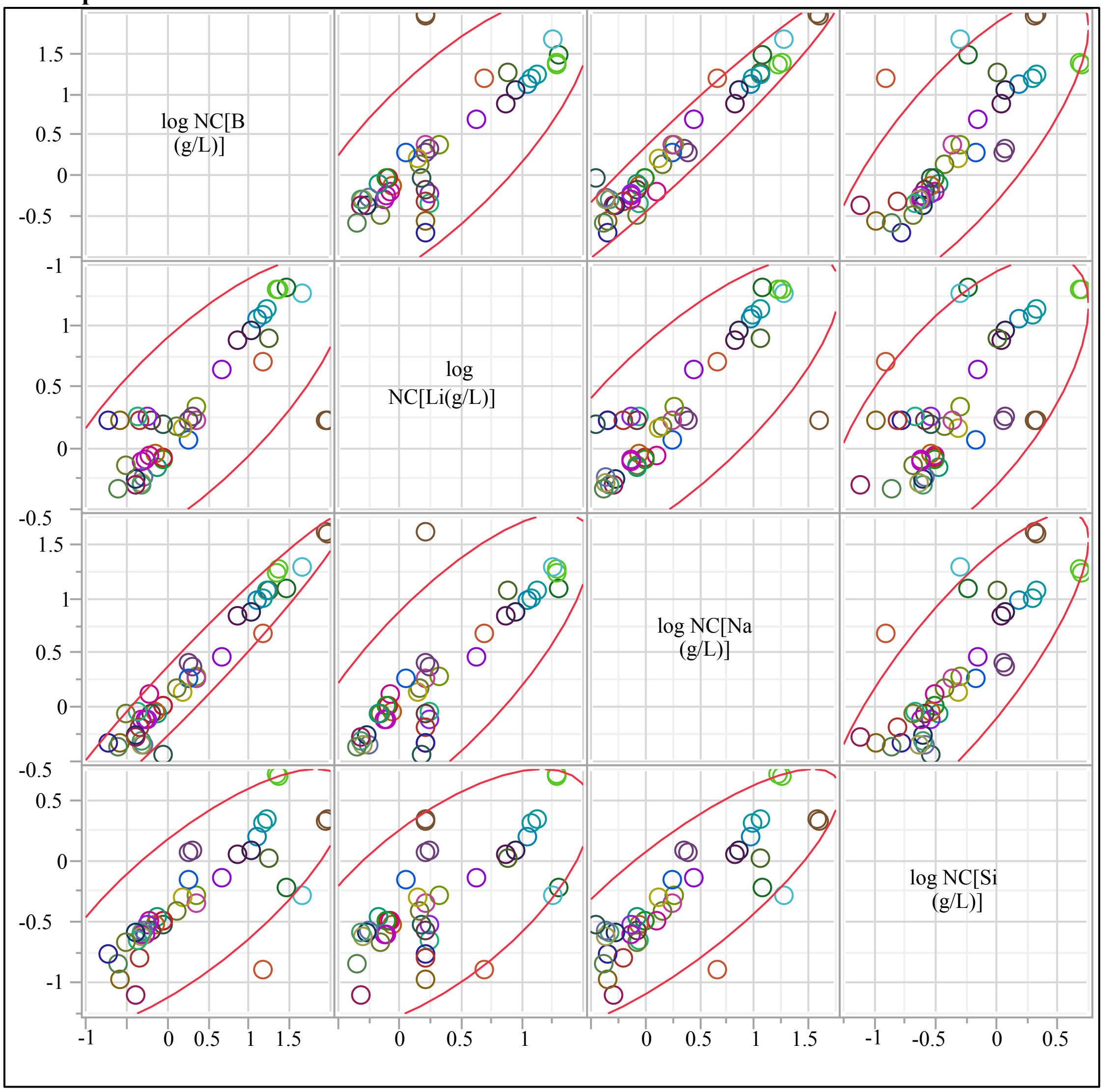


Exhibit A-2. Congruent Dissolution Evaluations for the CCC LAW Glasses (continued)

Multivariate, Comp View=measured, Heat Treatment=CCC
Correlations:

	log NC[B (g/L)]	log NC[Li(g/L)]	log NC[Na (g/L)]	log NC[Si (g/L)]
log NC[B (g/L)]	1.0000	0.7849	0.9759	0.7900
log NC[Li(g/L)]	0.7849	1.0000	0.7934	0.6799
log NC[Na (g/L)]	0.9759	0.7934	1.0000	0.8327
log NC[Si (g/L)]	0.7900	0.6799	0.8327	1.0000

Scatterplot Matrix



Distribution:

J. W. Amoroso, 999-W
T. B. Brown, 773-A
H. H. Burns, 773-41A
A. S. Choi, 999-W
Y. S. Chou, PNNL
A. D. Cozzi, 999-W
C. L. Crawford, 773-42A
D. E. Dooley, 999-W
A. P. Fellinger. 773-42A
S. D. Fink, 773-A
K. M. Fox, 999-W
E. K. Hansen, 999-W
C. C. Herman, 773-A
E. N. Hoffman, 999-W
J. E. Hyatt, 773-A
C. M. Jantzen, 773-A
F. C. Johnson, 999-W
D. S. Kim, PNNL
A. A. Kruger, DOE-ORP
D. J. McCabe, 773-42A
D. L. McClane, 999-W
D. H. Miller, 999-W
D. K. Peeler, PNNL
F. M. Pennebaker, 773-42A
M. R. Poirier, 773-42A
R. L. Russell, PNNL
M. J. Schweiger, PNNL
M. E. Stone, 999-W
C. L. Trivelpiece, 999-W
J. D. Vienna, PNNL
B. J. Wiedenman, 773-42A
W. R. Wilmarth, 773-A
Records Administration (EDWS)