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EWG-RPT-017, Rev 0

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**May 2018**

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U.S. DEPARTMENT OF ENERGY

Prepared for the U.S. Department of Energy
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# Summary

Hanford high-level waste (HLW) contains relatively high concentrations of $Al_2O_3$. A major constraint limiting the waste loading of high-$Al_2O_3$ glasses is nepheline (nominally $NaAlSiO_4$) formation upon slow cooling of HLW glasses after melts are poured into steel canisters. The model currently planned to be used at the Hanford Tank Waste Treatment and Immobilization Plant (WTP) for avoiding nepheline formation is too conservative and drastically limits waste loading.[1] To increase loadings of high-$Al_2O_3$ waste at the WTP, the effects of glass composition on glass properties must be determined and glass property-composition models must be developed.[2] Determining the impacts of glass composition on nepheline formation and the effects of nepheline on Product Consistency Test (PCT) response is an important part of this effort.

As a part of this task, data related to the Hanford high-$Al_2O_3$ HLW composition region were generated to supplement existing data for model development. Twenty-six glasses were fabricated, heat treated, analyzed for crystallinity, and tested for PCT response. The heat treatment was designed to mimic the canister centerline cooling (CCC) profile of Hanford HLW canisters.[3] X-ray diffraction was used to quantify the crystal fractions of the CCC samples. The PCT was performed at the Savannah River National Laboratory and normalized concentrations of B, Li, Na, and Si in the leachate (g/L) reported.[4] Additionally, composition analyses of the glasses were performed to support comparing the targeted and analyzed compositions of each glass.[4]

A review of the data showed a strong correlation between the fraction of nepheline formed during CCC and PCT responses. Generally, as the fraction of nepheline increased, so did the PCT leachate concentrations of B, Na, and Li.

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[1] Vienna, JD, D-S Kim, DC Skorski, and J Matyas. 2013. *Glass Property Models and Constraints for Estimating the Glass to Be Produced at Hanford by Implementing Current Advanced Glass Formulation Efforts.* PNNL-22631, (ORP-58289) Rev. 1, Pacific Northwest National Laboratory, Richland, Washington. Available at http://www.pnnl.gov/main/publications/external/technical_reports/PNNL-22631Rev1.pdf.

[2] Peeler, DK, JD Vienna, MJ Schweiger, and KM Fox. 2015. *Advanced High-Level Waste Glass Research and Development Plan.* PNNL-24450, Pacific Northwest National Laboratory Richland, Washington. Available at http://www.pnnl.gov/main/publications/external/technical_reports/PNNL-24450.pdf.

[3] Petkus, L. October 19, 2003. “Canister Centerline Cooling Data, 24590-PADC-F00029 Rev 1.” *Memorandum to C. Musick.* River Protection Project, Waste Treatment Plant,. Richland, Washington.

[4] Fox, KM, TB Edwards, ME Caldwell, and WT Riley. 2018. *Chemical Composition Analysis and Product Consistency Tests of the ORP Phase 5 Nepheline Study Glasse*. SRNL-STI-2017-00759, Rev. 0, Savannah River National Laboratory, Aiken, South Carolina.

# Acronyms and Abbreviations

| | |
|---|---|
| 2CAT | two-components-at-a-time |
| 3CAT | three-components-at-a-time |
| AD | acid dissolution |
| ARM-1 | Approved Reference Material number one |
| BDL | below detectable limit |
| CC | collection code |
| CCC | canister centerline cooling |
| DOE | U.S. Department of Energy |
| DWPF | Defense Waste Processing Facility |
| EA | environmental assessment |
| EU | eucryptite |
| EWG | enhanced waste glass |
| FIO | for information only |
| HLW | high-level waste |
| ICP-OES | inductively coupled plasma – optical emission spectroscopy |
| ICSD | Inorganic Crystal Structure Database |
| LAW | low-activity waste |
| LRM | low-activity reference material |
| ND | nepheline discriminator |
| NN | neural network |
| NP | nepheline |
| NQAP | PNNL Nuclear Quality Assurance Program |
| OB | optical basicity |
| OCAT | one-component-at-a-time |
| ORP | U.S. Department of Energy, Office of River Protection |
| PCT | Product Consistency Test |
| PF | peroxide fusion |
| PNNL | Pacific Northwest National Laboratory |
| QA | quality assurance |
| SRNL | Savannah River National Laboratory |
| WTP | Hanford Tank Waste Treatment and Immobilization Plant |
| XRD | x-ray diffraction |

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# 1.0 Introduction

The U.S. Department of Energy (DOE) stores roughly 200,000 $m^3$ of radioactive waste in underground tanks on the Hanford site. The Hanford Tank Waste Treatment and Immobilization Plant (WTP) is currently being constructed to vitrify the waste in borosilicate glass. The WTP will segregate high-level waste (HLW) and low-activity waste (LAW) fractions, blend each with glass forming chemicals, melt the mixtures at ~1150°C, and then pour the molten glasses into stainless steel canisters to cool and solidify (DOE 2000).

According to the 2008 HLW feed vector reported by Vienna et al. (2013), many HLW compositions contain high concentrations of $Al_2O_3$. The $Al_2O_3$ fraction is projected to range from roughly 10 to 70 mass% on a calcined oxide basis after caustic leaching.

The loading of high-$Al_2O_3$ HLW's may be drastically reduced by the constraint(s) applied to reduce the probability of forming nepheline (nominally $NaAlSiO_4$) upon slow cooling of glasses. Nepheline precipitation from a HLW glass during cooling is a major concern because it can reduce the durability of the resulting glass by removing three moles of glass former oxides (one mole of $Al_2O_3$ and two moles of $SiO_2$) for every mole of $Na_2O$ (Kim et al. 1995). When nepheline is present in a waste glass, it is difficult to predict the Product Consistency Test (PCT) (ASTM 2014) response (Peeler et al. 2015). It has also been documented that eucryptite ($LiAlSiO_4$) has a similar effect on glass durability (McCloy and Vienna 2010). To meet disposal requirements, nepheline formation must be avoided, or the amount of nepheline formed and its impact on the PCT response must be predicted. Being able to predict the amount of nepheline formed and the PCT response would provide a basis for specifying a constraint to avoid HLW glass compositions that would yield unacceptable PCT response.

The current model used to avoid nepheline formation is too conservative and limits waste loading (Vienna et al. 2016). A nepheline discriminator (ND) was developed to reduce the risk of nepheline precipitation during canister centerline cooling (CCC) heat treatment (Li et al. 1997). This approach is based on limiting the normalized $SiO_2$ concentration ($N_{Si}$) using the inequality

$$N_{Si} = \frac{g_{SiO_2}}{g_{SiO_2} + g_{Al_2O_3} + g_{Na_2O}} < 0.62 \qquad (1.1)$$

Here, $g_i$ is the mass fraction of the $i^{th}$ component in the glass. The ND predicts that glasses with $N_{Si} < 0.62$ are prone to nepheline crystallization during CCC heat treatment.

In an effort to reduce some of the conservatism in the ND, optical basicity (OB) was proposed as a constraint (Rodriguez et al. 2011, McCloy et al. 2011). The revised constraint allows glasses with $N_{Si} < 0.62$ as long as the OB of the melt is less than 0.55. The OB of a glass ($\Lambda_{glass}$) can be calculated from a glass composition using

$$\Lambda_{glass} = \frac{\sum_i x_i q_i \Lambda_i}{\sum_i x_i q_i} \qquad (1.2)$$

where

$q_i$ = the number of oxygen atoms in the $i^{th}$ component oxide

$x_i$ = the mole fraction of the $i^{th}$ component oxide in glass

$\Lambda_i$ = the molar basicity of the $i^{th}$ component oxide

This approach did reduce some of the conservatism, but still limits the waste loading of high-$Al_2O_3$ glasses (Vienna et al. 2016).

A neural network (NN) model has also been proposed to estimate the probability of nepheline formation for a specific glass composition (Vienna et al. 2013). This approach was selected because it can account for highly non-linear effects of components. The NN model consists of three nodes using the hyperbolic tangent (TanH) transfer function. The output from the three nodes was compared to a probability cutoff value to assign a binary response (i.e., whether nepheline formed or not) for a glass composition. However, the NN method involves complex calculations and determining the uncertainties of predictions made with the model would be difficult (Vienna et al. 2016).

To optimize waste loading, a new approach is needed to limit nepheline precipitation during slow cooling of HLW canisters. It has been demonstrated that $B_2O_3$, CaO, $Fe_2O_3$, $K_2O$, and $Li_2O$ affect nepheline formation (Li et al. 1997). A ternary sub-mixture model that takes advantage of the success of the ND while considering the effects of other components in the melt has been developed (Vienna et al. 2016).

The Phase 1 experimental design was developed around a baseline glass (BL0) roughly in the center of the predicted Hanford high-$Al_2O_3$ HLW glass composition region. The glasses in this study were formulated to meet all property constraints for fabrication in the WTP (described in Vienna et al. 2013) without any constraint for nepheline formation. A set of 15 glass compositions were then developed by adjusting $Al_2O_3$, $B_2O_3$, CaO, $Fe_2O_3$, $Li_2O$, $Na_2O$, and $SiO_2$ one-component-at-a-time (OCAT) from their values in the BL0 composition. Each OCAT change was offset by changes to the remaining glass components, keeping them in the same relative proportions as in the BL0 baseline glass, so that the component mass fractions summed to unity. All 15 glasses were fabricated, underwent CCC heat treatment, and then analyzed for the fraction of nepheline formed. Only one glass (Neph-NN-1-12) from this study precipitated nepheline after CCC heat treatment. Both quenched and CCC heat treated samples of Neph-NN-1-12 were tested for durability using the PCT. The other 14 glasses in this study were not tested for durability due to the lack of nepheline formation following CCC heat treatment. Further details about the Phase 1 study can be found in Kroll et al. (2016).

Phase 2 of the study was designed to supplement data from the Phase 1 study. Preliminary testing was done to select a new baseline composition from four formulations (BL1, BL2, BL3, and BL4). These were fabricated, subjected to the CCC heat treatment, and analyzed for nepheline fraction. BL3 was found to precipitate ~10 mass% nepheline after CCC heat treatment and was the baseline glass composition selected for Phase 2. Thirteen glasses were formulated by adjusting $Al_2O_3$, $B_2O_3$, $Li_2O$, $Na_2O$, and $SiO_2$ OCAT from their values in the BL3 baseline composition, resulting in 14 unique glasses (including BL3). Only two glasses (NP2-Very High Si and NP2-Low Na) from Phase 2 were found to not precipitate nepheline following CCC heat treatment, however one of these (NP2-Low Na) did precipitate eucryptite.

Consequently, the durability of the quenched and CCC versions of all 14 glasses from Phase 2 were evaluated by the PCT. Further details about the Phase 2 study can also be found in Kroll et al. (2016).

Results from Phase 2 provided some insight to the HLW glass composition region over which nepheline did and did not precipitate, as well as the effects of glass composition on the fraction of nepheline formed during CCC heat treatment. These results suggested that the fraction of nepheline in the CCC glass was not easily described by first-order composition effects (i.e., linear combinations of component concentrations).

Phase 3 was designed to investigate the fraction of nepheline formed during CCC heat treatment when changing two-components-at-a-time (2CAT) and three-components-at-a-time (3CAT). The same components ($Al_2O_3$, $B_2O_3$, $Li_2O$, $Na_2O$, and $SiO_2$) adjusted in the Phase 2 study and baseline glass (BL3) were also used here. For this phase of the study, all possible combinations of components varied 2CAT and 3CAT were generated. The 2CAT and 3CAT adjustments were offset by changes to the remaining glass components, keeping them in the same relative proportions as in the BL3 baseline glass (as done in previously described OCAT studies). An *optimal experimental design* approach (Atkinson et al. 2007) was chosen to optimally augment existing glass compositions to select a set of 16 new glasses for testing (further details on this design are described in Kroll et al. 2016) . Of the 16 glasses in the Phase 3 test matrix, 11 precipitated nepheline after CCC heat treatment. Quenched and CCC heat treated samples of all 16 glasses were subjected to the PCT. Further details about the Phase 3 study can be found in Kroll et al. (2016).

The Phase 4 matrix was statistically designed to cover the Hanford high-alumina HLW glass composition region as efficiently as possible using an extreme vertices mixture design. Unlike Phases 1 through 3, this test matrix was not specifically targeted at investigating nepheline crystallization. The Phase 4 matrix consisted of 45 glass compositions with the concentrations of $Al_2O_3$, $B_2O_3$, $Bi_2O_3$, CaO, $Cr_2O_3$, $Fe_2O_3$, Li2O, MgO, MnO, $Na_2O$, $P_2O_5$, $SiO_2$, and $ZrO_2$ varied all-at-a-time. Test results showed that 14 of the 45 glasses from the Phase 4 matrix precipitated nepheline after CCC heat treatment (Vienna et al. 2014).

The sub-mixture model uses a polynomial discriminating curve to divide glasses that precipitated nepheline during slow cooling from those that do not and was initially fit using 657 existing waste glasses from the literature (summarized by Vienna et al. 2016). Phases 1 – 4 described above added 90 new compositions to the data set and the model was further refined using logistic regression (Vienna et al. 2016). The logistic regression fitting approach allows the sub-mixture model to predict the probability of nepheline formation, whereas the polynomial discriminating curve provided a binary (yes/no) response (Vienna et al. 2016).

Further data focused on the Hanford high-alumina HLW composition region are needed to refine and validate this model or to develop improved models. This report describes the testing and results associated with the newly designed Phase 5 study. The results will be used to supplement existing data to predict the formation of nepheline, the amount of nepheline formed, and/or the PCT responses of glasses forming nepheline.

# 2.0 Quality Assurance

## 2.1 PNNL Quality Assurance Program

The PNNL Quality Assurance (QA) Program was adhered to during the conduct of the Phase 5 of this study. This program is based on the requirements as defined in the DOE Order 414.1D, *Quality Assurance*, and 10 CFR 830, *Energy/Nuclear Safety Management*, Subpart A, *Quality Assurance Requirements* (a.k.a., the Quality Rule). PNNL has chosen to implement the following consensus standards in a graded approach:

- ASME NQA-1-2000, *Quality Assurance Requirements for Nuclear Facility Applications*, Part I, "Requirements for Quality Assurance Programs for Nuclear Facilities"
- ASME NQA-1-2000, Part II, Subpart 2.7, "Quality Assurance Requirements for Computer Software for Nuclear Facility Applications," including problem reporting and corrective action
- ASME NQA-1-2000, Part IV, Subpart 4.2, "Guidance on Graded Application of Quality Assurance (QA) for Nuclear-Related Research and Development."

The PNNL *Quality Assurance Program Description/Quality Management M&O Program Description* describes the laboratory-level QA program that applies to all work performed by PNNL. Laboratory-level procedures for implementing the QA requirements described in the standards identified above are deployed through PNNL's web-based "How Do I…?" system, which is a standards-based system for managing and deploying requirements and procedures to PNNL staff. The procedures (called Workflows and Work Controls) provide detailed guidance for performing some types of tasks, such as protecting classified information and procuring items and services, as well as general guidelines for performing research-related tasks, such as preparing and reviewing calculations and calibrating and controlling measuring and test equipment.

## 2.2 Enhanced Waste Glass Quality Assurance Program

The Enhanced Waste Glass (EWG) project adhered to the PNNL Nuclear Quality Assurance Program (NQAP). This QA program is compliant with ASME-NQA-1-2012, 10 CFR 830, Subpart A, DOE Order (O) 414.1D, and is for use by research and development (R&D) projects and programs that are compatible with the editions of ASME NQA-1 2000 through 2012. The work described in this report was performed to the QA level of Applied Research, which is defined below:

> *"…nuclear and non-nuclear R&D (work activities or deliverables) that are processes initiated-with-the-intent of solving a specific problem or meeting a practical need. For applied research activities, grading is minimal and largely contingent upon the complexity of the research and the ability to duplicate the research if data were lost. The elements of QA grading, including the level of documentation, were applied to the program-, project-, and task-levels."*

The HLW nepheline study Phase 5 test matrix was generated under the NQAP QA program. The generation of the Phase 5 test matrix and glass batchsheets are documented in Calculation Packages EWG-CCP-024 (for the BL3 baseline glass only), EWG-CCP-031, EWG-CCP-048, and EWG-CCP-049. The calculations performed to construct Figure 6.1a-c and Figure 6.2 are documented in EWG-CCP-068.

Experimental procedures and analytical results for the Phase 5 nepheline study are documented in EWG-TI-0044 and EWG-TI-0052. All of this work was performed under the test plan EWG-TP-0033.

# 3.0 Experimental Design

This section discusses the experimental design for the Phase 5 study work for high-$Al_2O_3$ HLW glasses to (i) investigate the effects of HLW glass composition on nepheline formation, (ii) generate data to develop and validate models for nepheline formation and nepheline fraction, and (iii) investigate the effects of nepheline formation on glass durability by the PCT.

Phase 5 used a statically designed matrix targeting the HLW glass composition region prone to nepheline formation after CCC heat treatment. Here, JMP® version 13.0.0 (SAS Institute Inc. 2016) software was used to generate a test matrix of 27 glasses using space-filling technique (rather than the extreme vertices design used in Phase 4). This experimental design was chosen to augment previous data by spreading the nine components that were selected to vary throughout the Hanford high-alumina HLW composition region as evenly as possible. The nine components include $Al_2O_3$, $B_2O_3$, CaO, $Fe_2O_3$, $Li_2O$, $Na_2O$, $P_2O_5$, $SiO_2$, and an "Others" mixture. These nine components were varied all-at-a-time. Single- and multi- component constraints were used to set bounds on component concentrations and limit melt viscosity. Additionally, a replicate of BL3, the baseline glass used in the Phase 2 and Phase 3 studies, was also fabricated.

The single-component constraints that limit the lower and upper bounds of each of the nine varied components are listed in Table 3.1. The composition of the "Others" mixture is presented in Table 3.2. The values in Tables 3.1 and 3.2 are in mass fraction.

**Table 3.1.** Single Component Constraints Limiting Component Concentrations (in mass fraction)

| Component | Lower Limit | Upper Limit |
|---|---|---|
| $Al_2O_3$ | 0.2000 | 0.3000 |
| $B_2O_3$ | 0.1275 | 0.2300 |
| CaO | 0.0000 | 0.0700 |
| $Fe_2O_3$ | 0.0175 | 0.0600 |
| $Li_2O$ | 0.0000 | 0.0600 |
| $Na_2O$ | 0.0800 | 0.1500 |
| $P_2O_5$ | 0.0000 | 0.0200 |
| $SiO_2$ | 0.2250 | 0.3300 |
| Others | 0.0500 | 0.1500 |

**Table 3.2.** Composition of "Others" Mixture (in mass fraction)

| Component | Mass Fraction |
|---|---|
| $Bi_2O_3$ | 0.1740 |
| $Cr_2O_3$ | 0.1619 |
| F | 0.0559 |
| MnO | 0.3415 |
| NiO | 0.0541 |
| PbO | 0.0275 |
| $RuO_2$ | 0.0014 |
| $SO_3$ | 0.0574 |
| SrO | 0.0268 |
| $ZrO_2$ | 0.0996 |

The multi-component constraint was implemented to limit the natural logarithm of the melt viscosity (in Pascal-seconds) at 1150°C in the range of 1.39 to 2.30 (4 to 10 Pa·s). The glass compositions generated are shown in Table 3.3; these values are also in mass fraction. The composition of BL3 (replicate of the baseline glass from the Phase 2 and 3 studies) is also listed in Table 3.3.

**Table 3.3.** Targeted Glass Compositions (in mass fraction) for Nepheline Phase 5 Study Including BL3

| Glass ID | $Al_2O_3$ | $B_2O_3$ | CaO | $Fe_2O_3$ | $Li_2O$ | $Na_2O$ | $SiO_2$ | $P_2O_5$ | $Bi_2O_3$ | $Cr_2O_3$ | F | MnO | NiO | PbO | $RuO_2$ | $SO_3$ | SrO | $ZrO_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| NP5-01 | 0.2231 | 0.2112 | 0.0500 | 0.0506 | 0.0000 | 0.1333 | 0.2614 | 0.0095 | 0.0106 | 0.0098 | 0.0034 | 0.0208 | 0.0033 | 0.0017 | 0.0001 | 0.0035 | 0.0016 | 0.0061 |
| NP5-02 | 0.2396 | 0.2004 | 0.0198 | 0.0530 | 0.0247 | 0.0850 | 0.2504 | 0.0166 | 0.0192 | 0.0179 | 0.0062 | 0.0377 | 0.0060 | 0.0030 | 0.0002 | 0.0063 | 0.0030 | 0.0110 |
| NP5-03 | 0.2223 | 0.2280 | 0.0062 | 0.0595 | 0.0097 | 0.0949 | 0.2280 | 0.0031 | 0.0258 | 0.0240 | 0.0083 | 0.0506 | 0.0080 | 0.0041 | 0.0002 | 0.0085 | 0.0040 | 0.0148 |
| NP5-04 | 0.2020 | 0.1933 | 0.0169 | 0.0387 | 0.0385 | 0.1112 | 0.3145 | 0.0190 | 0.0114 | 0.0106 | 0.0037 | 0.0225 | 0.0036 | 0.0018 | 0.0001 | 0.0038 | 0.0018 | 0.0066 |
| NP5-05 | 0.2830 | 0.1503 | 0.0440 | 0.0350 | 0.0491 | 0.0808 | 0.2594 | 0.0189 | 0.0138 | 0.0129 | 0.0044 | 0.0272 | 0.0043 | 0.0022 | 0.0001 | 0.0046 | 0.0021 | 0.0079 |
| NP5-06 | 0.2350 | 0.2199 | 0.0070 | 0.0412 | 0.0457 | 0.0829 | 0.2985 | 0.0072 | 0.0109 | 0.0101 | 0.0035 | 0.0214 | 0.0034 | 0.0017 | 0.0001 | 0.0036 | 0.0017 | 0.0062 |
| NP5-07 | 0.2797 | 0.2166 | 0.0235 | 0.0284 | 0.0082 | 0.1450 | 0.2305 | 0.0156 | 0.0092 | 0.0085 | 0.0029 | 0.0180 | 0.0028 | 0.0014 | 0.0001 | 0.0030 | 0.0014 | 0.0052 |
| NP5-08 | 0.2971 | 0.1868 | 0.0044 | 0.0441 | 0.0533 | 0.1158 | 0.2317 | 0.0114 | 0.0096 | 0.0090 | 0.0031 | 0.0189 | 0.0030 | 0.0015 | 0.0001 | 0.0032 | 0.0015 | 0.0055 |
| NP5-09 | 0.2539 | 0.1821 | 0.0229 | 0.0311 | 0.0423 | 0.1208 | 0.2547 | 0.0066 | 0.0149 | 0.0139 | 0.0048 | 0.0292 | 0.0046 | 0.0024 | 0.0001 | 0.0049 | 0.0023 | 0.0085 |
| NP5-10 | 0.2457 | 0.1282 | 0.0571 | 0.0564 | 0.0403 | 0.1155 | 0.2700 | 0.0168 | 0.0122 | 0.0113 | 0.0039 | 0.0239 | 0.0038 | 0.0019 | 0.0001 | 0.0040 | 0.0019 | 0.0070 |
| NP5-11 | 0.2050 | 0.1374 | 0.0401 | 0.0208 | 0.0479 | 0.1328 | 0.3265 | 0.0126 | 0.0134 | 0.0124 | 0.0043 | 0.0262 | 0.0042 | 0.0021 | 0.0001 | 0.0044 | 0.0021 | 0.0077 |
| NP5-12 | 0.2022 | 0.1514 | 0.0677 | 0.0583 | 0.0272 | 0.0922 | 0.2860 | 0.0122 | 0.0179 | 0.0166 | 0.0058 | 0.0351 | 0.0056 | 0.0028 | 0.0001 | 0.0059 | 0.0028 | 0.0102 |
| NP5-13 | 0.2107 | 0.1468 | 0.0647 | 0.0444 | 0.0103 | 0.1358 | 0.2382 | 0.0197 | 0.0225 | 0.0209 | 0.0072 | 0.0442 | 0.0070 | 0.0036 | 0.0002 | 0.0074 | 0.0035 | 0.0129 |
| NP5-14 | 0.2170 | 0.1762 | 0.0007 | 0.0487 | 0.0322 | 0.1430 | 0.2766 | 0.0140 | 0.0159 | 0.0148 | 0.0051 | 0.0313 | 0.0050 | 0.0025 | 0.0001 | 0.0053 | 0.0025 | 0.0091 |
| NP5-15 | 0.2089 | 0.1608 | 0.0083 | 0.0551 | 0.0585 | 0.0998 | 0.3200 | 0.0049 | 0.0146 | 0.0136 | 0.0047 | 0.0286 | 0.0045 | 0.0023 | 0.0001 | 0.0048 | 0.0022 | 0.0083 |
| NP5-16 | 0.2747 | 0.1349 | 0.0342 | 0.0531 | 0.0348 | 0.1468 | 0.2364 | 0.0061 | 0.0137 | 0.0128 | 0.0044 | 0.0270 | 0.0043 | 0.0022 | 0.0001 | 0.0045 | 0.0021 | 0.0079 |
| NP5-17 | 0.2583 | 0.2227 | 0.0574 | 0.0234 | 0.0135 | 0.0813 | 0.2356 | 0.0058 | 0.0178 | 0.0165 | 0.0057 | 0.0348 | 0.0055 | 0.0028 | 0.0001 | 0.0059 | 0.0027 | 0.0102 |
| NP5-18 | 0.2135 | 0.2002 | 0.0593 | 0.0256 | 0.0160 | 0.1226 | 0.3118 | 0.0002 | 0.0088 | 0.0082 | 0.0028 | 0.0173 | 0.0028 | 0.0014 | 0.0001 | 0.0029 | 0.0014 | 0.0051 |
| NP5-19 | 0.2243 | 0.1381 | 0.0248 | 0.0377 | 0.0292 | 0.1349 | 0.2899 | 0.0014 | 0.0208 | 0.0194 | 0.0067 | 0.0408 | 0.0065 | 0.0033 | 0.0002 | 0.0069 | 0.0032 | 0.0119 |
| NP5-20 | 0.2061 | 0.1649 | 0.0322 | 0.0404 | 0.0335 | 0.1033 | 0.2691 | 0.0109 | 0.0243 | 0.0226 | 0.0078 | 0.0477 | 0.0076 | 0.0038 | 0.0002 | 0.0080 | 0.0037 | 0.0139 |
| NP5-21 | 0.2588 | 0.1353 | 0.0205 | 0.0261 | 0.0591 | 0.0933 | 0.2433 | 0.0194 | 0.0251 | 0.0233 | 0.0081 | 0.0492 | 0.0078 | 0.0040 | 0.0002 | 0.0083 | 0.0039 | 0.0143 |
| NP5-22 | 0.2291 | 0.1850 | 0.0516 | 0.0191 | 0.0120 | 0.1085 | 0.2455 | 0.0135 | 0.0236 | 0.0220 | 0.0076 | 0.0464 | 0.0073 | 0.0037 | 0.0002 | 0.0078 | 0.0036 | 0.0135 |
| NP5-23 | 0.2866 | 0.1730 | 0.0658 | 0.0196 | 0.0289 | 0.1145 | 0.2269 | 0.0003 | 0.0147 | 0.0137 | 0.0047 | 0.0288 | 0.0046 | 0.0023 | 0.0001 | 0.0048 | 0.0023 | 0.0084 |
| NP5-24 | 0.2442 | 0.1741 | 0.0308 | 0.0183 | 0.0205 | 0.1499 | 0.3022 | 0.0056 | 0.0095 | 0.0088 | 0.0030 | 0.0186 | 0.0029 | 0.0015 | 0.0001 | 0.0031 | 0.0015 | 0.0054 |
| NP5-25 | 0.2895 | 0.1287 | 0.0146 | 0.0176 | 0.0563 | 0.1302 | 0.2645 | 0.0036 | 0.0165 | 0.0154 | 0.0053 | 0.0325 | 0.0051 | 0.0026 | 0.0001 | 0.0055 | 0.0025 | 0.0095 |
| NP5-26 | 0.2079 | 0.2081 | 0.0134 | 0.0218 | 0.0051 | 0.1440 | 0.2630 | 0.0022 | 0.0234 | 0.0218 | 0.0075 | 0.0459 | 0.0073 | 0.0037 | 0.0002 | 0.0077 | 0.0036 | 0.0134 |
| NP5-27 | 0.2670 | 0.1657 | 0.0473 | 0.0225 | 0.0528 | 0.1008 | 0.2816 | 0.0010 | 0.0107 | 0.0099 | 0.0034 | 0.0210 | 0.0033 | 0.0017 | 0.0001 | 0.0035 | 0.0016 | 0.0061 |
| BL3 | 0.2850 | 0.1720 | 0.0065 | 0.0250 | 0.0500 | 0.1250 | 0.2935 | 0.0070 | 0.0065 | 0.0110 | 0.0030 | 0.0100 | 0.0000 | 0.0000 | 0.0005 | 0.0025 | 0.0000 | 0.0025 |

# 4.0 Experimental Methods

## 4.1 Glass Fabrication

The simulated HLW glasses were prepared in 200 g batches and melted in Pt-10% Rh crucibles. Oxides ($Bi_2O_3$, $Cr_2O_3$, $Fe_2O_3$, MnO, NiO, PbO, $SiO_2$, and $ZrO_2$) and carbonates ($CaCO_3$, $Li_2CO_3$, $Na_2CO_3$, and $SrCO_3$) comprised most of the batch additives. NaF, $NaPO_3$, and $Na_2SO_4$ were used as the sources of F, $P_2O_5$, and $SO_3$, respectively. Boric acid was used as the $B_2O_3$ source, $Al(OH)_3$ was chosen as the aluminum additive, and $RuNO(NO_3)_3$ was the source of $RuO_2$.

For glass fabrication, 1.5% $RuNO(NO_3)_3$ solution was added dropwise to pre-weighed $SiO_2$ on a large watch glass and dried at 90°C for at least one hour. The dried $RuNO(NO_3)_3$ on $SiO_2$ was then combined with the appropriate amount of each source chemical. The mixture was placed in an agate mill with an agate puck on a vibratory fixture for four minutes. The batch was loaded into a Pt-10% Rh crucible in two to four separate additions that were allowed to melt down for ~10 minutes at 1150°C in a preheated furnace before the subsequent addition. After the entire batch was added, a Pt-10% Rh lid was placed on the crucible and the melt was allowed to dwell for ~1 hour. The melt was quenched by pouring onto a stainless steel plate. The glass was then ground in a tungsten carbide mill for four minutes and re-melted using the same melt process. All of the glasses in this study were melted twice at 1150°C for ~1 hour each.

## 4.2 Canister Centerline Cooling Heat Treatment

To perform the CCC heat treatment, a crushed sample of quenched glass with particles sized roughly 5 mm in diameter was placed in either a 1.2 × 1.2 × 1.2 cm or a 2.54 × 2.54 × 2.54 cm Pt-10% Rh foil crucible with a tight-fitting Pt-10% Rh foil lid. The glass-loaded crucibles were placed in a furnace preheated to the melt temperature (1150°C) and allowed to dwell for 30 minutes. Then, the furnace temperature was quickly reduced to 1050°C (at an estimated rate of -12.5°C/minute) and cooled as prescribed in Table 4.1.

**Table 4.1.** CCC Heat Treatment Schedule

| Start-Stop Temp (°C) | Rate (°C/hour) |
|---|---|
| 1050-980 | -93.4 |
| 980-930 | -48.4 |
| 930-875 | -35.5 |
| 875-825 | -23.3 |
| 825-775 | -15.2 |
| 775-725 | -16.7 |
| 725-400 | -18.2 |

When the furnace temperature was below 400°C (below the glass transition temperature), the furnace power was turned off and the glass was allowed to cool naturally to room temperature.

To prepare enough sample material for the PCT measurements (sub-sample IDs ending with "-CCC-PCT" in Table 5.1, which is discussed in Section 5.1), the quenched glass-loaded crucibles were placed in a furnace preheated to the glass melt temperature for ~5 minutes to allow the crushed glass to melt. Then, the crucible was removed from the furnace and additional pieces of quenched glass added before continuing with the heat treatment as described above.

## 4.3 X-Ray Diffraction Analysis

X-ray diffraction (XRD) was performed to measure the phase assemblage in the CCC heat-treated samples and some of the quenched samples. For the CCC samples, the entire heat-treated glass specimen was used for analysis. These 1 to 3 g pieces were ground for ~15 s in a tungsten carbide mill, spiked with ~5.0 mass% of NIST 674b ZnO as an internal standard, and then milled for another ~45 s. The sub-sample IDs ending with -CCC-PCT or -Q-PCT in Table 5.1 (XRD results table, discussed in Section 5.1) were prepped for analysis from the excess (untested) fines returned from PCT sample preparation at Savannah River National Laboratory (SRNL) (Fox et al. 2018). The fines were spiked with ~5.0 mass% ZnO as an internal standard and then milled for ~45 s in a tungsten carbide mill. The powdered samples were then loaded into plastic holders and analyzed using a Bruker D8 Advance XRD (Bruker AXS Inc., Madison, Wisconsin) with Cu-tube. The detector used was a LynxEye™ position-sensitive detector with a collection window of 3° 2θ. Scan parameters were 5 to 75° 2θ with a step of 0.015° 2θ and 0.6 s dwell at each step. Bruker AXS DIFFRACplus EVA software was used to identify the crystalline phases. TOPAS software was used to quantify the fractions of each phase using a whole-pattern fitting technique (Cheary et al. 2004).

## 4.4 Composition Analyses

Composition analysis was performed on quenched samples of each glass in the study except NP5-02 and NP5-03. Glasses NP5-02 and NP5-03 were not subjected to testing and characterization due to difficulties encountered during the melting process (described in Section 5.0). The composition analyses of the study glasses were performed by Fox et al. (2018) and the procedure used is briefly summarized here.

A sample of the quenched version of each study glass was prepared in duplicate using three techniques: sodium peroxide fusion (PF), lithium metaborate fusion (LM), and potassium hydroxide fusion (KH). The PF preparation method was selected for the measurement of the analytes Al, B, Ca, Cr, Fe, Li, Mn, Ni, P, Si, and Zr. The LM preparation method was used for measurements of Bi, Na, Pb, Ru, S, and Sr. The KH preparation was used to measure the concentration of F.

Each prepared sample was analyzed twice for the elements of interest using inductively coupled plasma–optical emission spectroscopy (ICP-OES), except for F. The concentration of F was measured by ion chromatography (IC). The IC measurements were also performed twice on each duplicate sample. This provided a total of four measurements of each element per glass. Reference materials were also measured over the course of these analyses to ensure instrument accuracy. Further details on the composition analyses are reported by Fox et al. (2018).

## 4.5 Product Consistency Test

The PCT was conducted as described by Fox et al. (2018). PCT Method-A (ASTM 2014) was performed in triplicate on the quenched and CCC heat treated samples of each study glass except NP5-02 and NP5-03 (see discussion in Section 5.0). Approved Reference Material (ARM) glass and blanks from the same vessel cleaning batch were also analyzed. Samples were ground, washed, and prepared according to the ASTM procedure. Fifteen milliliters of Type-I ASTM water were added to 1.5 g of glass in stainless steel vessels, which were then sealed and placed in a 90°C oven for seven days. After cooling, the solutions were filtered, acidified, and analyzed by ICP-OES. A multi-element standard solution was also analyzed to ensure instrument accuracy. The PCT leachate concentrations of B, Li, Na, and Si were normalized to the targeted and measured compositions using the average of the measured leachate concentrations (over the triplicate results). Further details on the PCT work on these glasses are reported by Fox et al. (2018).

Only the PCT results (B, Li, Na, and Si leachate concentrations) normalized using targeted glass compositions were used in the PNNL work described in this report. The experience at PNNL over many years using high-purity batching chemicals and careful weighing and melting procedures is that target glass compositions are generally more accurate representations of actual glass compositions than are analyzed glass compositions. This is because analyzed glass compositions are subject to varying magnitudes of uncertainty and can be subject to bias.

# 5.0 Results

Twenty-eight glasses were batched and melted in this study. This includes the 27 newly formulated glasses and a replicate of BL3. Two of the 27 new glasses (NP5-02 and NP5-03) were not fully characterized due to difficulties encountered during the melting process. Glasses NP5-02 and NP5-03 were both observed to have high viscosity making it difficult to pour the molten glass out of the crucible. Additionally, undissolved particles and high crystal content were noted for each of these glasses. Photos of the quenched glasses after two melts at 1150°C are a presented in Figure 5.1a-b.

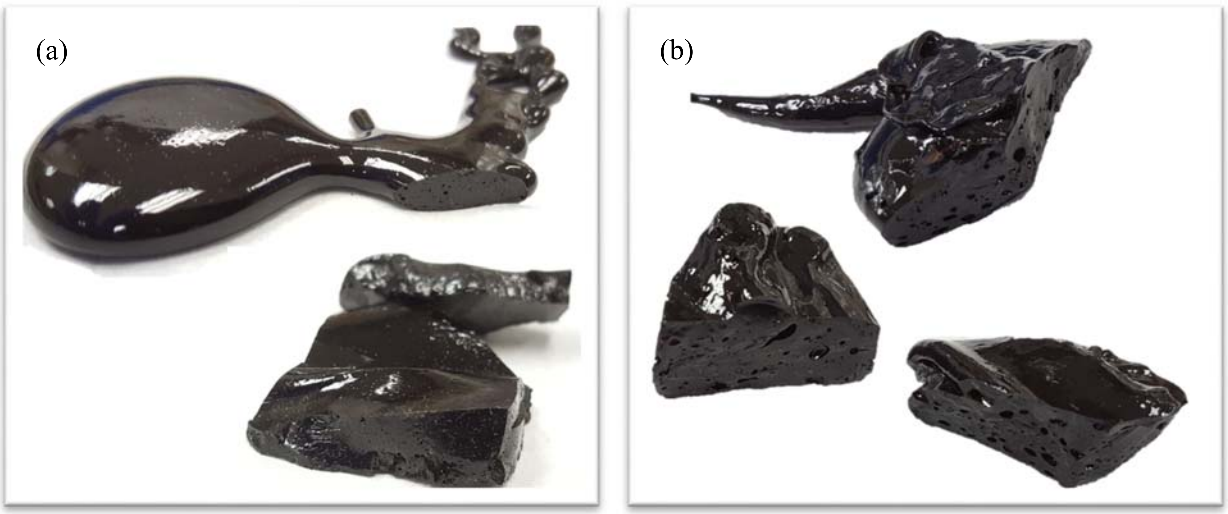


**Figure 5.1.** (a) Photo of glass NP5-02 and (b) NP5-03 after two melts at 1150°C

Bubbles and heterogeneities (likely either undissolved particles and/or crystals) are visible in the glass NP5-02 shown in Figure 5.1 (a). These same features were observed to a greater extent in glass NP5-03, which is pictured in Figure 5.1 (b). The heterogeneities observed in these glasses would make characterization troublesome. Consequently, these two glasses were abandoned and did not undergo further testing.

The quenched and CCC versions of the remaining 26 glasses (including BL3) in the Phase 5 matrix were evaluated for PCT response and analyzed for crystal fraction. Additionally, chemical compositions analysis was performed on samples of the quenched glasses. Nepheline was identified in 11 CCC glasses. The crystal fraction, PCT response, and composition analysis data are presented in this section.

## 5.1 X-Ray Diffraction Crystal Fraction Results

The quantitative crystal fraction results using XRD for all characterized glasses are summarized in Table 5.1. Glasses NP5-02 and NP5-03 were not analyzed for crystal fraction due to difficulties melting (see Section 5.0 above). In Table 5.1 the sub-sample ID can be deciphered as the GlassID-CoolingProcess-AnalysisType-ReplicateNumber. The measured nepheline and eucryptite fractions are listed in the “NP” and “EU” columns of Table 5.1, respectively.

XRD results including all major crystalline phases (≥1 mass%) identified are presented in Table A.1 in Appendix A. If available, the Inorganic Crystal Structure Database (ICSD) collection codes (CC) for the crystalline phases identified in the sample are also listed in Table A.1. That table also lists the CCC crucible size used for each sub-sample.

In Table 5.1, the sub-sample IDs ending with "-PCT" are untested powdered portions (fines collected during grinding and sieving for PCT preparation) of the sample sent for PCT. The glass powder was collected before beginning the PCT. These powders were analyzed by XRD to determine the nepheline fraction in the specific sample prepared for the PCT. XRD was performed on the quenched version of select glasses, these are identified by a "Q" in the sub-sample ID.

**Table 5.1.** XRD Crystal Content Results in Mass%

| Glass ID | Sub-Sample ID | NP[(a)] | EU[(b)] |
|---|---|---|---|
| NP5-01 | NP5-01-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-01-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-04 | NP5-04-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-04-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-05 | NP5-05-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-05-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-06 | NP5-06-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-06-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-07 | NP5-07-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-07-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-08 | NP5-08-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-08-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-09 | NP5-09-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-09-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-10 | NP5-10-CCC-XRD-1 | 56 | 0.0 |
| | NP5-10-CCC-PCT-1 | 60 | 0.0 |
| NP5-11 | NP5-11-CCC-XRD-1 | 56 | 0.0 |
| | NP5-11-CCC-PCT-1 | 58 | 0.0 |
| NP5-12 | NP5-12-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-12-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-13 | NP5-13-Q-XRD-1 | 0.0 | 0.0 |
| | NP5-13-CCC-XRD-1 | 2.8 | 0.0 |
| | NP5-13-CCC-PCT-1 | 0.88 | 0.0 |
| NP5-14 | NP5-14-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-14-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-15 | NP5-15-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-15-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-16 | NP5-16-CCC-XRD-1 | 49 | 0.0 |
| | NP5-16-CCC-PCT-1 | 51 | 0.0 |

**Table 5.1.** XRD Crystal Content Results in Mass% (cont'd)

| Glass ID | Sub-Sample ID | NP[a] | EU[b] |
|---|---|---|---|
| NP5-17 | NP5-17-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-17-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-18 | NP5-18-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-18-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-19 | NP5-19-Q-XRD-1 | 0.0 | 0.0 |
| | NP5-19-CCC-XRD-1 | 5.3 | 0.0 |
| | NP5-19-CCC-PCT-1 | 2.6 | 0.0 |
| NP5-20 | NP5-20-Q-XRD-1 | 0.0 | 0.0 |
| | NP5-20-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-20-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-21 | NP5-21-Q-XRD-1 | 0.0 | 0.0 |
| | NP5-21-CCC-XRD-1 | 19 | 0.28 |
| | NP5-21-CCC-PCT-1 | 25 | 1.0 |
| NP5-22 | NP5-22-Q-XRD-1 | 0.0 | 0.0 |
| | NP5-22-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-22-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-23 | NP5-23-CCC-XRD-1 | 8.9 | 0.0 |
| | NP5-23-CCC-PCT-1 | 11 | 0.0 |
| NP5-24 | NP5-24-CCC-XRD-1 | 4.9 | 0.0 |
| | NP5-24-CCC-PCT-1 | 4.4 | 0.0 |
| NP5-25 | NP5-25-CCC-XRD-1 | 54 | 0.0 |
| | NP5-25-CCC-PCT-1 | 61 | 0.0 |
| NP5-26 | NP5-26-Q-XRD-1 | 0.0 | 0.0 |
| | NP5-26-CCC-XRD-1 | 0.0 | 0.0 |
| | NP5-26-CCC-PCT-1 | 0.0 | 0.0 |
| NP5-27 | NP5-27-CCC-XRD-1 | 2.2 | 0.0 |
| | NP5-27-CCC-PCT-1 | 1.8 | 0.0 |
| BL3 | NP5-BL3-CCC-XRD-1 | 3.4 | 0.0 |
| | NP5-BL3-CCC-XRD-2 | 2.8 | 0.0 |
| | NP5-BL3-CCC-PCT-1 | 3.4 | 0.0 |

(a) NP = Nepheline
(b) EU = Eucryptite

## 5.2 Composition Analysis Results

The targeted and average measured oxide concentrations (mass%) in the quenched study glasses are presented in Appendix B. The composition analyses of glass samples were performed as described in Section 4.4. The following observations are summarized from composition analysis results reported by Fox et al. (2018).

- The measured concentrations of CaO are generally lower than the targeted value for glasses targeting CaO concentrations < ~2 mass%, while glasses with targeted CaO concentrations > ~2 mass% had measured concentrations above the targeted value. Five glasses (NP5-01, NP5-10, NP5-12, NP5-13, and NP5-22) containing > 5 mass% CaO were found to have greater than 10% relative difference between the measured and targeted concentrations of CaO, three of which (NP5-01, NP5-10, and NP5-12) also had greater than 10% relative difference between the measured and targeted values of $Fe_2O_3$.
- The measured concentrations of F are low for all of the study glasses, however the targeted concentrations of F are < 1 mass% for all of the study glasses.
- The measured concentrations of $Fe_2O_3$ are higher than the targeted values for all of the study glasses. Four glasses (NP5-01, NP5-10, NP5-12, and NP5-15) containing > 5 mass% $Fe_2O_3$ were found to have greater than 10% relative difference between the measured and targeted values. Three of these glasses (NP5-01, NP5-10, and NP5-12) were found to have greater than 10% relative difference between the measured and targeted values of CaO.
- The measured concentrations of $Li_2O$ are lower than the targeted values for all of the study glasses with compositions containing $Li_2O$.
- The measured concentrations of $Na_2O$ are lower than the targeted values for all of the study glasses. Five glasses (NP5-05, NP5-09, NP5-21, NP5-23, and NP5-25) containing > 5 mass% $Na_2O$ were found to have greater than 10% relative difference between the measured and targeted values.
- All measured sums of oxides for the study glasses fall within the interval of 96.4 to 102.5 mass%, indicating acceptable recovery of the glass components during digestion.

A total of 11 glasses were found to have greater than 10% relative difference between the measured and targeted values for oxides with concentrations ≥ 5 mass%. This was limited to three components (as described above): CaO, $Fe_2O_3$, and $Na_2O$. Table B.1 in Appendix B lists the average measured concentration of each component in all of the tested study glasses. Measurements below detectable limits (BDL) are identified by the less than symbol (<) in the BDL column of Table B.1.

## 5.3 Product Consistency Test Results

The PCT was performed for all of the Phase 5 study glasses except NP5-02 and NP5-03 (see discussion in Section 5.0). Glass samples for PCT were prepared as described previously in Section 4.5. The leachate concentrations of B, Li, Na, and Si were normalized using targeted compositions (denoted $NC_B$, $NC_{Li}$, $NC_{Na}$, and $NC_{Si}$) for the quenched and CCC samples. The normalized PCT concentrations are presented in Table 5.2. Normalized concentrations with a less than symbol (<) next to it indicates that the analyte was present in concentrations below the detectable limit (BDL) of the instrument.

The following observations were noted upon review of the PCT results (Fox et al. 2018).

- One quenched glass (NP5-08) has $NC_B$ values higher than the EA glass benchmark value of 16.695 g/L (Jantzen et al. 1993).

- Six CCC heat treated glasses (NP5-08, NP5-10, NP5-11, NP5-16, NP5-21, and NP5-25) have $NC_B$ values higher than the EA glass benchmark value of 16.695 g/L (Jantzen et al. 1993).

Additionally, the glass with the highest measured nepheline fraction after CCC heat treatment (NP5-11) also had the highest $NC_B$ value.

**Table 5.2.** PCT Results for Quenched and CCC Glasses Normalized to the Targeted Compositions

| Glass ID | Normalized $PCT_Q$[(a)] (g/L) | | | | Normalized $PCT_{CCC}$[(b)] (g/L) | | | |
|---|---|---|---|---|---|---|---|---|
| | B | Li | Na | Si | B | Li | Na | Si |
| NP5-01 | 1.077 | NA | 0.964 | 0.215 | 1.123 | NA | 0.967 | 0.195 |
| NP5-04 | 1.84 | 1.631 | 1.069 | 0.352 | 2.257 | 1.935 | 1.21 | 0.33 |
| NP5-05 | 0.892 | 0.935 | 0.67 | 0.242 | 0.673 | 0.728 | 0.519 | 0.242 |
| NP5-06 | 2.582 | 2.248 | 1.323 | 0.373 | 3.014 | 2.512 | 1.472 | 0.392 |
| NP5-07 | 2.38 | 2.17 | 1.822 | 0.26 | 3.323 | 2.958 | 2.276 | 0.244 |
| NP5-08 | 23.227 | 12.184 | 15.978 | < 0.019 | 18.985 | 9.358 | 12.876 | 0.031 |
| NP5-09 | 1.925 | 1.733 | 1.458 | 0.306 | 2.597 | 2.222 | 1.767 | 0.301 |
| NP5-10 | 0.412 | 0.498 | 0.493 | 0.161 | 52.605 | 55.893 | 6.418 | 0.026 |
| NP5-11 | 0.499 | 0.463 | 0.512 | 0.223 | 84.747 | 80.134 | 15.406 | 0.36 |
| NP5-12 | 0.255 | 0.353 | 0.315 | 0.091 | 0.302 | 0.354 | 0.251 | 0.081 |
| NP5-13 | 0.65 | 0.566 | 0.714 | 0.167 | 1.853 | 1.533 | 0.95 | < 0.018 |
| NP5-14 | 3.052 | 2.513 | 1.921 | 0.301 | 3.401 | 2.734 | 1.972 | 0.326 |
| NP5-15 | 1.109 | 1.073 | 0.697 | 0.416 | 1.068 | 1.031 | 0.721 | 0.45 |
| NP5-16 | 2.474 | 2.148 | 2.153 | 0.297 | 59.076 | 55.421 | 21.273 | < 0.021 |
| NP5-17 | 0.471 | 0.568 | 0.452 | 0.117 | 0.426 | 0.515 | 0.408 | 0.115 |
| NP5-18 | 0.507 | 0.558 | 0.45 | 0.169 | 0.427 | 0.486 | 0.391 | 0.16 |
| NP5-19 | 0.682 | 0.666 | 0.613 | 0.312 | 5.384 | 3.844 | 1.56 | 0.063 |
| NP5-20 | 0.775 | 0.82 | 0.644 | 0.277 | 0.822 | 0.868 | 0.66 | 0.318 |
| NP5-21 | 2.724 | 2.54 | 1.819 | 0.365 | 42.836 | 25.581 | 13.365 | 0.124 |
| NP5-22 | 0.48 | 0.552 | 0.476 | 0.142 | 0.588 | 0.687 | 0.569 | 0.205 |
| NP5-23 | 1.052 | 1.147 | 1.072 | 0.178 | 12.571 | 10.746 | 7.194 | < 0.016 |
| NP5-24 | 0.601 | 0.611 | 0.57 | 0.302 | 6.696 | 5.49 | 2.518 | < 0.015 |
| NP5-25 | 2.26 | 2.037 | 1.704 | 0.416 | 81.844 | 64.45 | 14.172 | 0.061 |
| NP5-26 | 3.273 | 3.048 | 2.281 | 0.227 | 3.309 | 3.167 | 2.309 | 0.236 |
| NP5-27 | 0.563 | 0.689 | 0.543 | 0.259 | 2.535 | 1.757 | 0.838 | 0.023 |
| BL3 | 1.632 | 1.566 | 1.04 | 0.458 | 5.562 | 3.454 | 1.947 | 0.218 |

(a) $PCT_Q$ = Product Consistency Test results for quenched glasses

(b) $PCT_{CCC}$ = Product Consistency Test results for canister centerline cooled glasses

# 6.0 Discussion and Conclusions

The Phase 5 study focused on nepheline formation during CCC of high-alumina waste glasses included formulating, fabricating, and CCC heat treating 26 glasses developed by adjusting nine components all-at-a-time using a space filling design. The test matrix initially included 28 glasses, however, two glasses (NP5-02 and NP5-03) were abandoned due to difficulties during the melting process (see discussion in Section 5.0). Additionally, one of the test glasses was a replicate of the baseline glass (BL3) used in the Phase 2 and Phase 3 studies.

Eleven of the 26 study glasses that were characterized were found to precipitate nepheline during CCC. One of these glasses also precipitated eucryptite. The amount of nepheline formed during CCC in the replicate of BL3 had a much lower measured concentration (~3 mass%) than in previous studies (~14 mass%). The cause of this discrepancy is unknown, but one possible explanation is the two differ slightly in composition. None of the composition analysis results (Table B.1) suggest a misbatch of BL3. However, when comparing the composition analysis results of BL3 in Kroll et al (2016) and the composition analysis results for this replicate, the differences in $B_2O_3$ and $Na_2O$ concentration could account for this observation. Kroll et al. (2016) reported BL3 contained -3.1% $B_2O_3$ and 5.6% $Na_2O$ relative to the targeted concentrations, while the replicate contained 9.0% $B_2O_3$ and -6.2% $Na_2O$ relative to the targeted concentrations.

In previous study phases it was observed that the -CCC-PCT results consistently showed higher nepheline fractions than the -CCC-XRD results (Kroll et al. 2016). The cause of this finding is unknown, however, Kroll et al. (2016) speculated the following slight differences between experimental parameters could explain the trend: (i) loading of quenched glass into the CCC crucible, (ii) size of the CCC crucible, and (iii) XRD sample preparation. For further details on differences between these experimental parameters see the discussion in Kroll et al. (2016). Seven of the 11 nepheline-containing CCC heat treated glasses in the Phase 5 study were also observed to follow this trend; four glasses (NP5-13, NP5-19, NP5-24, and NP5-27) did not. While it is unclear as to why this trend is observed and why four of the Phase 5 glasses did not follow the trend, it is important to mention that glasses NP5-13 and NP5-19 were observed to exhibit crystallization in the quenched glass during melting; this was confirmed by XRD (see Table A.1). Perhaps crystallization-induced heterogeneity of the quenched NP5-13 and NP5-19 glass samples can account for this deviation. As for glasses NP5-24 and NP5-27, the absolute difference between the measured nepheline fractions of the -CCC-XRD and -CCC-PCT samples were $\leq$ 0.5 mass%. Glasses NP5-24 and NP5-27 also formed relatively low amounts of nepheline during CCC (~5 mass% and ~2 mass%, respectively). When considering these observation in regards to glasses NP5-24 and NP5-27, it is reasonable to assume the numbers are within experimental error, but no attempt has been made to confirm this or determine the experimental error associated with the quantitative XRD methods used.

In general, the normalized concentrations of B, Li, and Na in the PCT leachate increased as the fraction of nepheline and eucryptite increased. The measured nepheline (NP) and eucryptite (EU) mass fractions are plotted against the PCT response for each CCC sample in Figure 6.1a-c. Each data point can be related back to Table 5.1 by the end of the sub-sample ID (shown in the legends of Figure 6.1a-c). All glasses that precipitated only nepheline are plotted in blue and the single glass with nepheline and eucryptite is plotted in red as the sum of the measured nepheline and eucryptite concentrations. These plots indicate a correlation between nepheline fraction and PCT responses. Normalized leachate concentrations of Na are generally lower and more scattered than that of B and Li; this was also noted by Kroll et al. (2016). This is likely observed because nepheline is a Na-containing crystal. As the fraction of nepheline increases, the fraction of Na in glass available for leaching is reduced. Assuming the experimental conditions of the PCT do not provide enough energy to make the decomposition of nepheline thermodynamically favorable would explain the scatter observed for Na.

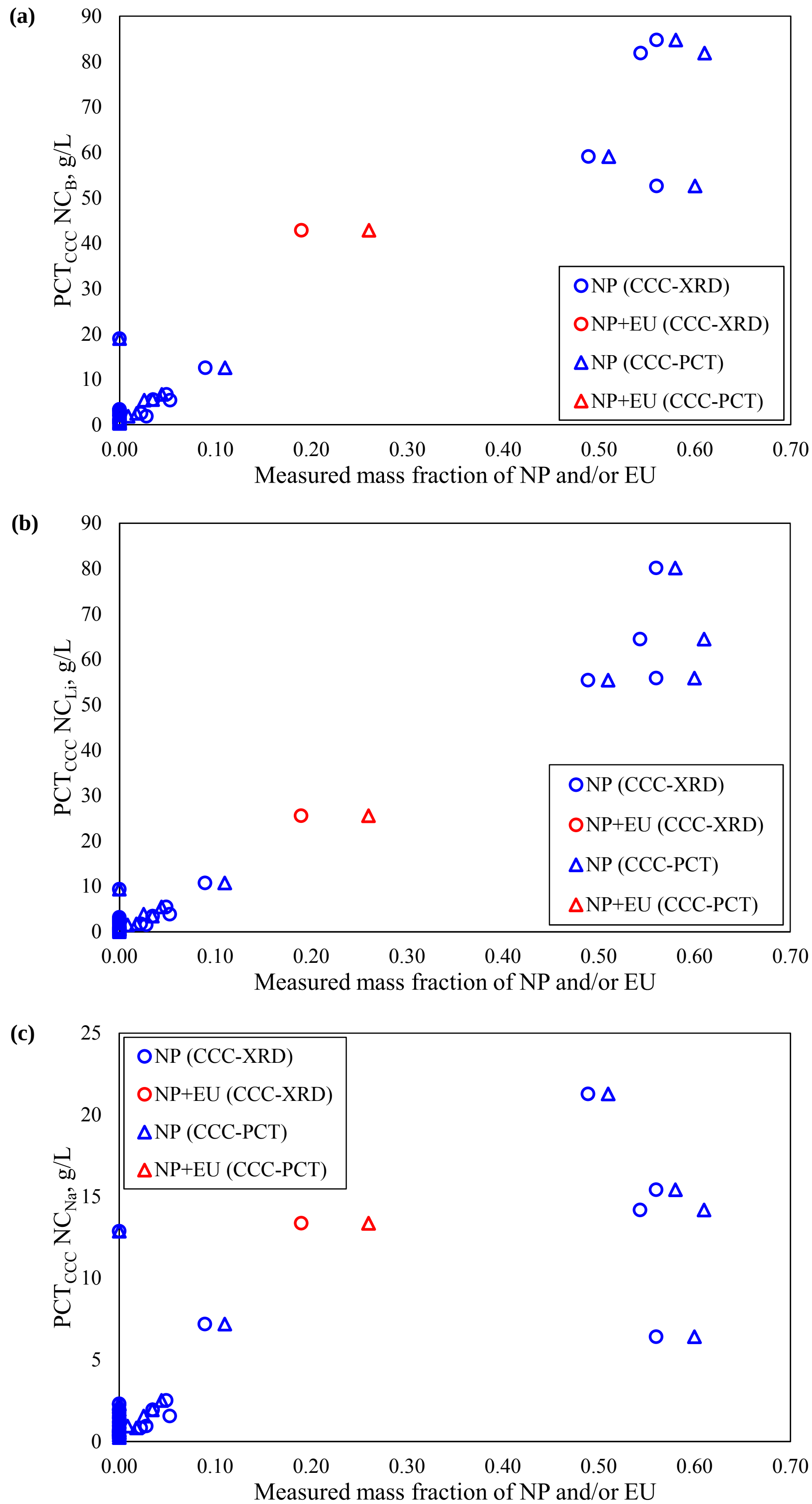


**Figure 6.1.** Measured mass% of Crystal vs Normalized PCT Leachate Concentrations of (a) B, (b) Li, and (c) Na (NP = Nepheline, EU = Eucryptite)

To help visualize the effect of nepheline crystal fraction on PCT response, the natural logarithm of the $NC_B$ of the quenched glasses were subtracted from the respective natural logarithm of $NC_B$ of CCC glasses and plotted against the measured nepheline plus eucryptite concentration in Figure 6.2. In Figure 6.2, glasses that precipitated nepheline (NP) are plotted in blue and the glass that precipitated nepheline and eucryptite (NP+EU) is plotted in red. The symbols represent the various samples analyzed, and can be related back to the sub-sample IDs in Table 5.1.

In Figure 6.2, the difference between the natural logarithms of PCT B concentrations for the quenched and CCC glasses increases with increasing crystal concentration. This relationship indicates the detrimental effect of nepheline on glass durability.

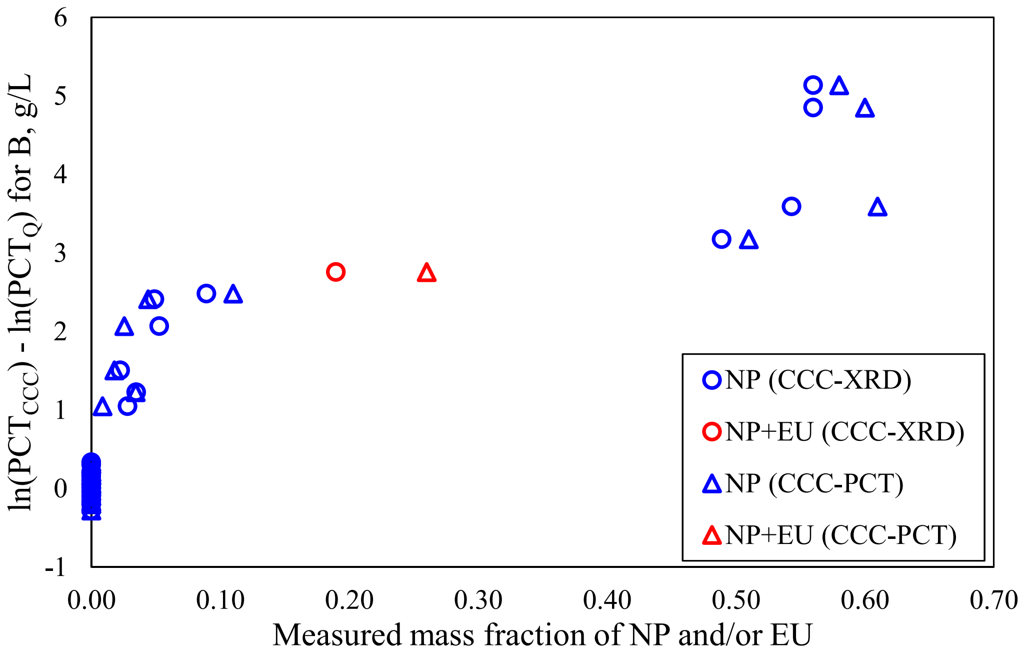


**Figure 6.2.** Natural Logarithm (ln) of Normalized B Leachate Concentrations of Quenched Glasses Subtracted from the Natural Logarithm of Normalized B Leachate Concentrations of CCC Glasses Plotted Against Measured Mass Fraction of Nepheline (NP) and/or Eucryptite (EU).

The purpose of the work presented in this report was to generate data for modeling the effects of HLW glass compositions on nepheline formation and nepheline fraction, and ultimately on the PCT response. Future modeling efforts should use the targeted glass compositions identified in Table 3.3.

# 7.0 References


10 CFR 830, Subpart A. 2010. "Nuclear Safety Management." Subpart A, "Quality Assurance Requirements." *Code of Federal Regulations*, U.S. Department of Energy. Available at http://ecfr.gpoaccess.gov/cgi/t/text/textidx?c=ecfr&rgn=div6&view=text&node=10:4.0.2.5.26.1&idno=10.

ASME. 2001. *Quality Assurance Requirements for Nuclear Facility Applications*. ASME NQA-1-2000, American Society of Mechanical Engineers, New York, New York.

ASME. 2008. *Quality Assurance Requirements for Nuclear Facility Applications*. ASME NQA-1-2008, American Society of Mechanical Engineers, New York, New York.

ASME. 2009. *Addenda to NQA-1-2008, Quality Assurance Requirements for Nuclear Facility Applications*. ASME NQA-1a-2009, American Society of Mechanical Engineers, New York, New York.

ASME. 2013. *Quality Assurance Requirements for Nuclear Facility Applications*. ASME NQA-1-2012, American Society of Mechanical Engineers, New York, New York.

ASTM. 2014. *Standard Test Methods for Determining Chemical Durability of Nuclear Waste Glasses: The Product Consistency Test (PCT)*. ASTM C-1285, ASTM International. West Conshohocken, Pennsylvania.

Atkinson, AC, AN Donev, and RD Tobias. 2007. *Optimum Experimental Designs, with SAS.* Oxford Statistical Science Series, Oxford University Press, Oxford, United Kingdom.

Cheary, RW, AA Coelho, and JP Cline. 2004. "Fundamental Parameters Line Profile Fitting in Laboratory Diffractometers." *Journal of Research of the National Institute of Standards and Technology* 109(1):1-25. Available at http://nvlpubs.nist.gov/nistpubs/jres/109/1/j91che.pdf.

Cornell, JA. 2002. *Experiments With Mixtures: Designs, Models, and the Analysis of Mixture Data, Third Edition.* John Wiley & Sons, New York. ISBN: 978-0-471-39367-2.

DOE Order 414.1D, *Quality Assurance*. U.S. Department of Energy, Washington, D.C.

U.S. Department of Energy, Office of River Protection (DOE-ORP). *Design, Construction, and Commissioning of the Hanford Tank Waste Treatment and Immobilization Plant.* Richland, Washington.

Fox, KM, TB Edwards, ME Caldwell, and WT Riley. 2018. *Chemical Composition Analysis and Product Consistency Tests of the ORP Phase 5 Nepheline Study Glasse*. SRNL-STI-2017-00759, Rev. 0, Savannah River National Laboratory, Aiken, South Carolina.

Hrma, PR and GF Piepel. 1994. *Property/Composition Relationships for Hanford High-Level Waste Glasses Melting at 1150°C, Volume 2, Chapters 12-16 and Appendices A-K*. PNL-10359. Pacific Northwest National Laboratory, Richland, Washington.

Jantzen, CM, NE Bibler, DC Beam, CL Crawford, and MA Pickett. 1993. *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.

JMP™ Pro, Ver. 13.0, Computer Software, SAS Institute Inc., Cary, NC (2016)

Kim, D-S, D Peeler, and P Hrma. 1995. "Effect of Crystallization on the Chemical Durability of Simulated Nuclear Waste Glasses." *Ceramic Transactions* 61:177-185.

Kroll, JO, JD Vienna, MJ Schweiger, GF Piepel, and SK Cooley. 2016. *Results from Phase 1, 2, and 3 Studies on Nepheline Formation in High-Level Waste Glasses Containing High Concentrations of Alumina.* PNNL-26057, Rev. 0.0/EWG-RPT-011, Rev. 0.0. Pacific Northwest National Laboratory, Richland, Washington.

Li, H, JD Vienna, P Hrma, DE Smith, and MJ Schweiger. 1997. "Nepheline Precipitation in High-Level Waste Glasses: Compositional Effects and Impact on the Waste Form Acceptability." *Scientific Basis for Nuclear Waste Management XX.* 465:261-268. Materials Research Society, Pittsburgh, Pennsylvania.

McCloy, J and JD Vienna. 2010. *Glass Composition Constraint Recommendations for Use in Life-Cycle Mission Modeling.* PNNL-19372. Pacific Northwest National Laboratory, Richland, Washington. Availale at http://www.pnl.gov/main/publications/external/technical_reports/PNNL-19372.pdf.

McCloy, JS, MJ Schweiger, CP Rodriguez, and JD Vienna. 2011. "Nepheline Crystallization in Nuclear Waste Glasses: Progress toward Acceptance of High-Alumina Formulations." *International Journal of Applied Glass Science* 2(3):201-214. Available at http://onlinelibrary.wiley.com/doi/10.1111/j.2041-1294.2011.00055.x/full.

Rodriguez, CP, JS McCloy, MJ Schweiger, JV Crum, and A Winschell. 2011. *Optical Basicity and Nepheline Crystallization in High Alumina Glasses.* PNNL-20184. Pacific Northwest National Laboratory, Richland, Washington. Available at http://www.pnnl.gov/main/publications/external/technical_reports/PNNL-20184.pdf.

Vienna, JD, A Fluegel, DS Kim, and P Hrma. 2009. *Glass Property Data and Models for Estimating High-Level Waste Glass Volume.* PNNL-18501, Pacific Northwest National Laboratory Richland, Washington. Available at http://www.pnl.gov/main/publications/external/technical_reports/PNNL-18501.pdf.

Vienna, JD, D-S Kim, DC Skorski, and J Matyas. 2013. *Glass Property Models and Constraints for Estimating the Glass to Be Produced at Hanford by Implementing Current Advanced Glass Formulation Efforts.* PNNL-22631, Rev. 1/ORP-58289, Pacific Northwest National Laboratory, Richland, Washington. Available at http://www.pnnl.gov/main/publications/external/technical_reports/PNNL-22631Rev1.pdf.

Vienna, JD, D-S Kim, MJ Schweiger, GF Piepel, JO Kroll, and AA Kruger. 2014. *Glass Formulation and Testing for U.S. High-Level Tank Wastes Project 17210 Year 1 Status Report: October 15, 2014.* PNNL-SA-84872, Pacific Northwest National Laboratory, Richland, Washington. Available at https://www.hanford.gov/files.cfm/Glass_Formulation_Year_1_Report_Final_10-15-14,_PNNL-SA-84872.pdf.

Vienna, JD, GF Piepel, DS Kim, JV Crum, CE Lonergan, BA Stanfill, BJ Riley, SK Cooley, and T Jin. 2016. *Update of Hanford Glass Property Models and Constraints for Use in Estimating the Glass Mass to be Produced at Hanford by Implementing Current Enhanced Glass Formulation Efforts*. PNNL-25835, Pacific Northwest National Laboratory, Richland, Washington. Available at http://www.pnnl.gov/main/publications/external/technical_reports/PNNL-25835.pdf.

Weier, DR and GF Piepel. 2003. *Methodology for Adjusting and Normalizing Analyzed Glass Composition*. PNWD-3260/WTP-RPT-049. Battelle Pacific Northwest Division, Richland, Washington. Available at http://www.pnl.gov/rpp-wtp/documents/WTP-RPT-049.pdf.

Welch, WJ. 1984. "Computer-Aided Design of Experiments for Response Estimation." *Technometrics* 26:217-224.

Welch, WJ. 1987. *ACED, Algorithms for the Construction of Experimental Designs, Users Guide Version 1.6.1.* University of Waterloo,Waterloo, Ontario, Canada.

# Appendix A

# Quantitative XRD Results for Major Crystalline Phases

# Appendix A

# Quantitative XRD Results for Major Crystalline Phases

This appendix contains the quantitative XRD results for all crystalline phases ≥1 mass% in quenched and CCC glasses. In Table A.1, the Glass IDs, sub-sample IDs, nepheline (NP) and eucryptite (EU) fractions, as well as the size of the crucible used for the CCC heat treatment. The crystalline phases identified in each sample (other than nepheline and eucryptite) are listed in the "Other Crystalline Species" column. The respective ICSD collection code for each crystalline phase identified is also listed in the table.

**Table A.1.** Quantitative XRD Results for all Major Crystalline Phases

| Glass ID | Sub-Sample ID | CCC Crucible Size | NP[a] (Mass%) | CC[b] | EU[c] (Mass%) | CC[b] | Other Crystalline Species (CC[b]), Mass% |
|---|---|---|---|---|---|---|---|
| NP5-01 | NP5-01-CCC-XRD-1 | 1.2$cm^3$ | 0 | | 0 | | Manganese Iron Chromium Oxide (044410), 6.1 |
| | NP5-01-CCC-PCT-1 | 1$in^3$ | 0 | | 0 | | Manganese Iron Chromium Oxide (044410), 6.2 |
| NP5-04 | NP5-04-CCC-XRD-1 | 1.2$cm^3$ | 0 | | 0 | | Iron Silicon Oxide (087458), 3.6<br>Fluorapatite (079653), 1.0<br>Magnetite (050272), 1.1 |
| | NP5-04-CCC-PCT-1 | 1$in^3$ | 0 | | 0 | | Iron Silicon Oxide (087458), 1.5<br>Fluorapatite (079653), 1.5<br>Magnetite (050272), 3.1 |
| NP5-05 | NP5-05-CCC-XRD-1 | 1.2$cm^3$ | 0 | | 0 | | Lithium Iron Oxide (035768), 6.8 |
| | NP5-05-CCC-PCT-1 | 1$in^3$ | 0 | | 0 | | Lithium Iron Manganese Oxide (088143), 9.9 |
| NP5-06 | NP5-06-CCC-XRD-1 | 1.2$cm^3$ | 0 | | 0 | | Iron Oxide (082433), 5.9 |
| | NP5-06-CCC-PCT-1 | 1$in^3$ | 0 | | 0 | | Magnetite (050273), 5.2 |
| NP5-07 | NP5-07-CCC-XRD-1 | 1.2$cm^3$ | 0 | | 0 | | Iron Oxide (082454), 4.6 |
| | NP5-07-CCC-PCT-1 | 1$in^3$ | 0 | | 0 | | Iron Oxide (082454), 4.9 |
| NP5-08 | NP5-08-CCC-XRD-1 | 1.2$cm^3$ | 0 | | 0 | | Nichromite (037427), 7.1 |
| | NP5-08-CCC-PCT-1 | 1$in^3$ | 0 | | 0 | | Nichromite (037427), 7.2 |
| NP5-09 | NP5-09-CCC-XRD-1 | 1.2$cm^3$ | 0 | | 0 | | Chromite (171121), 6.1 |
| | NP5-09-CCC-PCT-1 | 1$in^3$ | 0 | | 0 | | Chromite (171121), 5.8 |
| NP5-10 | NP5-10-CCC-XRD-1 | 1.2$cm^3$ | 56 | 155002 | 0 | | Nickel Manganese Chromium Oxide (009406), 7.7<br>Nosean (031137), 1.9<br>Fluorapatite (079653), 3.8 |
| | NP5-10-CCC-PCT-1 | 1$in^3$ | 60 | 155002 | 0 | | Nickel Manganese Chromium Oxide (009406), 7.9<br>Nosean (031137), 1.2<br>Fluorapatite (079653), 4.1 |
| NP5-11 | NP5-11-CCC-XRD-1 | 1.2$cm^3$ | 56 | 155002 | 0 | | Chromite (171121), 3.4<br>Nosean (031137), 1.8 |
| | NP5-11-CCC-PCT-1 | 1$in^3$ | 58 | 155002 | 0 | | Iron Oxide (035000), 3.1 |
| NP5-12 | NP5-12-CCC-XRD-1 | 1.2$cm^3$ | 0 | | 0 | | Iron Oxide (082454), 8.4<br>Fluorapatite (094081), 2.9 |
| | NP5-12-CCC-PCT-1 | 1$in^3$ | 0 | | 0 | | Iron Oxide (082440), 8.4<br>Fluorapatite (066454), 2.8 |

**Table A.1.** Quantitative XRD Results for all Major Crystalline Phases (cont'd)

| Glass ID | Sub-Sample ID | CCC Crucible Size | NP[a] (Mass%) | CC[b] | EU[c] (Mass%) | CC[b] | Other Crystalline Species (CC[b]), Mass% |
|---|---|---|---|---|---|---|---|
| NP5-13 | NP5-13-Q-XRD-1 | | 0 | | 0 | | Nickel Chromium Manganese Oxide (009405), 5.5 |
| | NP5-13-CCC-XRD-1 | 1.2cm$^3$ | 2.8 | 065958 | 0 | | Iron Oxide (082451), 10<br>Fluorapatite (079653), 5.1<br>Lazurite 1C (049759), 4.1<br>Sodium Aluminum Silicate - Sodalite (085513), 1.0 |
| | NP5-13-CCC-PCT-1 | 1in$^3$ | 0.88 | 065958 | 0 | | Iron Oxide (082451), 10<br>Fluorapatite (079653), 5.2<br>Lazurite 1C (049759), 4.5<br>Sodium Aluminum Silicate - Sodalite (085513), 1.1 |
| NP5-14 | NP5-14-CCC-XRD-1 | 1.2cm$^3$ | 0 | | 0 | | Iron Oxide (082440), 7.8 |
| | NP5-14-CCC-PCT-1 | 1in$^3$ | 0 | | 0 | | Iron Oxide (082450), 7.9 |
| NP5-15 | NP5-15-CCC-XRD-1 | 1.2cm$^3$ | 0 | | 0 | | Iron Oxide (082434), 7.2 |
| | NP5-15-CCC-PCT-1 | 1in$^3$ | 0 | | 0 | | Iron Oxide (082428), 7.3 |
| NP5-16 | NP5-16-CCC-XRD-1 | 1.2cm$^3$ | 49 | 155002 | 0 | | Trevorite (052387), 8.3<br>Lazurite (049759), 4.7<br>Sodium Aluminum Silicate (201588), 1.3 |
| | NP5-16-CCC-PCT-1 | 1in$^3$ | 51 | 155002 | 0 | | Trevorite (052387), 8.7<br>Lazurite (049759), 4.8<br>Sodium Aluminum Silicate (201588), 1.1 |
| NP5-17 | NP5-17-CCC-XRD-1 | 1.2cm$^3$ | 0 | | 0 | | Nickel Manganese Chromium Oxide (009406), 6.3 |
| | NP5-17-CCC-PCT-1 | 1in$^3$ | 0 | | 0 | | Nickel Manganese Chromium Oxide (009406), 6.2 |
| NP5-18 | NP5-18-CCC-XRD-1 | 1.2cm$^3$ | 0 | | 0 | | Iron Silicon Oxide (087458), 2.4 |
| | NP5-18-CCC-PCT-1 | 1in$^3$ | 0 | | 0 | | Iron Silicon Oxide (087458), 2.3 |
| NP5-19 | NP5-19-Q-XRD-1 | | 0 | | 0 | | Nickel Chromium Manganese Oxide (009405), 5.5 |
| | NP5-19-CCC-XRD-1 | 1.2cm$^3$ | 5.3 | 155002 | 0 | | Nickel Manganese Oxide (095232), 8.9<br>Haueyne (028479), 4.0<br>Sodium Aluminum Silicate - Sodalite (085513), 1.1 |
| | NP5-19-CCC-PCT-1 | 1in$^3$ | 2.6 | 155002 | 0 | | Nickel Manganese Oxide (095232), 8.3<br>Lazurite 1C (049759), 4.3<br>Sodium Aluminum Silicate (201588), 1.4 |
| NP5-20 | NP5-20-Q-XRD-1 | | 0 | | 0 | | Chromite (171121), 6.2 |
| | NP5-20-CCC-XRD-1 | 1.2cm$^3$ | 0 | | 0 | | Magnetite (082441), 9.1 |
| | NP5-20-CCC-PCT-1 | 1in$^3$ | 0 | | 0 | | Iron Oxide (082440), 9.6 |

**Table A.1.** Quantitative XRD Results for all Major Crystalline Phases (cont'd)

| Glass ID | Sub-Sample ID | CCC Crucible Size | NP[a] (Mass%) | CC[b] | EU[c] (Mass%) | CC[b] | Other Crystalline Species (CC[b]), Mass% |
|---|---|---|---|---|---|---|---|
| NP5-21 | NP5-21-Q-XRD-1 | | 0 | | 0 | | Nichromite (037427), 7.8 |
| | NP5-21-CCC-XRD-1 | 1.2cm$^3$ | 19 | 155002 | 0.28 | 002935 | Nickel Iron Oxide (084101), 10<br>Lithium Sulfate (030276), 5.7<br>Lazurite (095459), 5.2<br>Nosean (024243), 2.3 |
| | NP5-21-CCC-PCT-1 | 1in$^3$ | 25 | 155002 | 1.0 | 002935 | Nickel Iron Oxide (084101), 9.9<br>Lithium Sulfate (030276), 5.2<br>Lazurite (095459), 3.8<br>Nosean (024243), 2.0 |
| NP5-22 | NP5-22-Q-XRD-1 | | 0 | | 0 | | Trevorite (052387), 5.3 |
| | NP5-22-CCC-XRD-1 | 1.2cm$^3$ | 0 | | 0 | | Nickel Chromium Manganese Oxide (009405), 7.6<br>Fluorapatite (094081), 2.9 |
| | NP5-22-CCC-PCT-1 | 1in$^3$ | 0 | | 0 | | Nickel Chromium Manganese Oxide (009405), 7.1<br>Fluorapatite (079653), 2.8 |
| NP5-23 | NP5-23-CCC-XRD-1 | 1.2cm$^3$ | 8.9 | 200584 | 0 | | Maghemite Q (087121), 6.2<br>Hauyne (02441), 1.3 |
| | NP5-23-CCC-PCT-1 | 1in$^3$ | 11 | 155002 | 0 | | Maghemite Q (087121), 6.2<br>Lazurite (049759), 2.6 |
| NP5-24 | NP5-24-CCC-XRD-1 | 1.2cm$^3$ | 4.9 | 065959 | 0 | | Iron Silicon Oxide (087458), 2.8<br>Silicon Oxide (155247), 2.3 |
| | NP5-24-CCC-PCT-1 | 1in$^3$ | 4.4 | 065959 | 0 | | Iron Oxide (082434), 2.9 |
| NP5-25 | NP5-25-CCC-XRD-1 | 1.2cm$^3$ | 54 | 155002 | 0 | | Lithium Iron Manganese Oxide (150251), 8.3<br>Nosean (024243), 1.9<br>Lazurite (095459), 1.2 |
| | NP5-25-CCC-PCT-1 | 1in$^3$ | 61 | 155002 | 0 | | Lithium Iron Manganese Oxide (150251), 8.7<br>Lazurite (095459), 1.1 |
| NP5-26 | NP5-26-Q-XRD-1 | | 0 | | 0 | | Nickel Chromium Manganese Oxide (009404), 5.5 |
| | NP5-26-CCC-XRD-1 | 1.2cm$^3$ | 0 | | 0 | | Magnetite (0824546), 7.2 |
| | NP5-26-CCC-PCT-1 | 1in$^3$ | 0 | | 0 | | Magnetite (0824546), 7.3 |
| NP5-27 | NP5-27-CCC-XRD-1 | 1.2cm$^3$ | 2.2 | 200584 | 0 | | Iron Silicate (041006), 3.6 |
| | NP5-27-CCC-PCT-1 | 1in$^3$ | 1.8 | 200584 | 0 | | Iron Silicate (041006), 3.8 |
| BL3 | BL3-CCC-XRD-1 | 1.2cm$^3$ | 3.5 | 155002 | 0 | | Lithium Iron Oxide (051206), 2.2<br>Lithium Iron Manganese Oxide (093817), 1.6 |
| | BL3-CCC-XRD-2 | 1.2cm$^3$ | 2.8 | 155002 | 0 | | Lithium Iron Oxide (051206), 2.3<br>Lithium Iron Manganese Oxide (093817), 1.6 |
| | BL3-CCC-PCT-1 | 1in$^3$ | 3.4 | 155002 | 0 | | Lithium Iron Oxide (051206), 2.3<br>Lithium Iron Manganese Oxide (093817), 1.7 |

# Appendix B

# Composition Analysis Results

# Appendix B

# Composition Analysis Results

This appendix contains the composition analysis results reported by Fox et al. (2018). The "Measured" values in Table B.1 are the average of two measurements taken from each prepared sample. All measured oxides with concentrations below the associated detection limit are denoted by a less than symbol (<) in the below detectable limits (BDL) column. The difference and percent relative difference between measured and targeted values was not computed for oxides with concentrations below detectable limits. The average measured oxide concentrations in the duplicate EA glasses (EA1 and EA2), as well as the low-activity reference material (LRM) are also reported. For the EA and LRM glasses, the "Targeted" oxide concentrations represent the nominal oxide concentrations. The percent relative difference cell in Table B.1 is shaded for oxides with a percent relative difference ≥ 10% if the targeted oxide concentration is ≥ 5 mass%.

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| EA1 | $Al_2O_3$ | | 3.58 | 3.70 | -0.12 | -3.4% |
| EA1 | $B_2O_3$ | | 11.58 | 11.28 | 0.30 | 2.6% |
| EA1 | $Bi_2O_3$ | < | 0.11 | 0.00 | | |
| EA1 | CaO | | 1.06 | 1.12 | -0.06 | -5.5% |
| EA1 | $Cr_2O_3$ | < | 0.15 | 0.00 | | |
| EA1 | F | < | 0.05 | 0.00 | | |
| EA1 | $Fe_2O_3$ | | 9.49 | 8.99 | 0.50 | 5.5% |
| EA1 | $Li_2O$ | | 3.82 | 4.26 | -0.44 | -10.3% |
| EA1 | MnO | | 1.29 | 1.34 | -0.05 | -3.9% |
| EA1 | $Na_2O$ | | 16.72 | 16.81 | -0.10 | -0.6% |
| EA1 | NiO | | 0.50 | 0.57 | -0.07 | -11.8% |
| EA1 | $P_2O_5$ | < | 0.12 | 0.00 | | |
| EA1 | PbO | < | 0.11 | 0.00 | | |
| EA1 | $RuO_2$ | < | 0.07 | 0.00 | | |
| EA1 | $SiO_2$ | | 48.46 | 48.73 | -0.28 | -0.6% |
| EA1 | $SO_3$ | < | 0.13 | 0.00 | | |
| EA1 | SrO | < | 0.12 | 0.00 | | |
| EA1 | $ZrO_2$ | | 0.44 | 0.46 | -0.02 | -4.1% |
| EA1 | Sum | | 97.76 | 97.26 | 0.50 | 0.5% |
| EA2 | $Al_2O_3$ | | 3.62 | 3.70 | -0.08 | -2.1% |
| EA2 | $B_2O_3$ | | 11.86 | 11.28 | 0.58 | 5.1% |
| EA2 | $Bi_2O_3$ | < | 0.11 | 0.00 | | |
| EA2 | CaO | | 1.13 | 1.12 | 0.01 | 0.7% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[a] | Oxide | BDL (<)[b] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| EA2 | $Cr_2O_3$ | < | 0.15 | 0.00 | | |
| EA2 | F | < | 0.05 | 0.00 | | |
| EA2 | $Fe_2O_3$ | | 9.60 | 8.99 | 0.61 | 6.8% |
| EA2 | $Li_2O$ | | 3.83 | 4.26 | -0.43 | -10.0% |
| EA2 | MnO | | 1.31 | 1.34 | -0.03 | -2.2% |
| EA2 | $Na_2O$ | | 16.95 | 16.81 | 0.14 | 0.8% |
| EA2 | NiO | | 0.51 | 0.57 | -0.06 | -10.2% |
| EA2 | $P_2O_5$ | < | 0.12 | 0.00 | | |
| EA2 | PbO | < | 0.11 | 0.00 | | |
| EA2 | $RuO_2$ | < | 0.07 | 0.00 | | |
| EA2 | $SiO_2$ | | 48.51 | 48.73 | -0.22 | -0.5% |
| EA2 | $SO_3$ | < | 0.13 | 0.00 | | |
| EA2 | SrO | < | 0.12 | 0.00 | | |
| EA2 | $ZrO_2$ | | 0.43 | 0.46 | -0.03 | -6.5% |
| EA2 | Sum | | 98.59 | 97.26 | 1.33 | 1.4% |
| LRM | $Al_2O_3$ | | 9.49 | 9.51 | -0.02 | -0.2% |
| LRM | $B_2O_3$ | | 7.83 | 7.85 | -0.02 | -0.3% |
| LRM | $Bi_2O_3$ | < | 0.11 | 0.00 | | |
| LRM | CaO | | 0.39 | 0.54 | -0.15 | -27.2% |
| LRM | $Cr_2O_3$ | < | 0.16 | 0.19 | | |
| LRM | F | | 0.88 | 0.86 | 0.02 | 1.9% |
| LRM | $Fe_2O_3$ | | 1.37 | 1.38 | -0.01 | -0.5% |
| LRM | $Li_2O$ | < | 0.22 | 0.11 | | |
| LRM | MnO | < | 0.13 | 0.08 | | |
| LRM | $Na_2O$ | | 19.66 | 20.03 | -0.37 | -1.8% |
| LRM | NiO | < | 0.12 | 0.19 | | |
| LRM | $P_2O_5$ | | 0.41 | 0.54 | -0.13 | -24.4% |
| LRM | PbO | < | 0.11 | 0.10 | | |
| LRM | $RuO_2$ | < | 0.07 | 0.00 | | |
| LRM | $SiO_2$ | | 55.85 | 54.20 | 1.65 | 3.0% |
| LRM | $SO_3$ | | 0.23 | 0.30 | -0.07 | -24.0% |
| LRM | SrO | < | 0.12 | 0.00 | | |
| LRM | $ZrO_2$ | | 1.00 | 0.93 | 0.07 | 7.3% |
| LRM | Sum | | 98.14 | 96.81 | 1.33 | 1.4% |
| NP5-01-Q | $Al_2O_3$ | | 21.73 | 22.31 | -0.58 | -2.6% |
| NP5-01-Q | $B_2O_3$ | | 21.00 | 21.12 | -0.12 | -0.6% |
| NP5-01-Q | $Bi_2O_3$ | | 1.00 | 1.06 | -0.06 | -5.8% |
| NP5-01-Q | CaO | | 5.70 | 5.00 | 0.70 | 14.0% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-01-Q | $Cr_2O_3$ | | 1.10 | 0.98 | 0.12 | 12.1% |
| NP5-01-Q | F | | 0.18 | 0.34 | -0.16 | -47.4% |
| NP5-01-Q | $Fe_2O_3$ | | 5.66 | 5.06 | 0.60 | 11.8% |
| NP5-01-Q | MnO | | 2.15 | 2.08 | 0.07 | 3.5% |
| NP5-01-Q | $Na_2O$ | | 12.58 | 13.33 | -0.75 | -5.6% |
| NP5-01-Q | NiO | | 0.32 | 0.33 | -0.01 | -2.1% |
| NP5-01-Q | $P_2O_5$ | | 0.92 | 0.95 | -0.03 | -3.3% |
| NP5-01-Q | PbO | | 0.17 | 0.17 | -0.01 | -2.9% |
| NP5-01-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-01-Q | $SiO_2$ | | 27.12 | 26.14 | 0.98 | 3.7% |
| NP5-01-Q | $SO_3$ | | 0.28 | 0.35 | -0.07 | -20.0% |
| NP5-01-Q | SrO | | 0.15 | 0.16 | -0.01 | -3.8% |
| NP5-01-Q | $ZrO_2$ | | 0.69 | 0.61 | 0.08 | 13.0% |
| NP5-01-Q | Sum | | 100.82 | 100.00 | 1.03 | 1.0% |
| NP5-04-Q | $Al_2O_3$ | | 19.84 | 20.20 | -0.36 | -1.8% |
| NP5-04-Q | $B_2O_3$ | | 19.67 | 19.33 | 0.34 | 1.8% |
| NP5-04-Q | $Bi_2O_3$ | | 1.15 | 1.14 | 0.01 | 1.2% |
| NP5-04-Q | CaO | | 1.66 | 1.69 | -0.04 | -2.1% |
| NP5-04-Q | $Cr_2O_3$ | | 1.12 | 1.06 | 0.06 | 5.7% |
| NP5-04-Q | F | | 0.24 | 0.37 | -0.13 | -34.6% |
| NP5-04-Q | $Fe_2O_3$ | | 4.35 | 3.87 | 0.48 | 12.4% |
| NP5-04-Q | $Li_2O$ | | 3.64 | 3.85 | -0.21 | -5.4% |
| NP5-04-Q | MnO | | 2.18 | 2.25 | -0.07 | -3.3% |
| NP5-04-Q | $Na_2O$ | | 10.23 | 11.12 | -0.89 | -8.0% |
| NP5-04-Q | NiO | | 0.27 | 0.36 | -0.09 | -25.6% |
| NP5-04-Q | $P_2O_5$ | | 1.04 | 1.90 | -0.87 | -45.5% |
| NP5-04-Q | PbO | | 0.18 | 0.18 | 0.00 | 0.0% |
| NP5-04-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-04-Q | $SiO_2$ | | 30.91 | 31.45 | -0.54 | -1.7% |
| NP5-04-Q | $SO_3$ | | 0.35 | 0.38 | -0.03 | -7.9% |
| NP5-04-Q | SrO | | 0.18 | 0.18 | 0.00 | 0.0% |
| NP5-04-Q | $ZrO_2$ | < | 0.17 | 0.66 | | |
| NP5-04-Q | Sum | | 97.24 | 100.00 | -2.76 | -2.8% |
| NP5-05-Q | $Al_2O_3$ | | 28.01 | 28.30 | -0.29 | -1.0% |
| NP5-05-Q | $B_2O_3$ | | 15.01 | 15.03 | -0.02 | -0.1% |
| NP5-05-Q | $Bi_2O_3$ | | 1.35 | 1.38 | -0.03 | -2.2% |
| NP5-05-Q | CaO | | 4.90 | 4.40 | 0.50 | 11.3% |
| NP5-05-Q | $Cr_2O_3$ | | 1.40 | 1.29 | 0.11 | 8.8% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-05-Q | F | | 0.24 | 0.44 | -0.20 | -45.0% |
| NP5-05-Q | $Fe_2O_3$ | | 4.13 | 3.50 | 0.63 | 17.9% |
| NP5-05-Q | $Li_2O$ | | 4.69 | 4.91 | -0.22 | -4.5% |
| NP5-05-Q | MnO | | 2.79 | 2.72 | 0.07 | 2.5% |
| NP5-05-Q | $Na_2O$ | | 7.22 | 8.08 | -0.86 | -10.6% |
| NP5-05-Q | NiO | | 0.46 | 0.43 | 0.03 | 7.0% |
| NP5-05-Q | $P_2O_5$ | | 1.10 | 1.89 | -0.80 | -42.1% |
| NP5-05-Q | PbO | | 0.21 | 0.22 | -0.01 | -3.2% |
| NP5-05-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-05-Q | $SiO_2$ | | 26.96 | 25.94 | 1.02 | 3.9% |
| NP5-05-Q | $SO_3$ | | 0.40 | 0.46 | -0.06 | -12.6% |
| NP5-05-Q | SrO | | 0.21 | 0.21 | 0.00 | 0.0% |
| NP5-05-Q | $ZrO_2$ | | 0.20 | 0.79 | -0.60 | -75.3% |
| NP5-05-Q | Sum | | 99.34 | 100.00 | -0.67 | -0.7% |
| NP5-06-Q | $Al_2O_3$ | | 22.82 | 23.50 | -0.68 | -2.9% |
| NP5-06-Q | $B_2O_3$ | | 21.03 | 21.99 | -0.96 | -4.4% |
| NP5-06-Q | $Bi_2O_3$ | | 1.10 | 1.09 | 0.01 | 1.0% |
| NP5-06-Q | CaO | | 0.66 | 0.70 | -0.04 | -6.0% |
| NP5-06-Q | $Cr_2O_3$ | | 1.13 | 1.01 | 0.12 | 11.4% |
| NP5-06-Q | F | | 0.19 | 0.35 | -0.16 | -46.0% |
| NP5-06-Q | $Fe_2O_3$ | | 4.65 | 4.12 | 0.53 | 12.8% |
| NP5-06-Q | $Li_2O$ | | 4.24 | 4.57 | -0.33 | -7.2% |
| NP5-06-Q | MnO | | 2.14 | 2.14 | 0.00 | 0.0% |
| NP5-06-Q | $Na_2O$ | | 7.57 | 8.29 | -0.72 | -8.7% |
| NP5-06-Q | NiO | | 0.35 | 0.34 | 0.01 | 2.1% |
| NP5-06-Q | $P_2O_5$ | | 0.68 | 0.72 | -0.04 | -5.3% |
| NP5-06-Q | PbO | | 0.17 | 0.17 | -0.01 | -2.9% |
| NP5-06-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-06-Q | $SiO_2$ | | 30.11 | 29.85 | 0.26 | 0.9% |
| NP5-06-Q | $SO_3$ | | 0.28 | 0.36 | -0.09 | -23.6% |
| NP5-06-Q | SrO | | 0.17 | 0.17 | 0.00 | 0.0% |
| NP5-06-Q | $ZrO_2$ | | 0.69 | 0.62 | 0.07 | 10.6% |
| NP5-06-Q | Sum | | 98.01 | 100.00 | -1.99 | -2.0% |
| NP5-07-Q | $Al_2O_3$ | | 27.49 | 27.97 | -0.48 | -1.7% |
| NP5-07-Q | $B_2O_3$ | | 23.68 | 21.66 | 2.02 | 9.3% |
| NP5-07-Q | $Bi_2O_3$ | | 0.88 | 0.92 | -0.04 | -4.0% |
| NP5-07-Q | CaO | | 2.49 | 2.35 | 0.14 | 5.8% |
| NP5-07-Q | $Cr_2O_3$ | | 0.91 | 0.85 | 0.06 | 7.4% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-07-Q | F | | 0.19 | 0.29 | -0.10 | -34.5% |
| NP5-07-Q | $Fe_2O_3$ | | 3.30 | 2.84 | 0.46 | 16.3% |
| NP5-07-Q | $Li_2O$ | | 0.74 | 0.82 | -0.08 | -9.3% |
| NP5-07-Q | MnO | | 1.74 | 1.80 | -0.06 | -3.3% |
| NP5-07-Q | $Na_2O$ | | 13.90 | 14.50 | -0.60 | -4.1% |
| NP5-07-Q | NiO | | 0.18 | 0.28 | -0.10 | -34.6% |
| NP5-07-Q | $P_2O_5$ | | 1.09 | 1.56 | -0.47 | -30.1% |
| NP5-07-Q | PbO | | 0.14 | 0.14 | 0.00 | 0.0% |
| NP5-07-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-07-Q | $SiO_2$ | | 22.89 | 23.05 | -0.16 | -0.7% |
| NP5-07-Q | $SO_3$ | | 0.28 | 0.30 | -0.02 | -8.0% |
| NP5-07-Q | SrO | | 0.14 | 0.14 | 0.00 | 0.0% |
| NP5-07-Q | $ZrO_2$ | | 0.19 | 0.52 | -0.33 | -62.7% |
| NP5-07-Q | Sum | | 100.32 | 100.00 | 0.32 | 0.3% |
| NP5-08-Q | $Al_2O_3$ | | 28.63 | 29.71 | -1.08 | -3.6% |
| NP5-08-Q | $B_2O_3$ | | 18.06 | 18.68 | -0.62 | -3.3% |
| NP5-08-Q | $Bi_2O_3$ | | 0.86 | 0.96 | -0.10 | -10.2% |
| NP5-08-Q | CaO | | 0.35 | 0.44 | -0.09 | -20.2% |
| NP5-08-Q | $Cr_2O_3$ | | 1.00 | 0.90 | 0.10 | 11.3% |
| NP5-08-Q | F | | 0.20 | 0.31 | -0.11 | -36.5% |
| NP5-08-Q | $Fe_2O_3$ | | 4.86 | 4.41 | 0.45 | 10.1% |
| NP5-08-Q | $Li_2O$ | | 4.89 | 5.33 | -0.44 | -8.3% |
| NP5-08-Q | MnO | | 1.85 | 1.89 | -0.04 | -2.0% |
| NP5-08-Q | $Na_2O$ | | 10.67 | 11.58 | -0.91 | -7.8% |
| NP5-08-Q | NiO | | 0.29 | 0.30 | -0.01 | -3.3% |
| NP5-08-Q | $P_2O_5$ | | 0.90 | 1.14 | -0.24 | -20.8% |
| NP5-08-Q | PbO | | 0.15 | 0.15 | 0.00 | 0.0% |
| NP5-08-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-08-Q | $SiO_2$ | | 23.21 | 23.17 | 0.04 | 0.2% |
| NP5-08-Q | $SO_3$ | | 0.33 | 0.32 | 0.01 | 3.8% |
| NP5-08-Q | SrO | | 0.15 | 0.15 | 0.00 | 0.0% |
| NP5-08-Q | $ZrO_2$ | | 0.44 | 0.55 | -0.11 | -19.8% |
| NP5-08-Q | Sum | | 96.91 | 100.00 | -3.09 | -3.1% |
| NP5-09-Q | $Al_2O_3$ | | 23.86 | 25.39 | -1.54 | -6.0% |
| NP5-09-Q | $B_2O_3$ | | 17.56 | 18.21 | -0.65 | -3.6% |
| NP5-09-Q | $Bi_2O_3$ | | 1.38 | 1.49 | -0.11 | -7.2% |
| NP5-09-Q | CaO | | 2.48 | 2.29 | 0.19 | 8.3% |
| NP5-09-Q | $Cr_2O_3$ | | 1.50 | 1.39 | 0.11 | 8.1% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[a] | Oxide | BDL (<)[b] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-09-Q | F | | 0.33 | 0.48 | -0.15 | -30.8% |
| NP5-09-Q | $Fe_2O_3$ | | 3.68 | 3.11 | 0.57 | 18.3% |
| NP5-09-Q | $Li_2O$ | | 3.71 | 4.23 | -0.52 | -12.2% |
| NP5-09-Q | MnO | | 2.93 | 2.92 | 0.01 | 0.3% |
| NP5-09-Q | $Na_2O$ | | 10.87 | 12.08 | -1.22 | -10.1% |
| NP5-09-Q | NiO | | 0.50 | 0.46 | 0.04 | 8.5% |
| NP5-09-Q | $P_2O_5$ | | 0.67 | 0.66 | 0.01 | 1.4% |
| NP5-09-Q | PbO | | 0.23 | 0.24 | -0.01 | -3.7% |
| NP5-09-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-09-Q | $SiO_2$ | | 25.57 | 25.47 | 0.10 | 0.4% |
| NP5-09-Q | $SO_3$ | | 0.47 | 0.49 | -0.03 | -5.1% |
| NP5-09-Q | SrO | | 0.23 | 0.23 | 0.00 | 0.0% |
| NP5-09-Q | $ZrO_2$ | | 0.89 | 0.85 | 0.04 | 5.1% |
| NP5-09-Q | Sum | | 96.91 | 100.00 | -3.09 | -3.1% |
| NP5-10-Q | $Al_2O_3$ | | 24.33 | 24.57 | -0.24 | -1.0% |
| NP5-10-Q | $B_2O_3$ | | 13.66 | 12.82 | 0.84 | 6.6% |
| NP5-10-Q | $Bi_2O_3$ | | 1.25 | 1.22 | 0.03 | 2.5% |
| NP5-10-Q | CaO | | 6.37 | 5.71 | 0.66 | 11.5% |
| NP5-10-Q | $Cr_2O_3$ | | 1.20 | 1.13 | 0.07 | 6.0% |
| NP5-10-Q | F | | 0.27 | 0.39 | -0.13 | -32.1% |
| NP5-10-Q | $Fe_2O_3$ | | 6.28 | 5.64 | 0.64 | 11.3% |
| NP5-10-Q | $Li_2O$ | | 3.59 | 4.03 | -0.45 | -11.0% |
| NP5-10-Q | MnO | | 2.42 | 2.39 | 0.03 | 1.3% |
| NP5-10-Q | $Na_2O$ | | 10.83 | 11.55 | -0.72 | -6.2% |
| NP5-10-Q | NiO | | 0.32 | 0.38 | -0.07 | -17.1% |
| NP5-10-Q | $P_2O_5$ | | 1.39 | 1.68 | -0.29 | -17.2% |
| NP5-10-Q | PbO | | 0.19 | 0.19 | 0.00 | 0.0% |
| NP5-10-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-10-Q | $SiO_2$ | | 28.24 | 27.00 | 1.24 | 4.6% |
| NP5-10-Q | $SO_3$ | | 0.38 | 0.40 | -0.02 | -6.0% |
| NP5-10-Q | SrO | | 0.19 | 0.19 | 0.00 | 0.0% |
| NP5-10-Q | $ZrO_2$ | | 0.46 | 0.70 | -0.24 | -34.1% |
| NP5-10-Q | Sum | | 101.41 | 100.00 | 1.41 | 1.4% |
| NP5-11-Q | $Al_2O_3$ | | 20.03 | 20.50 | -0.47 | -2.3% |
| NP5-11-Q | $B_2O_3$ | | 13.86 | 13.74 | 0.12 | 0.9% |
| NP5-11-Q | $Bi_2O_3$ | | 1.21 | 1.34 | -0.13 | -9.9% |
| NP5-11-Q | CaO | | 4.33 | 4.01 | 0.32 | 7.9% |
| NP5-11-Q | $Cr_2O_3$ | | 1.35 | 1.24 | 0.11 | 9.0% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-11-Q | F | | 0.32 | 0.43 | -0.11 | -25.8% |
| NP5-11-Q | $Fe_2O_3$ | | 2.67 | 2.08 | 0.59 | 28.2% |
| NP5-11-Q | $Li_2O$ | | 4.52 | 4.79 | -0.27 | -5.6% |
| NP5-11-Q | MnO | | 2.67 | 2.62 | 0.05 | 1.8% |
| NP5-11-Q | $Na_2O$ | | 12.01 | 13.28 | -1.27 | -9.5% |
| NP5-11-Q | NiO | | 0.43 | 0.42 | 0.01 | 1.4% |
| NP5-11-Q | $P_2O_5$ | | 0.99 | 1.26 | -0.27 | -21.6% |
| NP5-11-Q | PbO | | 0.20 | 0.21 | -0.01 | -6.2% |
| NP5-11-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-11-Q | $SiO_2$ | | 33.16 | 32.65 | 0.51 | 1.6% |
| NP5-11-Q | $SO_3$ | | 0.45 | 0.44 | 0.01 | 2.5% |
| NP5-11-Q | SrO | | 0.21 | 0.21 | 0.00 | 0.0% |
| NP5-11-Q | $ZrO_2$ | | 0.61 | 0.77 | -0.16 | -20.9% |
| NP5-11-Q | Sum | | 99.06 | 100.00 | -0.94 | -0.9% |
| NP5-12-Q | $Al_2O_3$ | | 19.70 | 20.22 | -0.52 | -2.6% |
| NP5-12-Q | $B_2O_3$ | | 14.89 | 15.14 | -0.25 | -1.6% |
| NP5-12-Q | $Bi_2O_3$ | | 1.75 | 1.79 | -0.04 | -2.4% |
| NP5-12-Q | CaO | | 7.54 | 6.77 | 0.77 | 11.3% |
| NP5-12-Q | $Cr_2O_3$ | | 1.81 | 1.66 | 0.15 | 9.0% |
| NP5-12-Q | F | | 0.41 | 0.58 | -0.17 | -29.5% |
| NP5-12-Q | $Fe_2O_3$ | | 6.43 | 5.83 | 0.60 | 10.2% |
| NP5-12-Q | $Li_2O$ | | 2.49 | 2.72 | -0.23 | -8.6% |
| NP5-12-Q | MnO | | 3.57 | 3.51 | 0.06 | 1.6% |
| NP5-12-Q | $Na_2O$ | | 8.32 | 9.22 | -0.90 | -9.7% |
| NP5-12-Q | NiO | | 0.57 | 0.56 | 0.01 | 2.5% |
| NP5-12-Q | $P_2O_5$ | | 1.21 | 1.22 | -0.01 | -1.1% |
| NP5-12-Q | PbO | | 0.29 | 0.28 | 0.01 | 1.8% |
| NP5-12-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-12-Q | $SiO_2$ | | 29.42 | 28.60 | 0.82 | 2.8% |
| NP5-12-Q | $SO_3$ | | 0.55 | 0.59 | -0.04 | -6.9% |
| NP5-12-Q | SrO | | 0.28 | 0.28 | 0.00 | 0.0% |
| NP5-12-Q | $ZrO_2$ | | 1.08 | 1.02 | 0.06 | 6.1% |
| NP5-12-Q | Sum | | 100.35 | 100.00 | 0.35 | 0.4% |
| NP5-13-Q | $Al_2O_3$ | | 21.02 | 21.07 | -0.05 | -0.2% |
| NP5-13-Q | $B_2O_3$ | | 15.20 | 14.68 | 0.52 | 3.5% |
| NP5-13-Q | $Bi_2O_3$ | | 2.08 | 2.25 | -0.17 | -7.5% |
| NP5-13-Q | CaO | | 7.17 | 6.47 | 0.70 | 10.8% |
| NP5-13-Q | $Cr_2O_3$ | | 2.19 | 2.09 | 0.10 | 4.7% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-13-Q | F | | 0.52 | 0.72 | -0.20 | -27.4% |
| NP5-13-Q | $Fe_2O_3$ | | 4.94 | 4.44 | 0.50 | 11.3% |
| NP5-13-Q | $Li_2O$ | | 0.95 | 1.03 | -0.09 | -8.3% |
| NP5-13-Q | MnO | | 4.50 | 4.42 | 0.08 | 1.8% |
| NP5-13-Q | $Na_2O$ | | 12.51 | 13.58 | -1.07 | -7.9% |
| NP5-13-Q | NiO | | 0.66 | 0.70 | -0.04 | -5.6% |
| NP5-13-Q | $P_2O_5$ | | 0.86 | 1.97 | -1.11 | -56.4% |
| NP5-13-Q | PbO | | 0.35 | 0.36 | -0.01 | -1.7% |
| NP5-13-Q | $RuO_2$ | < | 0.07 | 0.02 | | |
| NP5-13-Q | $SiO_2$ | | 24.01 | 23.82 | 0.19 | 0.8% |
| NP5-13-Q | $SO_3$ | | 0.68 | 0.74 | -0.06 | -8.2% |
| NP5-13-Q | SrO | | 0.36 | 0.35 | 0.01 | 2.0% |
| NP5-13-Q | $ZrO_2$ | | 0.45 | 1.29 | -0.84 | -65.3% |
| NP5-13-Q | Sum | | 98.51 | 100.00 | -1.49 | -1.5% |
| NP5-14-Q | $Al_2O_3$ | | 20.55 | 21.70 | -1.15 | -5.3% |
| NP5-14-Q | $B_2O_3$ | | 16.90 | 17.62 | -0.72 | -4.1% |
| NP5-14-Q | $Bi_2O_3$ | | 1.60 | 1.59 | 0.01 | 0.4% |
| NP5-14-Q | CaO | < | 0.07 | 0.07 | | |
| NP5-14-Q | $Cr_2O_3$ | | 1.58 | 1.48 | 0.10 | 6.4% |
| NP5-14-Q | F | | 0.36 | 0.51 | -0.16 | -30.4% |
| NP5-14-Q | $Fe_2O_3$ | | 5.32 | 4.87 | 0.45 | 9.2% |
| NP5-14-Q | $Li_2O$ | | 2.82 | 3.22 | -0.40 | -12.4% |
| NP5-14-Q | MnO | | 3.17 | 3.13 | 0.04 | 1.2% |
| NP5-14-Q | $Na_2O$ | | 13.24 | 14.30 | -1.06 | -7.4% |
| NP5-14-Q | NiO | | 0.48 | 0.50 | -0.02 | -3.8% |
| NP5-14-Q | $P_2O_5$ | | 1.33 | 1.40 | -0.07 | -4.8% |
| NP5-14-Q | PbO | | 0.24 | 0.25 | -0.01 | -3.2% |
| NP5-14-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-14-Q | $SiO_2$ | | 27.65 | 27.66 | -0.01 | 0.0% |
| NP5-14-Q | $SO_3$ | | 0.47 | 0.53 | -0.06 | -10.6% |
| NP5-14-Q | SrO | | 0.26 | 0.25 | 0.01 | 2.0% |
| NP5-14-Q | $ZrO_2$ | | 0.94 | 0.91 | 0.03 | 3.4% |
| NP5-14-Q | Sum | | 97.03 | 100.00 | -2.97 | -3.0% |
| NP5-15-Q | $Al_2O_3$ | | 20.50 | 20.89 | -0.39 | -1.9% |
| NP5-15-Q | $B_2O_3$ | | 16.66 | 16.08 | 0.58 | 3.6% |
| NP5-15-Q | $Bi_2O_3$ | | 1.40 | 1.46 | -0.06 | -4.4% |
| NP5-15-Q | CaO | | 0.72 | 0.83 | -0.11 | -13.4% |
| NP5-15-Q | $Cr_2O_3$ | | 1.42 | 1.36 | 0.06 | 4.1% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-15-Q | F | | 0.34 | 0.47 | -0.14 | -28.7% |
| NP5-15-Q | $Fe_2O_3$ | | 6.08 | 5.51 | 0.57 | 10.3% |
| NP5-15-Q | $Li_2O$ | | 5.32 | 5.85 | -0.53 | -9.1% |
| NP5-15-Q | MnO | | 2.91 | 2.86 | 0.05 | 1.7% |
| NP5-15-Q | $Na_2O$ | | 8.99 | 9.98 | -0.99 | -9.9% |
| NP5-15-Q | NiO | | 0.37 | 0.45 | -0.08 | -18.2% |
| NP5-15-Q | $P_2O_5$ | | 0.35 | 0.49 | -0.14 | -29.0% |
| NP5-15-Q | PbO | | 0.23 | 0.23 | 0.00 | 0.0% |
| NP5-15-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-15-Q | $SiO_2$ | | 33.05 | 32.00 | 1.05 | 3.3% |
| NP5-15-Q | $SO_3$ | | 0.46 | 0.48 | -0.02 | -4.0% |
| NP5-15-Q | SrO | | 0.23 | 0.22 | 0.01 | 3.6% |
| NP5-15-Q | $ZrO_2$ | | 0.79 | 0.83 | -0.04 | -4.8% |
| NP5-15-Q | Sum | | 99.86 | 100.00 | -0.14 | -0.1% |
| NP5-16-Q | $Al_2O_3$ | | 26.74 | 27.47 | -0.73 | -2.7% |
| NP5-16-Q | $B_2O_3$ | | 13.63 | 13.49 | 0.14 | 1.0% |
| NP5-16-Q | $Bi_2O_3$ | | 1.34 | 1.37 | -0.03 | -2.1% |
| NP5-16-Q | CaO | | 3.72 | 3.42 | 0.30 | 8.8% |
| NP5-16-Q | $Cr_2O_3$ | | 1.40 | 1.28 | 0.12 | 9.2% |
| NP5-16-Q | F | | 0.32 | 0.44 | -0.12 | -26.4% |
| NP5-16-Q | $Fe_2O_3$ | | 5.82 | 5.31 | 0.51 | 9.5% |
| NP5-16-Q | $Li_2O$ | | 3.21 | 3.48 | -0.27 | -7.8% |
| NP5-16-Q | MnO | | 2.73 | 2.70 | 0.03 | 1.1% |
| NP5-16-Q | $Na_2O$ | | 13.81 | 14.68 | -0.87 | -5.9% |
| NP5-16-Q | NiO | | 0.45 | 0.43 | 0.02 | 4.7% |
| NP5-16-Q | $P_2O_5$ | | 0.55 | 0.61 | -0.06 | -9.5% |
| NP5-16-Q | PbO | | 0.22 | 0.22 | 0.00 | 0.0% |
| NP5-16-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-16-Q | $SiO_2$ | | 23.96 | 23.64 | 0.32 | 1.4% |
| NP5-16-Q | $SO_3$ | | 0.46 | 0.45 | 0.01 | 2.4% |
| NP5-16-Q | SrO | | 0.21 | 0.21 | 0.00 | 0.0% |
| NP5-16-Q | $ZrO_2$ | | 0.84 | 0.79 | 0.05 | 5.7% |
| NP5-16-Q | Sum | | 99.47 | 100.00 | -0.53 | -0.5% |
| NP5-17-Q | $Al_2O_3$ | | 25.23 | 25.83 | -0.61 | -2.3% |
| NP5-17-Q | $B_2O_3$ | | 23.92 | 22.27 | 1.65 | 7.4% |
| NP5-17-Q | $Bi_2O_3$ | | 1.68 | 1.78 | -0.10 | -5.7% |
| NP5-17-Q | CaO | | 6.10 | 5.74 | 0.36 | 6.2% |
| NP5-17-Q | $Cr_2O_3$ | | 1.71 | 1.65 | 0.06 | 3.9% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-17-Q | F | | 0.38 | 0.57 | -0.19 | -33.7% |
| NP5-17-Q | $Fe_2O_3$ | | 2.89 | 2.34 | 0.55 | 23.4% |
| NP5-17-Q | $Li_2O$ | | 1.22 | 1.35 | -0.14 | -10.0% |
| NP5-17-Q | MnO | | 3.41 | 3.48 | -0.07 | -2.0% |
| NP5-17-Q | $Na_2O$ | | 7.42 | 8.13 | -0.71 | -8.7% |
| NP5-17-Q | NiO | | 0.45 | 0.55 | -0.11 | -19.1% |
| NP5-17-Q | $P_2O_5$ | | 0.36 | 0.58 | -0.22 | -37.9% |
| NP5-17-Q | PbO | | 0.27 | 0.28 | -0.01 | -3.9% |
| NP5-17-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-17-Q | $SiO_2$ | | 23.27 | 23.56 | -0.30 | -1.3% |
| NP5-17-Q | $SO_3$ | | 0.33 | 0.59 | -0.26 | -44.7% |
| NP5-17-Q | SrO | | 0.27 | 0.27 | 0.00 | 0.0% |
| NP5-17-Q | $ZrO_2$ | | 0.91 | 1.02 | -0.11 | -10.5% |
| NP5-17-Q | Sum | | 99.86 | 100.00 | -0.14 | -0.1% |
| NP5-18-Q | $Al_2O_3$ | | 20.60 | 21.35 | -0.75 | -3.5% |
| NP5-18-Q | $B_2O_3$ | | 20.33 | 20.02 | 0.31 | 1.5% |
| NP5-18-Q | $Bi_2O_3$ | | 0.82 | 0.88 | -0.06 | -6.9% |
| NP5-18-Q | CaO | | 6.43 | 5.93 | 0.50 | 8.5% |
| NP5-18-Q | $Cr_2O_3$ | | 0.85 | 0.82 | 0.03 | 3.4% |
| NP5-18-Q | F | | 0.16 | 0.28 | -0.13 | -44.6% |
| NP5-18-Q | $Fe_2O_3$ | | 3.04 | 2.56 | 0.48 | 18.6% |
| NP5-18-Q | $Li_2O$ | | 1.25 | 1.60 | -0.36 | -22.2% |
| NP5-18-Q | MnO | | 1.68 | 1.73 | -0.06 | -3.2% |
| NP5-18-Q | $Na_2O$ | | 11.37 | 12.26 | -0.89 | -7.3% |
| NP5-18-Q | NiO | | 0.21 | 0.28 | -0.08 | -26.8% |
| NP5-18-Q | $P_2O_5$ | < | 0.12 | 0.02 | | |
| NP5-18-Q | PbO | | 0.13 | 0.14 | -0.01 | -5.0% |
| NP5-18-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-18-Q | $SiO_2$ | | 31.88 | 31.18 | 0.70 | 2.2% |
| NP5-18-Q | $SO_3$ | | 0.25 | 0.29 | -0.04 | -14.8% |
| NP5-18-Q | SrO | | 0.14 | 0.14 | 0.00 | 0.0% |
| NP5-18-Q | $ZrO_2$ | | 0.46 | 0.51 | -0.05 | -9.6% |
| NP5-18-Q | Sum | | 99.74 | 100.00 | -0.26 | -0.3% |
| NP5-19-Q | $Al_2O_3$ | | 21.40 | 22.43 | -1.03 | -4.6% |
| NP5-19-Q | $B_2O_3$ | | 13.50 | 13.81 | -0.31 | -2.3% |
| NP5-19-Q | $Bi_2O_3$ | | 1.97 | 2.08 | -0.11 | -5.3% |
| NP5-19-Q | CaO | | 2.66 | 2.48 | 0.18 | 7.3% |
| NP5-19-Q | $Cr_2O_3$ | | 2.02 | 1.94 | 0.08 | 4.2% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-19-Q | F | | 0.51 | 0.67 | -0.16 | -24.5% |
| NP5-19-Q | $Fe_2O_3$ | | 4.24 | 3.77 | 0.47 | 12.4% |
| NP5-19-Q | $Li_2O$ | | 2.59 | 2.92 | -0.33 | -11.3% |
| NP5-19-Q | MnO | | 4.16 | 4.08 | 0.08 | 2.1% |
| NP5-19-Q | $Na_2O$ | | 12.77 | 13.49 | -0.72 | -5.3% |
| NP5-19-Q | NiO | | 0.62 | 0.65 | -0.03 | -5.1% |
| NP5-19-Q | $P_2O_5$ | | 0.19 | 0.14 | 0.05 | 38.6% |
| NP5-19-Q | PbO | | 0.31 | 0.33 | -0.03 | -7.6% |
| NP5-19-Q | $RuO_2$ | < | 0.07 | 0.02 | | |
| NP5-19-Q | $SiO_2$ | | 29.36 | 28.99 | 0.37 | 1.3% |
| NP5-19-Q | $SO_3$ | | 0.63 | 0.69 | -0.06 | -9.1% |
| NP5-19-Q | SrO | | 0.33 | 0.32 | 0.01 | 2.2% |
| NP5-19-Q | $ZrO_2$ | | 1.23 | 1.19 | 0.04 | 3.7% |
| NP5-19-Q | Sum | | 98.55 | 100.00 | -1.45 | -1.4% |
| NP5-20-Q | $Al_2O_3$ | | 20.22 | 20.61 | -0.39 | -1.9% |
| NP5-20-Q | $B_2O_3$ | | 17.08 | 16.49 | 0.59 | 3.6% |
| NP5-20-Q | $Bi_2O_3$ | | 2.39 | 2.43 | -0.04 | -1.6% |
| NP5-20-Q | CaO | | 3.50 | 3.22 | 0.28 | 8.5% |
| NP5-20-Q | $Cr_2O_3$ | | 2.31 | 2.26 | 0.05 | 2.3% |
| NP5-20-Q | F | | 0.56 | 0.78 | -0.22 | -28.3% |
| NP5-20-Q | $Fe_2O_3$ | | 4.52 | 4.04 | 0.48 | 11.8% |
| NP5-20-Q | $Li_2O$ | | 2.91 | 3.35 | -0.44 | -13.1% |
| NP5-20-Q | MnO | | 4.87 | 4.77 | 0.10 | 2.1% |
| NP5-20-Q | $Na_2O$ | | 9.64 | 10.33 | -0.69 | -6.7% |
| NP5-20-Q | NiO | | 0.69 | 0.76 | -0.07 | -9.5% |
| NP5-20-Q | $P_2O_5$ | | 0.96 | 1.09 | -0.14 | -12.4% |
| NP5-20-Q | PbO | | 0.36 | 0.38 | -0.02 | -4.2% |
| NP5-20-Q | $RuO_2$ | < | 0.07 | 0.02 | | |
| NP5-20-Q | $SiO_2$ | | 27.60 | 26.91 | 0.69 | 2.6% |
| NP5-20-Q | $SO_3$ | | 0.70 | 0.80 | -0.11 | -13.1% |
| NP5-20-Q | SrO | | 0.38 | 0.37 | 0.01 | 3.8% |
| NP5-20-Q | $ZrO_2$ | | 1.34 | 1.39 | -0.05 | -3.5% |
| NP5-20-Q | Sum | | 100.09 | 100.00 | 0.09 | 0.1% |
| NP5-21-Q | $Al_2O_3$ | | 25.60 | 25.88 | -0.28 | -1.1% |
| NP5-21-Q | $B_2O_3$ | | 13.73 | 13.53 | 0.20 | 1.5% |
| NP5-21-Q | $Bi_2O_3$ | | 2.35 | 2.51 | -0.16 | -6.5% |
| NP5-21-Q | CaO | | 2.26 | 2.05 | 0.21 | 10.2% |
| NP5-21-Q | $Cr_2O_3$ | | 2.53 | 2.33 | 0.20 | 8.4% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-21-Q | F | | 0.61 | 0.81 | -0.20 | -24.7% |
| NP5-21-Q | $Fe_2O_3$ | | 3.29 | 2.61 | 0.68 | 26.0% |
| NP5-21-Q | $Li_2O$ | | 5.69 | 5.91 | -0.22 | -3.7% |
| NP5-21-Q | MnO | | 5.18 | 4.92 | 0.26 | 5.4% |
| NP5-21-Q | $Na_2O$ | | 8.31 | 9.33 | -1.02 | -11.0% |
| NP5-21-Q | NiO | | 0.81 | 0.78 | 0.03 | 4.2% |
| NP5-21-Q | $P_2O_5$ | | 1.96 | 1.94 | 0.02 | 0.9% |
| NP5-21-Q | PbO | | 0.39 | 0.40 | -0.01 | -3.3% |
| NP5-21-Q | $RuO_2$ | < | 0.07 | 0.02 | | |
| NP5-21-Q | $SiO_2$ | | 25.19 | 24.33 | 0.86 | 3.5% |
| NP5-21-Q | $SO_3$ | | 0.77 | 0.83 | -0.06 | -6.7% |
| NP5-21-Q | SrO | | 0.39 | 0.39 | 0.00 | 0.0% |
| NP5-21-Q | $ZrO_2$ | | 1.52 | 1.43 | 0.09 | 6.5% |
| NP5-21-Q | Sum | | 100.65 | 100.00 | 0.65 | 0.7% |
| NP5-22-Q | $Al_2O_3$ | | 22.91 | 22.91 | 0.00 | 0.0% |
| NP5-22-Q | $B_2O_3$ | | 18.88 | 18.50 | 0.38 | 2.0% |
| NP5-22-Q | $Bi_2O_3$ | | 2.34 | 2.36 | -0.02 | -0.7% |
| NP5-22-Q | CaO | | 6.01 | 5.16 | 0.85 | 16.4% |
| NP5-22-Q | $Cr_2O_3$ | | 2.39 | 2.20 | 0.19 | 8.6% |
| NP5-22-Q | F | | 0.55 | 0.76 | -0.21 | -27.6% |
| NP5-22-Q | $Fe_2O_3$ | | 2.53 | 1.91 | 0.62 | 32.5% |
| NP5-22-Q | $Li_2O$ | | 1.12 | 1.20 | -0.08 | -7.0% |
| NP5-22-Q | MnO | | 4.95 | 4.64 | 0.31 | 6.7% |
| NP5-22-Q | $Na_2O$ | | 10.28 | 10.85 | -0.58 | -5.3% |
| NP5-22-Q | NiO | | 0.77 | 0.73 | 0.04 | 5.3% |
| NP5-22-Q | $P_2O_5$ | | 1.42 | 1.35 | 0.07 | 4.9% |
| NP5-22-Q | PbO | | 0.36 | 0.37 | -0.01 | -2.2% |
| NP5-22-Q | $RuO_2$ | < | 0.07 | 0.02 | | |
| NP5-22-Q | $SiO_2$ | | 25.57 | 24.55 | 1.02 | 4.1% |
| NP5-22-Q | $SO_3$ | | 0.55 | 0.78 | -0.23 | -29.7% |
| NP5-22-Q | SrO | | 0.37 | 0.36 | 0.01 | 3.3% |
| NP5-22-Q | $ZrO_2$ | | 1.46 | 1.35 | 0.11 | 7.8% |
| NP5-22-Q | Sum | | 102.50 | 100.00 | 2.50 | 2.5% |
| NP5-23-Q | $Al_2O_3$ | | 28.44 | 28.66 | -0.22 | -0.8% |
| NP5-23-Q | $B_2O_3$ | | 17.94 | 17.30 | 0.64 | 3.7% |
| NP5-23-Q | $Bi_2O_3$ | | 1.41 | 1.47 | -0.07 | -4.4% |
| NP5-23-Q | CaO | | 7.13 | 6.58 | 0.55 | 8.4% |
| NP5-23-Q | $Cr_2O_3$ | | 1.45 | 1.37 | 0.08 | 5.5% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-23-Q | F | | 0.33 | 0.47 | -0.15 | -30.9% |
| NP5-23-Q | $Fe_2O_3$ | | 2.48 | 1.96 | 0.52 | 26.7% |
| NP5-23-Q | $Li_2O$ | | 2.69 | 2.89 | -0.20 | -6.9% |
| NP5-23-Q | MnO | | 2.84 | 2.88 | -0.04 | -1.4% |
| NP5-23-Q | $Na_2O$ | | 10.23 | 11.45 | -1.22 | -10.7% |
| NP5-23-Q | NiO | | 0.36 | 0.46 | -0.10 | -21.1% |
| NP5-23-Q | $P_2O_5$ | < | 0.12 | 0.03 | | |
| NP5-23-Q | PbO | | 0.23 | 0.23 | 0.00 | 0.0% |
| NP5-23-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-23-Q | $SiO_2$ | | 22.68 | 22.69 | -0.01 | -0.1% |
| NP5-23-Q | $SO_3$ | | 0.46 | 0.48 | -0.02 | -3.3% |
| NP5-23-Q | SrO | | 0.23 | 0.23 | 0.00 | 0.0% |
| NP5-23-Q | $ZrO_2$ | | 0.77 | 0.84 | -0.07 | -8.1% |
| NP5-23-Q | Sum | | 99.84 | 100.00 | -0.17 | -0.2% |
| NP5-24-Q | $Al_2O_3$ | | 24.19 | 24.42 | -0.23 | -1.0% |
| NP5-24-Q | $B_2O_3$ | | 18.28 | 17.41 | 0.87 | 5.0% |
| NP5-24-Q | $Bi_2O_3$ | | 0.89 | 0.95 | -0.06 | -6.6% |
| NP5-24-Q | CaO | | 3.35 | 3.08 | 0.27 | 8.8% |
| NP5-24-Q | $Cr_2O_3$ | | 0.94 | 0.88 | 0.06 | 6.4% |
| NP5-24-Q | F | | 0.20 | 0.30 | -0.10 | -34.0% |
| NP5-24-Q | $Fe_2O_3$ | | 2.38 | 1.83 | 0.55 | 30.1% |
| NP5-24-Q | $Li_2O$ | | 1.72 | 2.05 | -0.33 | -16.0% |
| NP5-24-Q | MnO | | 1.83 | 1.86 | -0.03 | -1.8% |
| NP5-24-Q | $Na_2O$ | | 13.71 | 14.99 | -1.28 | -8.5% |
| NP5-24-Q | NiO | | 0.22 | 0.29 | -0.07 | -25.5% |
| NP5-24-Q | $P_2O_5$ | | 0.41 | 0.56 | -0.15 | -27.0% |
| NP5-24-Q | PbO | | 0.15 | 0.15 | 0.00 | 0.0% |
| NP5-24-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-24-Q | $SiO_2$ | | 31.50 | 30.22 | 1.28 | 4.2% |
| NP5-24-Q | $SO_3$ | | 0.30 | 0.31 | -0.01 | -4.2% |
| NP5-24-Q | SrO | | 0.15 | 0.15 | 0.00 | 0.0% |
| NP5-24-Q | $ZrO_2$ | | 0.49 | 0.54 | -0.05 | -9.3% |
| NP5-24-Q | Sum | | 100.75 | 100.00 | 0.75 | 0.8% |
| NP5-25-Q | $Al_2O_3$ | | 27.87 | 28.95 | -1.08 | -3.7% |
| NP5-25-Q | $B_2O_3$ | | 13.29 | 12.87 | 0.42 | 3.3% |
| NP5-25-Q | $Bi_2O_3$ | | 1.56 | 1.65 | -0.09 | -5.4% |
| NP5-25-Q | CaO | | 1.42 | 1.46 | -0.04 | -2.7% |
| NP5-25-Q | $Cr_2O_3$ | | 1.54 | 1.54 | 0.00 | 0.0% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-25-Q | F | | 0.38 | 0.53 | -0.15 | -28.1% |
| NP5-25-Q | $Fe_2O_3$ | | 2.20 | 1.76 | 0.44 | 25.1% |
| NP5-25-Q | $Li_2O$ | | 5.15 | 5.63 | -0.48 | -8.5% |
| NP5-25-Q | MnO | | 3.11 | 3.25 | -0.14 | -4.3% |
| NP5-25-Q | $Na_2O$ | | 11.72 | 13.02 | -1.30 | -10.0% |
| NP5-25-Q | NiO | | 0.40 | 0.51 | -0.11 | -22.2% |
| NP5-25-Q | $P_2O_5$ | < | 0.14 | 0.36 | | |
| NP5-25-Q | PbO | | 0.25 | 0.26 | -0.01 | -5.0% |
| NP5-25-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-25-Q | $SiO_2$ | | 25.67 | 26.45 | -0.78 | -2.9% |
| NP5-25-Q | $SO_3$ | | 0.53 | 0.55 | -0.03 | -4.5% |
| NP5-25-Q | SrO | | 0.26 | 0.25 | 0.01 | 3.2% |
| NP5-25-Q | $ZrO_2$ | | 0.84 | 0.95 | -0.11 | -11.2% |
| NP5-25-Q | Sum | | 96.39 | 100.00 | -3.61 | -3.6% |
| NP5-26-Q | $Al_2O_3$ | | 20.12 | 20.79 | -0.67 | -3.2% |
| NP5-26-Q | $B_2O_3$ | | 20.29 | 20.81 | -0.53 | -2.5% |
| NP5-26-Q | $Bi_2O_3$ | | 2.32 | 2.34 | -0.02 | -0.9% |
| NP5-26-Q | CaO | | 1.34 | 1.34 | 0.00 | 0.0% |
| NP5-26-Q | $Cr_2O_3$ | | 2.23 | 2.18 | 0.05 | 2.4% |
| NP5-26-Q | F | | 0.53 | 0.75 | -0.22 | -28.9% |
| NP5-26-Q | $Fe_2O_3$ | | 2.66 | 2.18 | 0.48 | 21.8% |
| NP5-26-Q | $Li_2O$ | | 0.46 | 0.51 | -0.05 | -10.0% |
| NP5-26-Q | MnO | | 4.56 | 4.59 | -0.03 | -0.6% |
| NP5-26-Q | $Na_2O$ | | 13.72 | 14.40 | -0.68 | -4.7% |
| NP5-26-Q | NiO | | 0.73 | 0.73 | 0.00 | 0.5% |
| NP5-26-Q | $P_2O_5$ | | 0.27 | 0.22 | 0.05 | 21.4% |
| NP5-26-Q | PbO | | 0.36 | 0.37 | -0.01 | -2.4% |
| NP5-26-Q | $RuO_2$ | < | 0.07 | 0.02 | | |
| NP5-26-Q | $SiO_2$ | | 26.37 | 26.30 | 0.07 | 0.3% |
| NP5-26-Q | $SO_3$ | | 0.56 | 0.77 | -0.21 | -27.1% |
| NP5-26-Q | SrO | | 0.37 | 0.36 | 0.01 | 1.4% |
| NP5-26-Q | $ZrO_2$ | | 1.40 | 1.34 | 0.06 | 4.3% |
| NP5-26-Q | Sum | | 98.35 | 100.00 | -1.65 | -1.6% |
| NP5-27-Q | $Al_2O_3$ | | 25.60 | 26.70 | -1.10 | -4.1% |
| NP5-27-Q | $B_2O_3$ | | 16.15 | 16.57 | -0.42 | -2.5% |
| NP5-27-Q | $Bi_2O_3$ | | 1.07 | 1.07 | -0.01 | 0.0% |
| NP5-27-Q | CaO | | 5.14 | 4.73 | 0.41 | 8.7% |
| NP5-27-Q | $Cr_2O_3$ | | 1.08 | 0.99 | 0.09 | 9.5% |

**Table B.1.** Composition Analysis Results Including Reference Material and EA Glasses (cont'd)

| Glass ID[(a)] | Oxide | BDL (<)[(b)] | Measured (mass%) | Targeted (mass%) | Difference of Measured Versus Targeted (mass%) | %Relative Difference of Measured versus Targeted |
|---|---|---|---|---|---|---|
| NP5-27-Q | F | | 0.21 | 0.34 | -0.13 | -38.2% |
| NP5-27-Q | $Fe_2O_3$ | | 2.78 | 2.25 | 0.53 | 23.4% |
| NP5-27-Q | $Li_2O$ | | 4.88 | 5.28 | -0.40 | -7.7% |
| NP5-27-Q | MnO | | 2.04 | 2.10 | -0.06 | -2.7% |
| NP5-27-Q | $Na_2O$ | | 9.32 | 10.08 | -0.76 | -7.6% |
| NP5-27-Q | NiO | | 0.33 | 0.33 | 0.00 | 0.0% |
| NP5-27-Q | $P_2O_5$ | < | 0.18 | 0.10 | | |
| NP5-27-Q | PbO | | 0.16 | 0.17 | -0.01 | -4.1% |
| NP5-27-Q | $RuO_2$ | < | 0.07 | 0.01 | | |
| NP5-27-Q | $SiO_2$ | | 28.03 | 28.16 | -0.14 | -0.5% |
| NP5-27-Q | $SO_3$ | | 0.33 | 0.35 | -0.02 | -5.7% |
| NP5-27-Q | SrO | | 0.16 | 0.16 | 0.00 | 0.0% |
| NP5-27-Q | $ZrO_2$ | | 0.67 | 0.61 | 0.06 | 9.3% |
| NP5-27-Q | Sum | | 98.18 | 100.00 | -1.82 | -1.8% |
| BL3 | $Al_2O_3$ | | 28.86 | 28.50 | 0.36 | 1.3% |
| BL3 | $B_2O_3$ | | 18.75 | 17.20 | 1.55 | 9.0% |
| BL3 | $Bi_2O_3$ | | 0.62 | 0.65 | -0.03 | -5.2% |
| BL3 | CaO | | 0.50 | 0.65 | -0.15 | -22.8% |
| BL3 | $Cr_2O_3$ | | 1.20 | 1.10 | 0.10 | 9.0% |
| BL3 | F | | 0.20 | 0.30 | -0.11 | -35.0% |
| BL3 | $Fe_2O_3$ | | 3.06 | 2.50 | 0.56 | 22.2% |
| BL3 | $Li_2O$ | | 4.90 | 5.00 | -0.10 | -2.0% |
| BL3 | MnO | | 0.96 | 1.00 | -0.04 | -3.9% |
| BL3 | $Na_2O$ | | 11.73 | 12.50 | -0.77 | -6.2% |
| BL3 | $P_2O_5$ | | 0.40 | 0.70 | -0.30 | -42.6% |
| BL3 | $RuO_2$ | < | 0.07 | 0.05 | | |
| BL3 | $SiO_2$ | | 30.06 | 29.35 | 0.71 | 2.4% |
| BL3 | $SO_3$ | | 0.24 | 0.25 | -0.01 | -4.4% |
| BL3 | $ZrO_2$ | | 0.17 | 0.25 | -0.08 | -30.4% |
| BL3 | Sum | | 101.71 | 100.00 | 2.06 | 2.1% |

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