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Compositions of Simulated LAW Glasses Saturated with Sulfur at Various Temperature and Time Conditions

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EXECUTIVE SUMMARY

In this report, the Savannah River National Laboratory (SRNL) provides chemical analysis for several simulated low-activity waste (LAW) glass compositions, as well as chemical analysis of the wash solutions resulting from the preparation of these glasses. The Pacific Northwest National Laboratory (PNNL) selected and fabricated these glasses as part of a study on sulfur retention in glasses as a function of melting conditions.

Chemical analyses were performed on a representative sample of each of the study glasses to allow for comparisons with the targeted compositions and to determine the amount of sulfur retained. Three dissolution techniques, sodium peroxide fusion, lithium metaborate fusion, and potassium hydroxide fusion, were used for preparing each of the glass samples for analysis. Each of the samples was analyzed twice for each element of interest by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) or Ion Chromatography (IC). Average concentrations of each of the glass components were determined and reported.

Chemical analyses were also performed on a representative sample of each of the wash solutions resulting from the preparation of the sulfur saturated melt (SSM) versions of the study glasses. The samples were analyzed in duplicate for each element of interest by ICP-AES or IC. Average concentrations of each analyte were determined and reported.

These results will be used by PNNL in the development of improved property/composition models for LAW glass production at Hanford.

TABLE OF CONTENTS

LIST OF TABLES viii
1.0 Introduction 1
2.0 Experimental Procedure 1
2.1 Quality Assurance 1
2.2 Glasses Selected for Study 1
2.3 Glass Composition Analysis 2
2.4 Wash Solution Analysis 3
3.0 Results and Discussion 4
3.1 Review of the Glass Composition Measurements 4
3.2 Review of the Wash Solution Measurements 5
4.0 Summary 5
5.0 References 6
Appendix A Tables Supporting the Chemical Analysis of the Study Glasses A-1
Appendix B Tables Supporting the Wash Solution Chemical Composition Analysis B-1

LIST OF TABLES

Table 2-1. Glass and Wash Solution Identifiers Included in This Study 2

Table 2-2. Preparation and Measurement Methods Used in Reporting the Concentrations of Each of the Analytes of the Study Glasses 3

Table 2-3. Measurement Methods Used in Reporting the Concentrations of Each of the Analytes of the Wash Solutions 4

LIST OF ABBREVIATIONS

DOE	U.S. Department of Energy
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
HLW	High-Level Waste
KH	Potassium hydroxide fusion
LAW	Low-Activity Waste
LM	Lithium Metaborate fusion
LRM	Low-level Reference Material
ORP	Office of River Protection
PF	Sodium Peroxide Fusion
PNNL	Pacific Northwest National Laboratory
SRNL	Savannah River National Laboratory
SSM	Sulfur Saturated Melt
TTQAP	Task Technical and Quality Assurance Plan
wt %	weight percent
WTP	Hanford Tank Waste Treatment and Immobilization Plant

1.0 Introduction

The U.S. Department of Energy (DOE) Office of River Protection (ORP) has requested that the Savannah River National Laboratory (SRNL) provide expert evaluation and experimental work in support of the River Protection Project vitrification technology development program. DOE is 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 low-activity waste (LAW) fraction will be partitioned from the high-level waste (HLW). Both the LAW and HLW will then be vitrified into borosilicate glass using Joule-heated ceramic melters.

Efforts are being made to increase the loading of Hanford tank wastes in the glass while conforming to processing requirements and product quality regulations. DOE-ORP has requested that SRNL support the advancement of glass formulations and process control strategies in key technical areas, as defined in the Task Technical and Quality Assurance Plan (TTQAP).[1] Two of these areas are enhancing waste glass property/composition models and broadening the compositional regions over which those models are applicable.

In this report, SRNL provides chemical analysis for several simulated LAW glass compositions, as well as chemical analysis of the wash solutions resulting from the preparation of these glasses. The Pacific Northwest National Laboratory (PNNL) selected and fabricated these glasses as part of a study on sulfur retention. The resulting data will be used in the development of improved property/composition models for LAW glass production at Hanford.

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 Manual E7, Procedure 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2. Laboratory data for this study were recorded in the SRNL Electronic Laboratory Notebook system, experiment C3489-00079-24. The glasses provided by PNNL were fabricated following Test Instruction EWG-TI-0062.

2.2 Glasses Selected for Study

The glass compositions in this study were selected and fabricated at PNNL. Identifiers for each of the 8 glasses and their corresponding wash solutions are listed in Table 2-1. The SSM suffix indicates that these glasses were fabricated as sulfur saturated melts. The suffix -S indicates the solid glass samples, and the suffix -W indicates the wash solutions. In the sections that follow, the methods used for measuring chemical compositions of the glasses and wash solutions are described, and brief reviews of the resulting data are provided. Detailed data from these analyses are included in the appendices.

Table 2-1. Glass and Wash Solution Identifiers Included in This Study

Glass ID		Wash Solution ID
LAWA161-950-3hrs-SSM-S		LAWA161-950-3hrs-SSM-W
LAWA161-950-3hrs-2-SSM-S		LAWA161-950-3hrs-2-SSM-W
LAWA161-950-3hrs-2xNa2SO4-SSM-S		LAWA161-950-3hrs-2xNa2SO4-SSM-W
LAWA161-950-6hrs-SSM-S		LAWA161-950-6hrs-SSM-W
LAWA161-1150-1hr-SSM-S		LAWA161-1150-1hr-SSM-W
LAWA161-950-24hrs-SSM-S		LAWA161-950-24hrs-SSM-W
LAWA161-950-12hrs-2xNa2SO4-SSM-S		LAWA161-950-12hrs-2xNa2SO4-SSM-W
LAWA161-950-24hrs-2xNa2SO4-SSM-S		LAWA161-950-24hrs-2xNa2SO4-SSM-W

2.3 Glass Composition Analysis

Chemical analyses were performed on a representative sample of each of the glasses listed in Table 2-1 to allow for comparisons with the targeted compositions and determine the amount of sulfur retained as a function of melting conditions. Three dissolution techniques, sodium peroxide fusion (PF),[2] lithium metaborate fusion (LM),[3] and potassium hydroxide fusion (KH),[4] were used for preparing each of the glass samples for analysis.

Each of the prepared samples was analyzed twice for each element of interest by Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES)[5] or ion chromatography (IC).[6] The instruments were then recalibrated and each sample was again measured in duplicate, for a total of four measurements for each analyte for each glass. A sample of the low-level reference material (LRM)[7] glass was included as a check of the performance of the ICP-AES and IC instruments. The LRM composition reported as the "Consensus Average" is used as the reference composition of this glass for the purposes of this study.[7] 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 Concentrations of Each of the Analytes of the Study Glasses

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

2.4 Wash Solution Analysis

Chemical analyses were performed on a representative sample of each of the wash solutions from the SSM glasses listed in Table 2-1. These wash solutions were prepared at PNNL and provided to SRNL for analysis. Each of the samples was analyzed in duplicate for each element of interest by ICP-AES[5] and IC.[6] The measurement methods used for each of the reported wash solution components are listed in Table 2-3.

Table 2-3. Measurement Methods Used in Reporting the Concentrations of Each of the Analytes of the Wash Solutions

Analyte	Measurement Method
Al	ICP-AES
B	ICP-AES
Ca	ICP-AES
Cl^-	IC
Cr	ICP-AES
F^-	IC
Fe	ICP-AES
K	ICP-AES
Li	ICP-AES
Mg	ICP-AES
Mn	ICP-AES
Na	ICP-AES
Ni	ICP-AES
P	ICP-AES
S	ICP-AES
Si	ICP-AES
V	ICP-AES
Zn	ICP-AES
Zr	ICP-AES

3.0 Results and Discussion

3.1 Review of the Glass Composition Measurements

Table A-1 and Table A-2 in Appendix A provide the elemental concentration measurements in weight percent (wt %) for the study glasses measured by ICP-AES. Table A-3 in Appendix A provides the elemental concentration measurements in wt % for the study glasses measured by IC. The quadruplicate measurements for each prepared glass sample are shown. Elemental measurements for the LRM glass are also included in these tables. The LRM measurements are within the acceptability limits utilized by SRNL.[5] The data in these tables are provided so that the values are readily available should they be of interest for future reviews.

The quadruplicate measurements for each analyte were converted to oxides using the appropriate gravimetric factors and averaged to determine a representative chemical composition for each glass. A sum of oxides was also computed for each glass using the averaged, measured values. An elemental concentration measurement that was reported to be below the detection limit of the analytical process used was set to the detection limit as the oxide concentration was determined for calculating the sum of oxides. Those oxides with one or more concentration measurements that were below the associated detection limit will be denoted with a less than symbol (<) as the measured compositions are reported.

The measured sums of oxides for the study glasses fall within the interval of 97.9 to 99.4 wt %, indicating acceptable recovery of the glass components. Table A-4 in Appendix A provides a summary of the average compositions as well as the targeted compositions and some relative differences. Entries in Table A-4 show the relative differences between the measured values and the targeted values for those oxides with measured values that were above the limits of detection.

3.2 Review of the Wash Solution Measurements

Table B-1 and Table B-2 in Appendix B provide the elemental concentration measurements in mg/L for the wash solutions as measured by ICP-AES. Table B-3 in Appendix B provides the anion concentration measurements in mg/L for the wash solutions as measured by IC. The data in these tables are provided so that the values are readily available should they be of interest for future reviews.

The duplicate measurements for each analyte for each wash solution were averaged to determine a representative chemical composition for each solution. Table B-4 in Appendix B provides a summary of the average measured compositions of the wash solutions.

4.0 Summary

In this report, SRNL provides chemical analysis for several simulated LAW glass compositions, as well as chemical analysis of the wash solutions resulting from the preparation of these glasses. PNNL selected and fabricated these glasses as part of a study on sulfur retention in glasses as a function of melting temperature.

Chemical analyses were performed on a representative sample of each of the study glasses to allow for comparisons with the targeted compositions. Three dissolution techniques, sodium peroxide fusion, lithium metaborate fusion, and potassium hydroxide fusion, were used for preparing each of the glass samples for analysis. Each of the samples was analyzed four times for each element of interest by ICP-AES or IC. A glass standard was also measured as a check of the performance of the analytical instruments. Average concentrations of each of the glass components were determined and reported.

Chemical analyses were also performed on a representative sample of each of the wash solutions resulting from the preparation of the SSM versions of the study glasses. The samples were analyzed in duplicate for each element of interest by ICP-AES or IC. Average concentrations of each analyte were determined and reported.

These results will be used by PNNL in the development of improved property/composition models for LAW glass production at Hanford.

5.0 References

1. Fox, K. M., "Task Technical and Quality Assurance Plan for Hanford Waste Glass Development and Characterization," *U.S. Department of Energy Report SRNL-RP-2013-00692, Revision 1,* Savannah River National Laboratory, Aiken, SC (2016).

2. Best, D. R., "Dissolution of Glass, Sludge, and Slurry Samples Using Na_2O_2/NaOH/HCl," *Manual L29, ITS-0040, Revision 2,* Savannah River National Laboratory, Aiken, SC (2013).

3. Best, D. R., "Lithium Metaborate Fusion Preparation," *Manual L29, ITS-0071, Revision 3,* Savannah River National Laboratory, Aiken, SC (2015).

4. "Sample Dissolution Using Potassium Hydroxide Fusion," *Manual L29, ITS-0035, Revision 3,* Savannah River National Laboratory, Aiken, SC (2015).

5. Best, D. R., "Inductively Coupled Plasma-Atomic Emission Spectrometer, Agilent 730 ES," *Manual L29, Procedure ITS-0079, Revision 5,* Savannah River National Laboratory, Aiken, SC (2014).

6. Best, D. R., "Anion Analysis Using the Dionex DX-500 and ICS-5000 Ion Chromatograph," *Manual L29, Procedure ITS-0027, Revision 3,* Savannah River National Laboratory, Aiken, SC (2011).

7. Ebert, W. L. and S. F. Wolfe, "Round-robin Testing of a Reference Glass for Low-Activity Waste Forms," *U.S. Department of Energy Report ANL-99/22,* Argonne National Laboratory, Argonne, IL (1999).

Appendix A Tables Supporting the Chemical Analysis of the Study Glasses

Table A-1. ICP-AES Measurement Data (elemental wt %) for the Study Glasses (Part 1 of 2)

Glass ID	Lab ID	Al	B	Ca	Cr	Fe	K	Li	Mg	Mn
LRM	LRM	4.80	2.27	0.219	0.122	0.966	1.01	<0.100	<0.100	<0.100
LAWA161-950-3hrs-SSM-S	S-8188	5.03	3.92	5.76	<0.100	0.685	0.295	<0.100	0.560	<0.100
LAWA161-950-3hrs-SSM-S	S-8188	5.01	3.82	5.76	<0.100	0.665	0.284	<0.100	0.568	<0.100
LAWA161-950-3hrs-2-SSM-S	S-8189	5.08	4.05	5.90	<0.100	0.695	0.295	<0.100	0.547	<0.100
LAWA161-950-3hrs-2-SSM-S	S-8189	5.02	4.07	5.84	<0.100	0.699	0.291	<0.100	0.556	<0.100
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	5.01	4.06	5.89	<0.100	0.718	0.243	<0.100	0.559	<0.100
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	5.05	4.10	5.90	<0.100	0.723	0.255	<0.100	0.553	<0.100
LAWA161-950-6hrs-SSM-S	S-8191	4.99	4.10	5.92	<0.100	0.717	0.302	<0.100	0.553	<0.100
LAWA161-950-6hrs-SSM-S	S-8191	5.06	3.94	5.73	<0.100	0.691	0.290	<0.100	0.576	<0.100
LAWA161-1150-1hr-SSM-S	S-8192	4.96	4.05	5.84	<0.100	0.710	0.292	<0.100	0.568	<0.100
LAWA161-1150-1hr-SSM-S	S-8192	4.96	3.97	5.81	<0.100	0.697	0.279	<0.100	0.576	<0.100
LAWA161-950-24hrs-SSM-S	S-8193	5.13	3.94	5.74	<0.100	0.693	0.278	<0.100	0.580	<0.100
LAWA161-950-24hrs-SSM-S	S-8193	5.11	3.97	5.81	<0.100	0.698	0.285	<0.100	0.571	<0.100
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	5.08	3.81	5.73	<0.100	0.677	0.254	<0.100	0.573	<0.100
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	5.04	3.90	5.69	<0.100	0.685	0.253	<0.100	0.566	<0.100
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	5.00	3.86	5.80	<0.100	0.680	0.243	<0.100	0.570	<0.100
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	5.06	4.05	5.85	<0.100	0.710	0.259	<0.100	0.559	<0.100
LRM	LRM	4.83	2.30	0.196	0.120	0.959	0.998	<0.100	<0.100	<0.100
LAWA161-950-3hrs-SSM-S	S-8188	4.94	3.97	5.74	<0.100	0.674	0.280	<0.100	0.560	<0.100
LAWA161-950-3hrs-SSM-S	S-8188	5.24	3.90	5.81	<0.100	0.658	0.271	<0.100	0.562	<0.100
LAWA161-950-3hrs-2-SSM-S	S-8189	5.21	3.90	5.74	<0.100	0.660	0.267	<0.100	0.543	<0.100
LAWA161-950-3hrs-2-SSM-S	S-8189	5.25	3.81	5.67	<0.100	0.645	0.251	<0.100	0.543	<0.100
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	5.20	3.72	5.58	<0.100	0.654	0.219	<0.100	0.540	<0.100
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	5.13	3.61	5.53	<0.100	0.637	0.201	<0.100	0.531	<0.100
LAWA161-950-6hrs-SSM-S	S-8191	5.21	4.15	5.99	<0.100	0.700	0.279	<0.100	0.551	<0.100
LAWA161-950-6hrs-SSM-S	S-8191	5.09	3.97	5.83	<0.100	0.677	0.276	<0.100	0.561	<0.100
LAWA161-1150-1hr-SSM-S	S-8192	5.10	4.02	5.84	<0.100	0.685	0.256	<0.100	0.553	<0.100
LAWA161-1150-1hr-SSM-S	S-8192	5.02	4.07	5.93	<0.100	0.680	0.256	<0.100	0.542	<0.100
LAWA161-950-24hrs-SSM-S	S-8193	5.07	3.94	5.81	<0.100	0.670	0.261	<0.100	0.553	<0.100
LAWA161-950-24hrs-SSM-S	S-8193	4.97	4.01	5.83	<0.100	0.678	0.257	<0.100	0.552	<0.100
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	5.27	3.98	5.82	<0.100	0.672	0.233	<0.100	0.545	<0.100
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	5.20	3.89	5.75	<0.100	0.652	0.229	<0.100	0.546	<0.100
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	5.31	3.80	5.65	<0.100	0.651	0.214	<0.100	0.550	<0.100
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	5.24	3.85	5.77	<0.100	0.654	0.223	<0.100	0.540	<0.100

Table A-2. ICP-AES Measurement Data (elemental wt %) for the Study Glasses (Part 2 of 2)

Glass ID	Lab ID	Na	Ni	P	S	Si	V	Zn	Zr
LRM	LRM	15.2	0.135	0.191	0.0900	26.9	<0.100	<0.100	0.630
LAWA161-950-3hrs-SSM-S	S-8188	13.8	<0.100	<0.100	0.430	18.5	0.508	2.22	2.12
LAWA161-950-3hrs-SSM-S	S-8188	13.9	<0.100	<0.100	0.435	18.6	0.509	2.16	2.12
LAWA161-950-3hrs-2-SSM-S	S-8189	14.7	<0.100	<0.100	0.518	18.5	0.498	2.28	2.13
LAWA161-950-3hrs-2-SSM-S	S-8189	14.6	<0.100	<0.100	0.512	18.5	0.503	2.26	2.12
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	14.9	<0.100	<0.100	0.585	17.9	0.491	2.32	2.12
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	14.6	<0.100	<0.100	0.554	18.0	0.487	2.30	2.14
LAWA161-950-6hrs-SSM-S	S-8191	14.9	<0.100	<0.100	0.460	17.5	0.499	2.29	2.11
LAWA161-950-6hrs-SSM-S	S-8191	14.4	<0.100	<0.100	0.462	17.7	0.499	2.25	2.14
LAWA161-1150-1hr-SSM-S	S-8192	14.3	<0.100	<0.100	0.464	18.2	0.504	2.28	2.11
LAWA161-1150-1hr-SSM-S	S-8192	14.3	<0.100	<0.100	0.465	18.4	0.524	2.25	2.10
LAWA161-950-24hrs-SSM-S	S-8193	14.6	<0.100	<0.100	0.474	18.4	0.517	2.24	2.17
LAWA161-950-24hrs-SSM-S	S-8193	14.1	<0.100	<0.100	0.453	18.7	0.520	2.25	2.16
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	14.4	<0.100	<0.100	0.597	17.8	0.509	2.21	2.15
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	14.5	<0.100	<0.100	0.572	17.7	0.503	2.24	2.12
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	14.8	<0.100	<0.100	0.567	18.1	0.503	2.22	2.13
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	14.7	<0.100	<0.100	0.569	17.8	0.505	2.33	2.15
LRM	LRM	14.4	0.133	0.180	0.0856	26.5	<0.100	<0.100	0.610
LAWA161-950-3hrs-SSM-S	S-8188	14.4	<0.100	<0.100	0.448	17.0	0.472	2.19	2.07
LAWA161-950-3hrs-SSM-S	S-8188	14.4	<0.100	<0.100	0.456	17.0	0.502	2.15	2.19
LAWA161-950-3hrs-2-SSM-S	S-8189	14.5	<0.100	<0.100	0.507	17.6	0.494	2.20	2.17
LAWA161-950-3hrs-2-SSM-S	S-8189	14.5	<0.100	<0.100	0.522	16.2	0.497	2.13	2.18
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	14.5	<0.100	<0.100	0.602	17.1	0.483	2.14	2.19
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	14.4	<0.100	<0.100	0.592	15.7	0.481	2.06	2.16
LAWA161-950-6hrs-SSM-S	S-8191	14.2	<0.100	<0.100	0.517	16.7	0.495	2.28	2.18
LAWA161-950-6hrs-SSM-S	S-8191	14.7	<0.100	<0.100	0.490	16.6	0.484	2.21	2.13
LAWA161-1150-1hr-SSM-S	S-8192	14.6	<0.100	<0.100	0.503	17.6	0.483	2.19	2.14
LAWA161-1150-1hr-SSM-S	S-8192	14.6	<0.100	<0.100	0.479	16.8	0.486	2.23	2.10
LAWA161-950-24hrs-SSM-S	S-8193	14.4	<0.100	<0.100	0.506	17.5	0.504	2.19	2.12
LAWA161-950-24hrs-SSM-S	S-8193	14.5	<0.100	<0.100	0.496	16.8	0.508	2.19	2.08
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	14.6	<0.100	<0.100	0.610	17.0	0.495	2.24	2.21
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	14.5	<0.100	<0.100	0.597	16.7	0.491	2.18	2.17
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	14.5	<0.100	<0.100	0.581	17.3	0.497	2.14	2.22
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	14.2	<0.100	<0.100	0.570	17.0	0.501	2.16	2.19

Table A-3. IC Measurement Data (elemental wt %) for the Study Glasses

Glass ID	Lab ID	Cl	F
LRM	LRM	<0.025	0.868
LAWA161-950-3hrs-SSM-S	S-8188	0.491	<0.025
LAWA161-950-3hrs-SSM-S	S-8188	0.490	<0.025
LAWA161-950-3hrs-2-SSM-S	S-8189	0.445	<0.025
LAWA161-950-3hrs-2-SSM-S	S-8189	0.433	<0.025
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	0.302	<0.025
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	0.313	<0.025
LAWA161-950-6hrs-SSM-S	S-8191	0.440	<0.025
LAWA161-950-6hrs-SSM-S	S-8191	0.425	<0.025
LAWA161-1150-1hr-SSM-S	S-8192	0.374	<0.025
LAWA161-1150-1hr-SSM-S	S-8192	0.369	<0.025
LAWA161-950-24hrs-SSM-S	S-8193	0.270	<0.025
LAWA161-950-24hrs-SSM-S	S-8193	0.253	<0.025
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	0.256	<0.025
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	0.264	<0.025
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	0.236	<0.025
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	0.236	<0.025
LRM	LRM	<0.025	0.871
LAWA161-950-3hrs-SSM-S	S-8188	0.505	<0.025
LAWA161-950-3hrs-SSM-S	S-8188	0.489	<0.025
LAWA161-950-3hrs-2-SSM-S	S-8189	0.453	<0.025
LAWA161-950-3hrs-2-SSM-S	S-8189	0.438	<0.025
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	0.318	<0.025
LAWA161-950-3hrs-2xNa2SO4-SSM-S	S-8190	0.301	<0.025
LAWA161-950-6hrs-SSM-S	S-8191	0.448	<0.025
LAWA161-950-6hrs-SSM-S	S-8191	0.425	<0.025
LAWA161-1150-1hr-SSM-S	S-8192	0.384	<0.025
LAWA161-1150-1hr-SSM-S	S-8192	0.376	<0.025
LAWA161-950-24hrs-SSM-S	S-8193	0.250	<0.025
LAWA161-950-24hrs-SSM-S	S-8193	0.273	<0.025
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	0.255	<0.025
LAWA161-950-12hrs-2xNa2SO4-SSM-S	S-8194	0.252	<0.025
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	0.228	<0.025
LAWA161-950-24hrs-2xNa2SO4-SSM-S	S-8195	0.225	<0.025

Table A-4. Summary of Measured Composition Data (wt % oxide) for the Study Glasses

Glass ID	View	Al_2O_3	B_2O_3	CaO	Cl	Cr_2O_3	F	Fe_2O_3	K_2O	Li_2O	MgO	MnO	Na_2O	NiO	P_2O_5	SO_3	SiO_2	V_2O_5	ZnO	ZrO_2	Sum
LRM	Targeted	9.51	7.85	0.54	-	0.19	0.86	1.38	1.48	0.11	0.10	-	20.03	0.19	0.54	0.30	54.2	-	-	0.93	98.2
	Measured	9.10	7.36	0.291	<0.025	0.177	0.869	1.38	1.21	<0.215	<0.166	<0.129	20.0	0.171	0.426	0.219	57.2	<0.179	<0.124	0.838	100.1
	Percent Error	-4%	-6%	-46%	-	-7%	1%	0%	-18%	-	-	-	0%	-10%	-21%	-27%	6%	-	-	-10%	-
LAWA161 (all heat treatments)	Targeted	10.16	13.67	7.99	1.17	0.02	0.00	1.00	0.44	0.00	1.00	0.00	20.66	0.00	0.00	0.19	36.58	1.00	2.99	2.99	99.9
LAWA161-950-3hrs-SSM-S	Measured	9.55	12.6	8.07	0.494	<0.146	<0.025	0.959	0.340	<0.215	0.933	<0.129	19.0	<0.127	<0.229	1.10	38.0	0.889	2.71	2.87	98.4
	Percent Error	-6%	-8%	1%	-58%	-	-	-4%	-23%	-	-7%	-	-8%	-	-	479%	4%	-11%	-9%	-4%	-
LAWA161-950-3hrs-2-SSM-S	Measured	9.71	12.7	8.10	0.443	<0.146	<0.025	0.964	0.333	<0.215	0.908	<0.129	19.6	<0.127	<0.229	1.29	37.9	0.889	2.76	2.90	99.4
	Percent Error	-4%	-7%	1%	-62%	-	-	-4%	-24%	-	-9%	-	-5%	-	-	579%	4%	-11%	-8%	-3%	-
LAWA161-950-3hrs-2xNa2SO4-SSM-S	Measured	9.63	12.5	8.01	0.308	<0.146	<0.025	0.976	0.276	<0.215	0.905	<0.129	19.7	<0.127	<0.229	1.46	36.7	0.867	2.74	2.91	97.9
	Percent Error	-5%	-9%	0%	-74%	-	-	-2%	-37%	-	-10%	-	-5%	-	-	668%	0%	-13%	-8%	-3%	-
LAWA161-950-6hrs-SSM-S	Measured	9.61	13.0	8.21	0.435	<0.146	<0.025	0.996	0.346	<0.215	0.929	<0.129	19.6	<0.127	<0.229	1.20	36.6	0.883	2.81	2.89	98.4
	Percent Error	-5%	-5%	3%	-63%	-	-	0%	-21%	-	-7%	-	-5%	-	-	532%	0%	-12%	-6%	-3%	-
LAWA161-1150-1hr-SSM-S	Measured	9.47	13.0	8.19	0.376	<0.146	<0.025	0.991	0.326	<0.215	0.928	<0.129	19.5	<0.127	<0.229	1.19	38.0	0.891	2.79	2.85	99.4
	Percent Error	-7%	-5%	3%	-68%	-	-	-1%	-26%	-	-7%	-	-6%	-	-	526%	4%	-11%	-7%	-5%	-
LAWA161-950-24hrs-SSM-S	Measured	9.58	12.8	8.11	0.261	<0.146	<0.025	0.979	0.326	<0.215	0.936	<0.129	19.4	<0.127	<0.229	1.20	38.2	0.915	2.76	2.88	99.2
	Percent Error	-6%	-6%	2%	-78%	-	-	-2%	-26%	-	-6%	-	-6%	-	-	532%	4%	-9%	-8%	-4%	-
LAWA161-950-12hrs-2xNa2SO4-SSM-S	Measured	9.73	12.5	8.04	0.257	<0.146	<0.025	0.960	0.292	<0.215	0.925	<0.129	19.6	<0.127	<0.229	1.48	37.0	0.892	2.76	2.92	98.2
	Percent Error	-4%	-9%	1%	-78%	-	-	-4%	-34%	-	-8%	-	-5%	-	-	679%	1%	-11%	-8%	-2%	-
LAWA161-950-24hrs-2xNa2SO4-SSM-S	Measured	9.73	12.5	8.07	0.231	<0.146	<0.025	0.964	0.283	<0.215	0.920	<0.129	19.6	<0.127	<0.229	1.43	37.6	0.895	2.76	2.94	98.8
	Percent Error	-4%	-9%	1%	-80%	-	-	-4%	-36%	-	-8%	-	-5%	-	-	653%	3%	-11%	-8%	-2%	-

Appendix B Tables Supporting the Wash Solution Chemical Composition Analysis

Table B-1. ICP-AES Measurement Data (mg/L) for the Study Glass Wash Solutions (Part 1 of 2)

Sample ID	Lab ID	Al	B	Ca	Cr	Fe	K	Li	Mg	Mn	Na
Custom Standard	-	3.65	20.5	<1.00	<1.00	4.09	9.99	9.98	<1.00	<1.00	77.9
High-Purity Standards SM-744-063	-	51.8	1.38	<1.00	<1.00	50.1	<1.00	<1.00	<1.00	20.4	146
LAWA161-950-3hrs-SSM-W	S-8180	<1.00	11.1	3.99	<1.00	<1.00	10.8	<1.00	<1.00	<1.00	395
LAWA161-950-3hrs-SSM-W	S-8180	<1.00	11.9	4.03	<1.00	<1.00	10.9	<1.00	<1.00	<1.00	391
LAWA161-950-3hrs-2-SSM-W	S-8181	<1.00	12.2	5.43	<1.00	<1.00	10.9	<1.00	<1.00	<1.00	427
LAWA161-950-3hrs-2-SSM-W	S-8181	<1.00	12.3	5.45	<1.00	<1.00	10.9	<1.00	<1.00	<1.00	419
LAWA161-950-3hrs-2xNa2SO4-SSM-W	S-8182	<1.00	27.6	24.8	<1.00	<1.00	39.3	<1.00	<1.00	<1.00	1530
LAWA161-950-3hrs-2xNa2SO4-SSM-W	S-8182	<1.00	27.7	24.9	<1.00	<1.00	38.5	<1.00	<1.00	<1.00	1560
LAWA161-950-6hrs-SSM-W	S-8183	<1.00	8.85	2.19	<1.00	<1.00	3.36	<1.00	<1.00	<1.00	189
LAWA161-950-6hrs-SSM-W	S-8183	<1.00	8.86	2.19	<1.00	<1.00	3.48	<1.00	<1.00	<1.00	200
LAWA161-1150-1hr-SSM-W	S-8184	<1.00	18.4	9.26	1.24	<1.00	16.9	<1.00	<1.00	<1.00	579
LAWA161-1150-1hr-SSM-W	S-8184	<1.00	17.9	9.14	1.23	<1.00	16.9	<1.00	<1.00	<1.00	575
LAWA161-950-24hrs-SSM-W	S-8185	<1.00	6.90	1.22	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	54.7
LAWA161-950-24hrs-SSM-W	S-8185	<1.00	6.82	1.22	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	53.8
LAWA161-950-12hrs-2xNa2SO4-SSM-W	S-8186	<1.00	12.8	8.41	<1.00	<1.00	13.1	<1.00	<1.00	<1.00	633
LAWA161-950-12hrs-2xNa2SO4-SSM-W	S-8186	<1.00	13.4	8.50	<1.00	<1.00	13.4	<1.00	<1.00	<1.00	648
LAWA161-950-24hrs-2xNa2SO4-SSM-W	S-8187	<1.00	13.1	9.08	<1.00	<1.00	11.6	<1.00	<1.00	<1.00	579
LAWA161-950-24hrs-2xNa2SO4-SSM-W	S-8187	<1.00	13.1	9.17	<1.00	<1.00	11.7	<1.00	<1.00	<1.00	592

Table B-2. ICP-AES Measurement Data (mg/L) for the Study Glass Wash Solutions (Part 2 of 2)

Sample ID	Lab ID	Ni	P	S	Si	V	Zn	Zr
Custom Standard	-	<1.00	<1.00	<1.00	45.3	<1.00	<1.00	<1.00
High-Purity Standards SM-744-063	-	10.0	<1.00	10.3	<1.00	<1.00	<1.00	<1.00
LAWA161-950-3hrs-SSM-W	S-8180	<1.00	<1.00	225	5.15	2.94	<1.00	<1.00
LAWA161-950-3hrs-SSM-W	S-8180	<1.00	<1.00	239	5.11	3.21	<1.00	<1.00
LAWA161-950-3hrs-2-SSM-W	S-8181	<1.00	<1.00	259	6.44	3.38	<1.00	<1.00
LAWA161-950-3hrs-2-SSM-W	S-8181	<1.00	<1.00	276	6.29	3.41	<1.00	<1.00
LAWA161-950-3hrs-2xNa2SO4-SSM-W	S-8182	<1.00	<1.00	1030	6.14	9.89	<1.00	<1.00
LAWA161-950-3hrs-2xNa2SO4-SSM-W	S-8182	<1.00	<1.00	1050	5.92	10.0	<1.00	<1.00
LAWA161-950-6hrs-SSM-W	S-8183	<1.00	<1.00	97.3	5.45	1.97	<1.00	<1.00
LAWA161-950-6hrs-SSM-W	S-8183	<1.00	<1.00	98.3	5.37	1.89	<1.00	<1.00
LAWA161-1150-1hr-SSM-W	S-8184	<1.00	<1.00	366	4.94	4.15	<1.00	<1.00
LAWA161-1150-1hr-SSM-W	S-8184	<1.00	<1.00	413	4.95	4.17	<1.00	<1.00
LAWA161-950-24hrs-SSM-W	S-8185	<1.00	<1.00	10.7	5.32	1.10	<1.00	<1.00
LAWA161-950-24hrs-SSM-W	S-8185	<1.00	<1.00	10.4	5.38	1.05	<1.00	<1.00
LAWA161-950-12hrs-2xNa2SO4-SSM-W	S-8186	<1.00	<1.00	448	4.01	4.31	<1.00	<1.00
LAWA161-950-12hrs-2xNa2SO4-SSM-W	S-8186	<1.00	<1.00	450	3.70	4.42	<1.00	<1.00
LAWA161-950-24hrs-2xNa2SO4-SSM-W	S-8187	<1.00	<1.00	410	5.46	3.96	<1.00	<1.00
LAWA161-950-24hrs-2xNa2SO4-SSM-W	S-8187	<1.00	<1.00	418	5.45	3.98	<1.00	<1.00

Table B-3. IC Measurement Data (mg/L) for the Study Glass Wash Solutions

Sample ID	Lab ID	Cl	F
Check Standard-1 PPM	-	0.924	0.969
Check Standard-5 PPM	-	4.86	4.98
LAWA161-950-3hrs-SSM-W	S-8180	76.4	<1.00
LAWA161-950-3hrs-SSM-W	S-8180	76.5	<1.00
LAWA161-950-3hrs-2-SSM-W	S-8181	66.8	<1.00
LAWA161-950-3hrs-2-SSM-W	S-8181	66.8	<1.00
LAWA161-950-3hrs-2xNa2SO4-SSM-W	S-8182	192	<1.00
LAWA161-950-3hrs-2xNa2SO4-SSM-W	S-8182	193	<1.00
LAWA161-950-6hrs-SSM-W	S-8183	19.0	<1.00
LAWA161-950-6hrs-SSM-W	S-8183	18.9	<1.00
Check Standard-1 PPM	-	0.973	0.965
Check Standard-5 PPM	-	4.82	4.94
LAWA161-1150-1hr-SSM-W	S-8184	62.9	<1.00
LAWA161-1150-1hr-SSM-W	S-8184	63.0	<1.00
LAWA161-950-24hrs-SSM-W	S-8185	2.04	<1.00
LAWA161-950-24hrs-SSM-W	S-8185	1.90	<1.00
LAWA161-950-12hrs-2xNa2SO4-SSM-W	S-8186	41.3	<1.00
LAWA161-950-12hrs-2xNa2SO4-SSM-W	S-8186	39.8	<1.00
LAWA161-950-24hrs-2xNa2SO4-SSM-W	S-8187	24.1	<1.00
LAWA161-950-24hrs-2xNa2SO4-SSM-W	S-8187	26.1	<1.00
Check Standard-1 PPM	-	0.934	0.974
Check Standard-5 PPM	-	4.97	4.98

Table B-4. Summary of Measured Composition Data (mg/L) for the Study Glass Wash Solutions

Sample ID	Al	B	Ca	Cl	Cr	F	Fe	K	Li	Mg	Mn	Na	Ni	P	S	Si	V	Zn	Zr
LAWA161-950-3hrs-SSM-W	<1.00	11.5	4.01	76.4	<1.00	<1.00	<1.00	10.9	<1.00	<1.00	<1.00	393	<1.00	<1.00	232	5.13	3.07	<1.00	<1.00
LAWA161-950-3hrs-2-SSM-W	<1.00	12.3	5.44	66.8	<1.00	<1.00	<1.00	10.9	<1.00	<1.00	<1.00	423	<1.00	<1.00	267	6.37	3.40	<1.00	<1.00
LAWA161-950-3hrs-2xNa2SO4-SSM-W	<1.00	27.7	24.9	192	<1.00	<1.00	<1.00	38.9	<1.00	<1.00	<1.00	1550	<1.00	<1.00	1040	6.03	9.95	<1.00	<1.00
LAWA161-950-6hrs-SSM-W	<1.00	8.85	2.19	18.9	<1.00	<1.00	<1.00	3.42	<1.00	<1.00	<1.00	195	<1.00	<1.00	97.8	5.41	1.93	<1.00	<1.00
LAWA161-1150-1hr-SSM-W	<1.00	18.2	9.20	63.0	1.24	<1.00	<1.00	16.9	<1.00	<1.00	<1.00	577	<1.00	<1.00	390	4.94	4.16	<1.00	<1.00
LAWA161-950-24hrs-SSM-W	<1.00	6.86	1.22	1.97	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	<1.00	54.2	<1.00	<1.00	10.6	5.35	1.07	<1.00	<1.00
LAWA161-950-12hrs-2xNa2SO4-SSM-W	<1.00	13.1	8.45	40.5	<1.00	<1.00	<1.00	13.3	<1.00	<1.00	<1.00	641	<1.00	<1.00	449	3.86	4.36	<1.00	<1.00
LAWA161-950-24hrs-2xNa2SO4-SSM-W	<1.00	13.1	9.13	25.1	<1.00	<1.00	<1.00	11.6	<1.00	<1.00	<1.00	586	<1.00	<1.00	414	5.45	3.97	<1.00	<1.00

Distribution:

J. W. Amoroso, 999-W
T. B. Brown, 773-A
M. E. Caldwell, 999-W
A. D. Cozzi, 999-W
C. L. Crawford, 773-42A
D. E. Dooley, 773-A
W. C. Eaton, PNNL
T. B. Edwards, 999-W
A. P. Fellinger, 773-42A
S. D. Fink, 773-A
K. M. Fox, 999-W
C. C. Herman, 773-A
A. M. Howe, 999-W
C. M. Jantzen, 773-A
T. Jin, PNNL
F. C. Johnson, 999-W
D. S. Kim, PNNL
A. A. Kruger, DOE-ORP
C. E. Lonergan, PNNL
D. J. McCabe, 773-42A
D. L. McClane, 999-W
G. A. Morgan, 999-W
F. M. Pennebaker, 773-42A
A. A. Ramsey, 999-W
W. G. Ramsey, 999-W
W. T. Riley, 999-1W
R. L. Russell, PNNL
M. J. Schweiger, PNNL
C. H. Skidmore, PNNL
G. N. Smoland, 999-1W
C. L. Trivelpiece, 999-W
J. D. Vienna, PNNL
B. J. Wiedenman, 773-42A
W. R. Wilmarth, 773-A
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