

Contract No:

This document was prepared in conjunction with work accomplished under Contract No. 89303321CEM000080 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Disclaimer:

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

- 1) warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
- 2) representation that such use or results of such use would not infringe privately owned rights; or
- 3) endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.



**Savannah River
National Laboratory®**

A U.S. DEPARTMENT OF ENERGY NATIONAL LAB • SAVANNAH RIVER SITE • AIKEN, SC • USA

Composition Measurements and Product Consistency Test Results for the EMHQ- LBE-04-B Glass

M. C. Hsieh

March 2022

SRNL-STI-2022-00118, Revision 0

SRNL.DOE.GOV

DISCLAIMER

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

**Prepared for
U.S. Department of Energy**

Keywords: *Hanford, WTP, low-activity waste, waste glass*

Retention: *Permanent*

Composition Measurements and Product Consistency Test Results for the EMHQ-LBE-04-B Glass

M. C. Hsieh

March 2022

Savannah River National Laboratory is operated by
Battelle Savannah River Alliance for the U.S. Department
of Energy under Contract No. 89303321CEM000080.



REVIEWS AND APPROVALS

AUTHORS:

M. C. Hsieh, Applied Materials Research

TECHNICAL REVIEW:

A. N. Stanfield, Applied Materials Research, Reviewed per E7 2.60

APPROVAL:

J. Manna, Director
Materials Technology Division

ACKNOWLEDGEMENTS

The author would like to thank Matt Alexander, Daniel Jones, Whitney Riley, and Kim Wyszynski at the Savannah River National Laboratory for their skilled assistance with the sample analyses described in this report. The author thanks Renee Russell at the Pacific Northwest National Laboratory for the review of these data and the report. Funding from the United States Department of Energy through Inter-Entity Work Order HAN-M0SRV00101 as managed by Albert A. Kruger is gratefully acknowledged.

EXECUTIVE SUMMARY

This report provides the results from the chemical analyses of the glass composition and the Product Consistency Test leachate analyses for the EMHQ-LBE-04-B glass. These data will be used in the development, validation, and implementation of enhanced property/composition models for waste glass vitrification at Hanford.

Chemical analyses were performed on a sample of the EMHQ-LBE-04-B glass. The relative differences between the targeted and measured concentrations of Li_2O , Na_2O , and SO_3 for the glass were greater than 10%. The relative differences between the measured compositions of the EMHQ-LBE-04-B and EMHQ-LBE-04 glasses were all less than 10%.

The measured and targeted EMHQ-04-LBE-B glass compositions were used in the normalization of the Product Consistency Test results. The NC_B , NC_{Na} , and NC_{Si} values were less than the Hanford Tank Waste Treatment Immobilization Plant low-activity waste constraint of 4 g/L. The measured concentrations of the analytes in the test blank samples included with the Product Consistency Test leachates were below detection limits. The average measured concentrations of B, Li, Na, and Si for the Approved Reference Material-1 glass fell within control chart values indicating proper test performance.

TABLE OF CONTENTS

LIST OF TABLES.....	viii
LIST OF ABBREVIATIONS.....	ix
1.0 Introduction.....	1
2.0 Experimental Procedure.....	1
2.1 Quality Assurance.....	1
2.2 Glass Selected for Study.....	1
2.3 Glass Composition Analysis.....	2
2.4 PCT Leachate Analysis.....	2
3.0 Results and Discussion.....	3
3.1 EMHQ-LBE-04-B Chemical Composition Measurements.....	3
3.1.1 Treatment of Detection Limits.....	3
3.1.2 Measured versus Target Compositions.....	3
3.1.3 Comparison of EMHQ-LBE-04-B and EMHQ-LBE-04 Measured Compositions.....	4
3.2 Measured Compositions of the PCT Leachates.....	5
3.3 Normalization of PCT Data.....	6
4.0 Summary.....	7
5.0 References.....	7
Appendix A. Tables Supporting the Glass Composition Measurements of EMHQ-LBE-04-B.....	A-1
Appendix B. Tables Containing the Measurement Data for the EMHQ-LBE-04-B PCT Leachates.....	B-1

LIST OF TABLES

Table 2-1. Identifiers for the PCT Leachates	2
Table 2-2. Preparation and Measurement Methods used in Reporting the Analyte Concentrations of the EMHQ-LBE-04-B Study Glass and LRM Standard Glass.....	2
Table 3-1. Comparison of Measured and Target EMHQ-LBE-04-B Glass Compositions	4
Table 3-2. Comparison of EMHQ-LBE-04-B and EMHQ-LBE-04 Measured Compositions.....	5
Table 3-3. Average PCT Leachate Measurements (mg/L).....	5
Table 3-4. Normalized PCT Results (g/L).....	7

LIST OF ABBREVIATIONS

ARM-1	Approved Reference Material-1
ASTM	American Society for Testing and Materials
BDL	below detection limit
DF	dilution factor
DOE	U. S. Department of Energy
IC	ion chromatography
ICP-OES	inductively coupled plasma – optical emission spectroscopy
ID	identifier
KH	potassium hydroxide fusion
LAW	low-activity waste
LM	lithium metaborate fusion
LRM	low-activity test reference material
NCi	normalized concentration of element “i”
ORP	Office of River Protection
PCT	Product Consistency Test
PF	sodium peroxide fusion
PNNL	Pacific Northwest National Laboratory
%RSD	percent relative standard deviation
seq	sequence
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
std	High Purity Standards ICP multi-element custom solution SM-744-013
TTQAP	Task Technical and Quality Assurance Plan
wt. %	weight percent
WTP	Waste Treatment and Immobilization Plant

1.0 Introduction

The U.S. Department of Energy (DOE) is responsible for building the Hanford Tank Waste Treatment and Immobilization Plant (WTP) at the Hanford site in Washington to remediate 55 million gallons of radioactive waste that is temporarily stored in 177 underground tanks. The Office of River Protection (ORP) has requested that the Savannah River National Laboratory (SRNL) contribute in areas of recognized capabilities and expertise for glass waste form development to support successful startup of the WTP.

Successful efforts have allowed for demonstration of greatly enhanced treatment efficiencies of those projected from the minimum requirements set forth in the WTP Contract^a Additional flexibility and expansion of the qualified glass forming region are the current focus.¹ SRNL support of this work is defined in the Task Technical and Quality Assurance Plan (TTQAP).²

This report provides results from the chemical analyses of the EMHQ-LBE-04-B glass and the resulting Product Consistency Test (PCT) leachates. The glasses were selected as part of a broader study of the influence of glass composition on chemical durability, sulfur retention, and other properties.³ The resulting data will be used in the development, validation, and implementation of enhanced property/composition models for nuclear waste glasses.¹

2.0 Experimental Procedure

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in Savannah River Site (SRS) Manual E7, Procedure 2.60.⁴ SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011.⁵ Laboratory data for this study were recorded in the SRNL Electronic Laboratory Notebook system, experiment L6390-00441-04. The glass provided by the Pacific Northwest National Laboratory (PNNL) was designed and fabricated following a Task Plan.¹

2.2 Glass Selected for Study

The glass composition in this study was selected and fabricated by PNNL. A sample of the glass was received at SRNL for chemical composition analysis. The PNNL identifier for this sample was “EMHQ-LBE-04-B” and the SRNL identifier was S-13644.

American Society for Testing and Materials (ASTM) C1285-21 PCT Method A⁶ was performed on the study glass. The resulting PCT leachates were sent to SRNL for chemical analysis. Identifiers (IDs) for the PCT leachates are listed in Table 2-1.

^a Contract DE-AC27-01RV14136, as amended, U.S. Department of Energy, Richland, WA (2000).

Table 2-1. Identifiers for the PCT Leachates

PNNL ID	SRNL ID
EMHQ-LBE-04-B-PCT-A	S-13647
EMHQ-LBE-04-B-PCT-B	S-13648
EMHQ-LBE-04-B-PCT-C	S-13649
ARM-1-STD-GLASS-PCT-A-020822	S-13650
ARM-1-STD-GLASS-PCT-B-020822	S-13651
ARM-1-STD-GLASS-PCT-C-020822	S-13652
MQ-WATER BLANK-PCT-A-020822	S-13653
MQ-WATER BLANK-PCT-B-020822	S-13654

2.3 Glass Composition Analysis

Three dissolution techniques (potassium hydroxide fusion (KH), lithium metaborate fusion (LM), and sodium peroxide fusion (PF)) were used for preparing the EMHQ-LBE-04-B glass sample for analysis.⁷⁻⁹

The EMHQ-LBE-04-B sample was analyzed twice for each element of interest by inductively coupled plasma – optical emission spectroscopy (ICP-OES)¹⁰ or ion chromatography (IC),¹¹ for a total of two measurements per element per glass. A sample of the low-activity test reference material (LRM) was included with the sample glass measurements. The LRM composition reported as the “Consensus Average” is used as the reference composition of this glass.¹² The preparation and measurement methods used for each of the reported glass components are listed in Table 2-2.

Table 2-2. Preparation and Measurement Methods used in Reporting the Analyte Concentrations of the EMHQ-LBE-04-B Study Glass and LRM Standard Glass

Analyte	Measurement Method	Preparation Method
Al	ICP-OES	PF
B	ICP-OES	PF
Ca	ICP-OES	LM
Cl	IC	KH
Cr	ICP-OES	LM
F	IC	KH
Fe	ICP-OES	LM
K	ICP-OES	LM
Li	ICP-OES	PF
Mg	ICP-OES	LM
Mn	ICP-OES	LM
Na	ICP-OES	LM
P	ICP-OES	PF
S	ICP-OES	LM
Si	ICP-OES	PF
Ti	ICP-OES	LM
V	ICP-OES	LM
Zr	ICP-OES	LM

2.4 PCT Leachate Analysis

PNNL supplied leachates resulting from the PCT of the EMHQ-LBE-04-B study glass. Samples were measured twice by ICP-OES along with samples of High Purity Standards ICP multi-element custom solution SM-744-013 (std). PNNL provided the dilution factor (DF) used in preparing the PCT leachates for analysis. The leachate measurements were adjusted using the provided DF of 5. Normalized release values were calculated for the study glass based on the targeted and measured glass compositions.

3.0 Results and Discussion

JMP® Version 16.0.0 (SAS Institute, Inc.)¹³ was used to support these analyses.

3.1 EMHQ-LBE-04-B Chemical Composition Measurements

Table A-1, Table A-2, and Table A-3 in Appendix A provide the elemental concentration measurements in weight percent (wt.%) from the EMHQ-LBE-04-B and LRM glasses prepared using KH, LM, and PF methods, respectively. Table A-4 in Appendix A provides a summary of the average compositions, targeted compositions and some associated differences and relative differences for the LRM glass.

3.1.1 Treatment of Detection Limits

The elemental concentrations in Table A-1, Table A-2, and Table A-3 in Appendix A were converted to oxide concentrations by multiplying the values of each element by the gravimetric factor for the corresponding oxide. A concentration measurement that was reported to be below the detection limit was set to the detection limit for the purposes of data review and calculating a sum of oxides for each glass. Concentration measurements that were below the detection limit (BDL) are denoted with a less than symbol (<).

3.1.2 Measured versus Target Compositions

Table 3-1 provides a summary of the average compositions, targeted compositions and some associated differences and relative differences. The measured sums of oxides for the EMHQ-LBE-04-B glass is 99.8 wt.%, indicating acceptable recovery of the glass components.¹⁴ Entries in Table 3-1 show the relative differences between the measured and targeted values for the analytes with measured and targeted values above 1 wt.%. The relative differences were shaded if they are 10% or more. The relative differences for Li₂O, Na₂O, and SO₃ were greater than 10%.

Table 3-1. Comparison of Measured and Target EMHQ-LBE-04-B Glass Compositions

PNNL ID	Oxide	Measured (wt.%)	Target (wt.%)	Difference of Measured versus Target	% Difference Measured versus Target
EMHQ-LBE-04-B	Al ₂ O ₃	3.62	3.54	0.078	2%
EMHQ-LBE-04-B	B ₂ O ₃	12.7	13.5	-0.781	-6%
EMHQ-LBE-04-B	CaO	12.7	12.2	0.491	4%
EMHQ-LBE-04-B	Cl ⁻	0.0704	0.0760	-0.006	
EMHQ-LBE-04-B	Cr ₂ O ₃	<0.0365	0.0250	0.012	
EMHQ-LBE-04-B	F ⁻	0.0707	0.0950	-0.024	
EMHQ-LBE-04-B	Fe ₂ O ₃	0.119	0.131	-0.012	
EMHQ-LBE-04-B	K ₂ O	0.0592	0.0870	-0.028	
EMHQ-LBE-04-B	Li ₂ O	1.96	1.67	0.286	17%
EMHQ-LBE-04-B	MgO	0.203	0.180	0.023	
EMHQ-LBE-04-B	MnO	<0.0129	0.0130	0	
EMHQ-LBE-04-B	Na ₂ O	11.6	9.81	1.769	18%
EMHQ-LBE-04-B	P ₂ O ₅	<0.229	0.158	0.071	
EMHQ-LBE-04-B	SiO ₂	48.8	49.6	-0.824	-2%
EMHQ-LBE-04-B	SO ₃	1.39	1.69	-0.303	-18%
EMHQ-LBE-04-B	TiO ₂	0.0799	0.0910	-0.011	
EMHQ-LBE-04-B	V ₂ O ₅	4.27	4.03	0.237	6%
EMHQ-LBE-04-B	ZrO ₂	1.88	2.00	-0.116	-6%
EMHQ-LBE-04-B	Sum of Oxides	99.8	98.9	0.861	1%

3.1.3 Comparison of EMHQ-LBE-04-B and EMHQ-LBE-04 Measured Compositions

Table 3-2 provides a summary of the average EMHQ-LBE-04-B measured compositions, average EMHQ-LBE-04 measured compositions¹⁵, and relative differences with measured values above 1 wt.%. None of the relative differences were greater than 10%.

Table 3-2. Comparison of EMHQ-LBE-04-B and EMHQ-LBE-04 Measured Compositions

PNNL ID	Oxide	Measured (wt.%)	Measured EMHQ-LBE-04 (wt.%)	Difference of Measured versus Target	% Difference Measured versus Target
EMHQ-LBE-04-B	Al ₂ O ₃	3.62	3.58	0.040	1%
EMHQ-LBE-04-B	B ₂ O ₃	12.7	12.4	0.300	2%
EMHQ-LBE-04-B	CaO	12.7	11.6	1.100	9%
EMHQ-LBE-04-B	Cl ⁻	0.0704	0.0679	0.003	
EMHQ-LBE-04-B	Cr ₂ O ₃	<0.0365	0.0223	0.014	
EMHQ-LBE-04-B	F ⁻	0.0707	0.0708	0.000	
EMHQ-LBE-04-B	Fe ₂ O ₃	0.119	0.12	-0.001	
EMHQ-LBE-04-B	K ₂ O	0.0592	0.0557	0.004	
EMHQ-LBE-04-B	Li ₂ O	1.96	1.85	0.110	6%
EMHQ-LBE-04-B	MgO	0.203	0.197	0.006	
EMHQ-LBE-04-B	MnO	<0.0129	<0.0129	0.000	
EMHQ-LBE-04-B	Na ₂ O	11.6	11.5	0.100	1%
EMHQ-LBE-04-B	P ₂ O ₅	<0.229	0.144	0.085	
EMHQ-LBE-04-B	SiO ₂	48.8	47.8	1.000	2%
EMHQ-LBE-04-B	SO ₃	1.39	1.35	0.040	3%
EMHQ-LBE-04-B	TiO ₂	0.0799	0.0746	0.005	
EMHQ-LBE-04-B	V ₂ O ₅	4.27	4.02	0.250	6%
EMHQ-LBE-04-B	ZrO ₂	1.88	1.96	-0.080	-4%
EMHQ-LBE-04-B	Sum of Oxides	99.8	96.9	2.900	3%

3.2 Measured Compositions of the PCT Leachates

Table 3-3 provides the average elemental concentration measurements in mg/L for the PCT leachates. These values have been adjusted using the provided DF of 5 to correct for dilutions performed at PNNL. Note that the measured concentrations of the analytes in the test blank samples were BDL and denoted by a less than symbol (<).

Table 3-3. Average PCT Leachate Measurements (mg/L)

PNNL ID	B	Li	Na	Si
EMHQ-LBE-04-B-PCT-A	32.5	8.33	81.8	93.0
EMHQ-LBE-04-B-PCT-B	34.6	8.75	86.8	96.0
EMHQ-LBE-04-B-PCT-C	29.5	7.40	76.8	94.0
ARM-1-STD-GLASS-PCT-A-020822	19.2	15.8	41.7	68.0
ARM-1-STD-GLASS-PCT-B-020822	18.6	14.2	40.4	66.8
ARM-1-STD-GLASS-PCT-C-020822	18.1	13.8	40.4	66.0
MQ-WATER BLANK-PCT-A-020822	<5.00	<5.00	<5.00	<5.00
MQ-WATER BLANK-PCT-B-020822	<5.00	<5.00	<5.00	<5.00

Table B-1 in Appendix B provides the elemental concentration measurements in mg/L for the PCT leachates and standard solutions as measured by ICP-OES in analytical sequence. These values are shown as received from the analytical laboratory and after using the provided DF of 5 to correct for dilutions performed at PNNL.

Table B-2 in Appendix B provides measurements for the samples of the approved reference material (ARM-1) reference glass included in the PCTs after using the provided DF of 5 to correct for dilutions performed at PNNL. One of the duplicate Li measurements for ARM-1-STD-GLASS-PCT-A-020822 was outside of the control chart values. This is likely due to differences in the ICP-OES calibration curves between the two analysis blocks. The average of the two Li measurements for this sample falls within the control chart

values. Note that all other measured concentrations of the analytes fell within control chart values indicating proper test performance.¹⁶

Following the guidance in ASTM C1285,⁶ the mean, standard deviation, and percent relative standard deviation (%RSD) were determined for each element present in the solution standard for each analytical block. As shown in Table B-3 in Appendix B, the mean value for each analytical block was found to be less than 10% from the reference value (i.e., a percent relative bias less than 10%), and the %RSD was less than 10% for each element. Thus, these analytical results are acceptable per the criteria in ASTM C1285⁶, which indicates no significant issues with the analytical outcomes for the measurements of the PCT leachates.

3.3 Normalization of PCT Data

The PCT leachate data were used to determine normalized concentrations for each element of interest using both the targeted and average measured compositions¹⁵ of the glasses following the expression given in ASTM C1285.⁶

$$NC_i = \frac{c_i(\text{sample})}{f_i}$$

where NC_i is the normalized concentration in units of $\text{g}_{\text{waste form}}/\text{L}_{\text{leachant}}$, $c_i(\text{sample})$ is the concentration of element “i” in the leachate in units of g/L (corrected for the dilutions performed at PNNL), and f_i is the mass fraction of element “i” in the unleached glass in units of $\text{g}_i/\text{g}_{\text{glass}}$.^b

An equation was developed to allow for calculation of the NC_i values using the units of measurement provided with the analytical results for this study and to accommodate the triplicate leachate measurements for each of the study glasses. Note that the symbols in this second equation were kept consistent with those used in ASTM C1285,⁶ but the units of measurement differ. The common logarithm of the normalized concentration for each element “i” (NC_i) for each of the study glasses was determined using the equation:

$$\log_{10}(NC_i) = \overline{\log_{10} c_i} - [1 + \log_{10} f_i]$$

where NC_i remains in units of $\text{g}_{\text{waste form}}/\text{L}_{\text{leachant}}$, $\overline{\log_{10} c_i}$ is the average of the common logarithms of the measured concentrations of element “i” in the triplicate leachates in units of mg/L (corrected for the dilutions performed at PNNL as discussed in Section 3.2), and $\log_{10} f_i$ is either the common logarithm of the targeted concentration of element “i” in the glass in units of wt.% or the common logarithm of the average measured concentration of element “i” in the glass in units of wt.% (reported in Table 3-1).

Table 3-4 provides the normalized PCT responses for B, Li, Na, and Si, for each of the EMHQ-LBE-04-B study glass as well as the response for the ARM-1 reference glass¹⁷. The results are grouped by compositional view.

The NC_B , NC_{Na} , and NC_{Si} values fell below 4 g/L for all glasses, meeting the WTP constraints.^c

^b Note that the waste forms in this study were assumed to be of similar density. The PCT-A reference volume of leachant to sample mass ratio was used, and the 100 to 200 mesh reference particle size was used. Thus, no adjustment for the density of the glasses was made in normalizing the PCT results. Data provided in the appendices of this report allow for the calculation of normalized elemental mass loss (NL_i) if glass densities are measured at a later date.

^c Contract DE-AC27-01RV14136, as amended, U.S. Department of Energy, Richland, WA (2000).

Table 3-4. Normalized PCT Results (g/L)

Glass ID	Comp. View	NC _B (g/L)	NC _{Li} (g/L)	NC _{Na} (g/L)	NC _{Si} (g/L)
EMHQ-LBE-04-B	Target	0.0764	1.05	1.12	0.407
EMHQ-LBE-04-B	Measured	0.812	0.893	0.948	0.413
ARM-1-STD-GLASS-PCT-020822	Reference	0.528	0.615	0.568	0.308
EMHQ-LBE-04 ¹⁵	Target	0.856	1.19	1.22	0.397
EMHQ-LBE-04 ¹⁵	Measured	0.932	1.07	1.04	0.412
EMHQ-LBE-STD ¹⁵	Reference	0.469	0.621	0.478	0.281

4.0 Summary

Chemical analyses were performed on a sample of the EMHQ-LBE-04-B glass. The relative differences between the targeted and measured concentrations of Li₂O, Na₂O, and SO₃ for the glass were greater than 10%. The relative differences between the measured compositions of the EMHQ-LBE-04-B and EMHQ-LBE-04 glasses were all less than 10%.

The measured and targeted EMHQ-04-LBE-B glass compositions were used in the normalization of the Product Consistency Test results. The NC_B, NC_{Na}, and NC_{Si} values were less than the WTP low-activity waste constraint of 4 g/L. The measured concentrations of the analytes in the test blank samples included with the PCT leachates were below detection limits. The average measured concentrations of B, Li, Na, and Si for the ARM-1 glass fell within control chart values indicating proper test performance.

5.0 References

1. R.L. Russell, "EMHQ LAW Glass Composition Boundary Expansion," Pacific Northwest National Laboratory, Richland, WA, EWG-TP-169, Revision 0.0, 2021.
2. K.M. Fox, "Task Technical and Quality Assurance Plan for Hanford Waste Glass Development and Characterization," Savannah River National Laboratory, Aiken, SC, SRNL-RP-2013-00692, Revision 1, 2016.
3. D.K. Peeler, D.S. Kim, J.D. Vienna, M.J. Schweiger, and G.F. Piepel, "Office of River Protection Advanced Low-Activity Waste Glass Research and Development Plan," Pacific Northwest National Laboratory, Richland, WA, PNNL-24883, EWG-RPT-008, Revision 0, 2015.
4. "Technical Reviews," Savannah River Site, Aiken, SC, Manual E7, Procedure 2.60, Rev. 20, 2021.
5. "Savannah River National Laboratory Technical Report Design Check Guidelines," Westinghouse Savannah River Company, Aiken, SC, WSRC-IM-2002-00011, Rev. 2, 2004.
6. ASTM, "Standard Test Methods for Determining Chemical Durability of Nuclear, Hazardous, and Mixed Waste Glasses and Multiphase Glass Ceramics: The Product Consistency Test (PCT)," ASTM International, West Conshohocken, PA, C1285 - 21, 2021.
7. "Sample Dissolution Using Potassium Hydroxide Fusion," Savannah River National Laboratory, Aiken, SC, Manual L29, Procedure ITS-0035, Rev. 3, 2015.
8. "Lithium Metaborate Fusion Preparation," Savannah River National Laboratory, Aiken, SC, Manual L33, Procedure 0071, Rev. 0, 2021.

9. “Dissolution of Glass, Sludge, and Slurry Samples Using $\text{Na}_2\text{O}_2/\text{NaOH}/\text{HCl}$,” Savannah River National Laboratory, Aiken, SC, Manual L33, Procedure 0040, Rev. 0, 2022.
10. “Calibration, Verification, and Operation of the Agilent 5110 ICP-OES Inductively Coupled Plasma-Optical Emission Spectrometer,” Savannah River National Laboratory, Aiken, SC, Manual L33, Procedure 0242, Rev. 1, 2021.
11. “Anion Analysis Using the Dionex ICS 6000 Ion Chromatograph,” Savannah River National Laboratory, Aiken, SC, Manual L33, Procedure 0244, Revision 1, 2020.
12. W.L. Ebert and S.F. Wolf, “Round-Robin Testing of a Reference Glass for Low-Activity Waste Forms,” Argonne National Laboratory, Argonne, IL, ANL-99/22, Revision 0, 1999.
13. JMP® Version 16.0.0, SAS Institute Inc., Cary, NC, 2021.
14. C.M. Jantzen, “Verification of Glass Composition and Strategy for SGM and DWPF Glass Composition Determination,” E. I du Pont de Nemours & Co., Savannah River Laboratory, Aiken, SC, DPST-86-708, 1987.
15. M.C. Hsieh, “Composition Measurements of the EMHQ LAW Glasses,” Savannah River National Laboratory, Aiken, SC, SRNL-STI-2021-00678. Revision 0, 2022.
16. C.M. Jantzen, J.B. Pickett, K.G. Brown, T.B. Edwards, and D.C. Beam, “Process/Product Models for the Defense Waste Processing Facility (DWPF): Part I. Predicting Glass Durability from Composition Using a Thermodynamic Hydration Energy Reaction Model (THERMO),” Westinghouse Savannah River Company, Aiken, SC, WSRC-TR-93-672, Rev. 1, 1995.
17. G.B. Mellinger and J.L. Daniel, “Approved Reference and Testing Materials for Use in Nuclear Waste Management Research and Development Programs,” Pacific Northwest Laboratory, Richland, WA, PNL-4955-2, Revision 0, 1984.

Appendix A. Tables Supporting the Glass Composition Measurements of EMHQ-LBE-04-B

Table A-1. KH Measurements (wt.%) of the LRM Glass and EMHQ-LBE-04-B

PNNL ID	Lab ID	Cl⁻	F⁻
LRM	LRM	<0.0250	0.904
EMHQ-LBE-04-B	S-13644	0.0667	0.0705
EMHQ-LBE-04-B	S-13644	0.0741	0.0709

Table A-2. LM Measurements (wt.%) of the LRM Glass and EMHQ-LBE-04-B

PNNL ID	Lab ID	Ca	Cr	Fe	K	Mg	Mn	Na	S	Ti	V	Zr
LRM	LRM	0.365	0.131	0.949	1.15	0.0658	0.0622	15.1	0.0858	0.0604	<0.100	0.659
EMHQ-LBE-04-B	S-13644	9.10	<0.0250	0.0836	0.0464	0.122	<0.0100	8.67	0.553	0.0478	2.40	1.39
EMHQ-LBE-04-B	S-13644	9.04	<0.0250	0.0822	0.0519	0.123	<0.0100	8.51	0.558	0.0480	2.38	1.40

Table A-3. PF Measurements (wt.%) of the LRM Glass and EMHQ-LBE-04-B

PNNL ID	Lab ID	Al	B	Li	P	Si
LRM	LRM	5.22	2.28	<0.100	0.204	25.1
EMHQ-LBE-04-B	S-13644	1.90	3.92	0.906	<0.100	22.5
EMHQ-LBE-04-B	S-13644	1.93	3.98	0.911	<0.100	23.1

Table A-4. Comparison of Measured and Reference Compositions for the LRM Glass

PNNL ID	Oxide	Measurement (wt.%)	Target (wt.%)	Difference of Measured versus Target	% Difference Measured versus Target
LRM	Al ₂ O ₃	9.86	9.51	0.353	4%
LRM	B ₂ O ₃	7.34	7.85	-0.509	-6%
LRM	CaO	0.511	0.54	-0.029	
LRM	Cl	<0.025	0	0.025	
LRM	Cr ₂ O ₃	0.191	0.19	0.001	
LRM	F	0.904	0.86	0.044	
LRM	Fe ₂ O ₃	1.36	1.38	-0.023	-2%
LRM	K ₂ O	1.39	1.48	-0.095	-6%
LRM	Li ₂ O	<0.215	0.11	0.105	
LRM	MgO	0.109	0.1	0.009	
LRM	MnO	0.0803	0.08	0	
LRM	Na ₂ O	20.4	20	0.325	2%
LRM	P ₂ O ₅	0.467	0.54	-0.073	
LRM	SiO ₂	53.7	54.2	-0.504	-1%
LRM	SO ₃	0.214	0.3	-0.086	
LRM	TiO ₂	0.101	0.1	0.001	
LRM	V ₂ O ₅	<0.179	0	0.179	
LRM	ZrO ₂	0.89	0.93	-0.04	
LRM	Sum of Oxides	97.9	98.1	-0.235	0%

Appendix B. Tables Containing the Measurement Data for the EMHQ-LBE-04-B PCT Leachates

Table B-1. PCT Measurements (mg/L)

				As Received Measurements					Dilution-Corrected Measurements			
PNNL ID	Block	Seq	Lab ID	B	Li	Na	Si	DF	B	Li	Na	Si
PCT std	1	1	PCT std 1	19.5	10.4	79.1	49.9	1	19.5	10.4	79.1	49.9
EMHQ-LBE-04-B-PCT-B	1	2	S-13648	6.75	1.73	16.9	19.0	5	33.8	8.65	84.5	95.0
ARM-1-STD-GLASS-PCT-C-020822	1	3	S-13652	3.43	2.86	7.89	12.9	5	17.2	14.3	39.5	64.5
EMHQ-LBE-04-B-PCT-A	1	4	S-13647	6.27	1.62	15.7	18.3	5	31.4	8.10	78.5	91.5
MQ-WATER BLANK-PCT-A-020822	1	5	S-13653	<1.00	<1.00	<1.00	<1.00	5	<5.00	<5.00	<5.00	<5.00
PCT std	1	6	PCT std 2	19.7	10.9	80.6	50.5	1	19.7	10.9	80.6	50.5
MQ-WATER BLANK-PCT-B-020822	1	7	S-13654	<1.00	<1.00	<1.00	<1.00	5	<5.00	<5.00	<5.00	<5.00
EMHQ-LBE-04-B-PCT-C	1	8	S-13649	5.62	1.41	14.6	18.3	5	28.1	7.05	73.0	91.5
ARM-1-STD-GLASS-PCT-A-020822	1	9	S-13650	3.61	2.97	8.02	13.2	5	18.1	14.9	40.1	66.0
ARM-1-STD-GLASS-PCT-B-020822	1	10	S-13651	3.48	2.91	7.72	12.9	5	17.4	14.6	38.6	64.5
PCT std	1	11	PCT std 3	18.9	10.1	75.9	48.7	1	18.9	10.1	75.9	48.7
PCT std	2	1	PCT std 1	20.6	10.9	85.4	51.6	1	20.6	10.9	85.4	51.6
EMHQ-LBE-04-B-PCT-B	2	2	S-13648	7.05	1.77	17.8	19.4	5	35.3	8.85	89.0	97.0
ARM-1-STD-GLASS-PCT-C-020822	2	3	S-13652	3.77	2.65	8.23	13.5	5	18.9	13.3	41.2	67.5
EMHQ-LBE-04-B-PCT-A	2	4	S-13647	6.69	1.71	17.0	18.9	5	33.5	8.55	85.0	94.5
MQ-WATER BLANK-PCT-A-020822	2	5	S-13653	<1.00	<1.00	<1.00	<1.00	5	<5.00	<5.00	<5.00	<5.00
PCT std	2	6	PCT std 2	19.9	10.5	81.7	50.5	1	19.9	10.5	81.7	50.5
MQ-WATER BLANK-PCT-B-020822	2	7	S-13654	<1.00	<1.00	<1.00	<1.00	5	<5.00	<5.00	<5.00	<5.00
EMHQ-LBE-04-B-PCT-C	2	8	S-13649	6.16	1.55	16.1	19.3	5	30.8	7.75	80.5	96.5
ARM-1-STD-GLASS-PCT-A-020822	2	9	S-13650	4.06	3.32	8.64	14.0	5	20.3	16.6	43.2	70.0
ARM-1-STD-GLASS-PCT-B-020822	2	10	S-13651	3.93	2.76	8.43	13.8	5	19.7	13.8	42.2	69.0
PCT std	2	11	PCT std 3	20.6	10.9	84.3	51.9	1	20.6	10.9	84.3	51.9

Table B-2. Dilution-Corrected ARM-1 Leachate Measurements (mg/L)

PNNL ID	Lab ID	B	Li	Na	Si
ARM-1-STD-GLASS-PCT-A-020822	S-13650	18.1	14.9	40.1	66.0
ARM-1-STD-GLASS-PCT-B-020822	S-13651	17.4	14.6	38.6	64.5
ARM-1-STD-GLASS-PCT-C-020822	S-13652	17.2	14.3	39.5	64.5
ARM-1-STD-GLASS-PCT-A-020822	S-13650	20.3	16.6	43.2	70.0
ARM-1-STD-GLASS-PCT-B-020822	S-13651	19.7	13.8	42.2	69.0
ARM-1-STD-GLASS-PCT-C-020822	S-13652	18.9	13.3	41.2	67.5

Ranges of Expected Test Results for ARM-1^d

Boron: 12.89 – 22.65 mg/L (17.7 ± 4.89 mg/L)

Lithium: 10.80 – 16.32 mg/L (13.6 ± 2.76 mg/L)

Sodium: 28.86 – 43.58 mg/L (36.2 ± 7.36 mg/L)

Silicon: 49.03 – 73.43 mg/L (61.2 ± 12.2 mg/L)

Values that fall outside of the reference ranges are shaded grey.

^d C.M. Jantzen, J.B. Pickett, K.G. Brown, T.B. Edwards, and D.C. Beam, "Process/Product Models for the Defense Waste Processing Facility (DWPF): Part I. Predicting Glass Durability from Composition Using a Thermodynamic Hydration Energy Reaction Model (THERMO)," Westinghouse Savannah River Company, Aiken, SC, WSRC-TR-93-672, Rev. 1, 1995.

Table B-3. Results from Samples of the Multi-Element Solution Standard Included with the PCT Leachates

Analytical Block	1	2	Reference Values (mg/L)
Mean (B (mg/L))	19.4	20.4	20.0
Mean (Li (mg/L))	10.5	10.8	10.0
Mean (Na (mg/L))	78.5	83.8	81.0
Mean (Si (mg/L))	49.7	51.3	50.0
% relative bias, B	-3.2	1.8	<10% per ASTM C1285
% relative bias, Li	4.7	7.7	
% relative bias, Na	-3.0	3.5	
% relative bias, Si	-0.6	2.7	
Standard Deviation (B (mg/L))	0.42	0.40	
Standard Deviation (Li (mg/L))	0.40	0.23	
Standard Deviation (Na (mg/L))	2.40	1.90	
Standard Deviation (Si (mg/L))	0.92	0.74	
%RSD (B)	2.1	2.0	<10% per ASTM C1285
%RSD (Li)	3.9	2.1	
%RSD (Na)	3.1	2.3	
%RSD (Si)	1.8	1.4	

Distribution:

Jake.Amoroso@srnl.doe.gov
CJ.Bannochie@srnl.doe.gov
William.Bates@srnl.doe.gov
Marion.Cofer@srnl.doe.gov
Alex.Cozzi@srnl.doe.gov
Charles.Crawford@srnl.doe.gov
Elaine_N_Diaz@orp.doe.gov
William.C.Eaton@pnnl.gov
Holly.Hall@srnl.doe.gov
Erich.Hansen@srnl.doe.gov
Connie.Herman@srnl.doe.gov
Anthony.Howe@srnl.doe.gov
Madison.Hsieh@srnl.doe.gov
Fabienne.Johnson@srnl.doe.gov
Albert_A_Kruger@orp.doe.gov
Christine.Langton@srnl.doe.gov
Brady.Lee@srnl.doe.gov
Joseph.Manna@srnl.doe.gov
Daniel.McCabe@srnl.doe.gov
Kandice.Miles@srnl.doe.gov
Gregg.Morgan@srnl.doe.gov
Eric_Nelson@orp.doe.gov
Ivan_G_Papp@orp.doe.gov
Frank.Pennebaker@srnl.doe.gov
Elaine_N_Porcaro@orp.doe.gov
William.Ramsey@srnl.doe.gov
Marissa.Reigel@srnl.doe.gov
Whitney.Riley@srnl.doe.gov
Renee.Russell@pnnl.gov
Eric.Skidmore@srnl.doe.gov
Anna.Stanfield@srnl.doe.gov
Michael.Stone@srnl.doe.gov
William.Swift@srnl.doe.gov
John.Vienna@pnnl.gov
Boyd.Wiedenman@srnl.doe.gov
Richard.Wyrwas@srnl.doe.gov
Records Administration (EDWS)