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06/09/2015 6/22/15 JRR

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- ☒ Report JRR 6/22/15
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B. Document Number ORP-59017 Revision 0

C. Title
Final Report: LAW Glass Formulation to Support AP-101 Actual Waste Testing, VSL-03R3470-2, Rev. 0

D. Proposed Internet Address

E. Required Information (MANDATORY)

1. Is document potentially Classified? (●) No ( ) Yes

noyes, delmar Approved via IDMS data file.
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ADC Required (Print and Sign) ( ) No ( ) Yes Classified

2. Official Use Only (●) No ( ) Yes Exemption No. ____
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9. Release Level? (●) Public ( ) Limited

F. Complete for a Journal Article

1. Title of Journal

G. Complete for a Presentation

1. Title for Conference or Meeting ______
2. Group Sponsoring ______
3. Date of Conference ______
4. City/State ______
5. Will Information be Published in Proceedings? (●) No ( ) Yes
6. Will Material be Handed Out? (●) No ( ) Yes

H. Information Owner/Author/Requestor
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(Print and Sign)

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Approval by Direct Report to President (Speech/Articles Only) ______
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Other	☐			Y / N
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J. Comments

Information Clearance Approval

APPROVED
By Julia Raymer at 10:23 am, Jun 22, 2015

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ORP-59017
Revision 0

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Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Office of River Protection

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Date Published
June 2009

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Office of River Protection

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APPROVED
By Julia Raymer at 10:24 am, Jun 22, 2015

Release Approval　　Date

VSL-03R3470-2

Final Report

LAW Glass Formulation to Support AP-101 Actual Waste Testing

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for

Duratek, Inc.

and

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January 24, 2003

Rev. 0, 8/13/03

Document Title: LAW Glass Formulation to Support AP-101 Actual Waste Testing

Document Number and Revision: VSL-03R3470-2, Rev. 0
Issue Date: **8/12/03**
Performing Organization: Vitreous State Laboratory, The Catholic University of America

Test Specification: 24590-LAW-TSP-RT-01-016, Rev. 0
Test Exceptions: None
Test Plan: VSL-01T5600-2, Rev. 1
R&T Focus Area(s): LAW Waste Form Qualification
Test Scoping Statement(s): VSL-27, Glass Formulation to Support Actual Waste Testing

Completeness of Testing:

This report describes the results of work and testing specified by the above-listed Test Specification(s), Test Plan(s), and Text Exception(s). The work and any associated testing followed established quality assurance requirements and was conducted as authorized. The descriptions provided in this test report are an accurate account of both the conduct of the work and the data collected. Results required by the Test Plan are reported. Also reported are any unusual or anomalous occurrences that are different from the starting hypotheses. The test results and this report have been reviewed and verified.

I.L. Pegg: I. L. Pegg **Date:** 8/21/03
VSL Program Director/Principal Investigator

I. Joseph: Innocent Joseph **Date:** 8/21/03
Duratek Sub-Contract Manager

Document Revision Log

Rev. A	1/24/03	Original draft for external review.
Rev. 0	8/13/03	Incorporated WTP comments.

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List of Abbreviations

ANL-LRM	Argonne National Laboratory – Low-Activity Reference Material
ANS	American Nuclear Society
ANSI	American National Standard Institute, Inc.
ASTM	American Society for Testing and Materials
CCC	Container Centerline Cooled
DCP-AES	Direct Current Plasma Atomic Emission Spectroscopy
DTA	Differential Thermal Analysis
EDS	Energy Dispersive X-Ray Spectroscopy
ILAW	Immobilized Low Activity Waste
LAW	Low Activity Waste
NIST	National Institutes of Standards and Technology
ORP	Office of River Protection
PCT	Product Consistency Test
PNNL	Pacific Northwest National Laboratory
PNWD	Pacific Northwest Division (Battelle)
PSWP	Products and Secondary Wastes Plan
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan for Testing Programs Generating Environmental Regulatory Data
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
RPP-WTP	River Protection Project-Waste Treatment Plant
SEM	Scanning Electron Microscopy
SEM/EDS	Scanning Electron Microscopy/Energy Dispersive X-Ray Spectroscopy
SRTC	Savannah River Technology Center
T_g	Glass Transition Temperature
TCLP	Toxicity Characteristic Leaching Procedure
TFA	Tank Focus Area
TF COUP	Tank Farm Contractor Operation and Utilization Plan
TOC	Total Organic Carbon
TTT	Time-Temperature-Transformation
TWRS	Tank Waste Remediation System
UTS	Universal Treatment Standards
VHT	Vapor Hydration Test
VSL	Vitreous State Laboratory
WTP	Waste Treatment and Immobilization Plant
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

SUMMARY OF TESTING

A) Objectives

The main objective of the work was to develop and select a glass formulation for vitrification testing of the actual waste sample of LAW AP-101 at Battelle - Pacific Northwest Division (PNWD). Other objectives of the work included preparation and characterization of glasses to demonstrate compliance with contract and processing requirements, evaluation of the ability to achieve waste loading requirements, testing to demonstrate compatibility of the glass melts with melter materials of construction, comparison of the properties of simulant and actual waste glasses, and identification of glass formulation issues with respect to contract specifications and processing requirements.

B) Conduct of Testing

LAW AP-101 actual waste composition data from PNWD was reviewed and glass formulations were developed for testing. Crucible melts of the glasses (about 400 g) were prepared by melting mixtures of reagent grade or higher purity chemicals in platinum-gold crucibles at 1200°C for 75 minutes. The melts were stirred and the molten glass was poured onto a graphite plate to cool; samples of the resulting glass were then analyzed. In addition, crucible melts of the glass composition that was selected for actual waste testing were prepared from a slurry feed and characterized.

The viscosity and electrical conductivity of the melts were measured in the range of 950-1250°C. Glass samples were heat treated at 700°C and 850°C to determine the bounds on the liquidus temperature and to determine the effect of heat treatment on crystallization. Since LAW glasses are designed to have a liquidus temperature of 950°C or less and generally show little tendency to crystallize, heat treatments were done at lower temperatures of 700°C and 850°C. Secondary phase were identified and quantified by optical microscopy, x-ray diffraction, or scanning electron microscopy in conjunction with energy dispersive x-ray spectroscopy. The Product Consistency Test (PCT), Vapor Hydration Test (VHT), and Toxicity Characteristic Leaching Procedure (TCLP) were performed on selected glass samples. The density of the glass at room temperature and the glass transition temperature were determined by pycnometry and differential thermal analysis, respectively.

C) Results and Performance Against Objectives

LAW AP-101 waste composition data from PNWD was reviewed and glass formulations were developed for testing. Crucible melts of the glasses were prepared and characterized. Based on the results of testing, the LAWA126 glass composition was selected for testing with the actual AP-101 waste sample at PNWD. Glass composition and batching instructions were submitted to the WTP to support actual waste testing. Samples of selected glasses were characterized for properties such as PCT, TCLP, VHT, viscosity, electrical conductivity, refractory corrosion, density, liquidus temperature, crystallization, and glass transition temperature. All of the measured properties of the glass selected for actual waste testing, LAWA126, are within the limits of contractual and processing requirements. Comparison of the properties of simulant and actual waste glasses, which was one of the Test Plan objectives, was not accomplished because results of actual waste testing were not available during the preparation of this report.

D) Quality Requirements

This work was conducted under an NQA-1 (1989) and NQA-2a (1990) Part 2.7 based quality assurance program that is in place at the VSL. This program is supplemented by a Quality Assurance Project Plan for RPP-WTP work that is conducted at VSL. Test and procedure requirements by which the testing activities are planned and controlled are also defined in that plan. The program is supported by VSL standard operating procedures that were used for this work. This work was not subject to DOE/RW-0333P, Rev. 10.

TCLP data were collected at the VSL. The TCLP data provided in this report were not collected by an external SW-846 laboratory and do not fully meet the requirements of the QAPjP.

E) Issues

Comparison of the properties of simulant and actual waste glasses was not performed because the results of actual waste testing were not available during preparation of this report.

SECTION 1
INTRODUCTION

1.1 Background

Acceptable glass formulations for vitrification of Hanford LAW waste streams must meet a number of product quality, processability, and waste loading requirements, as summarized in Table 1. Glass formulation work to address these requirements was performed during Part A and Part B1 [1, 2, 3]. The primary objective of the present work was to develop and test LAW glass formulations from which compliant compositions would be selected to support vitrification of actual AP-101 waste [4], the product of which will be used to demonstrate WTP's ability to meet the Immobilized Low Activity Waste (ILAW) product requirements. This report is responsive to the Test Specification [5] and Test Plan [6] for LAW glass formulation to support actual waste testing.

As part of the glass formulation development work, the Waste Treatment and Immobilization Plant (WTP) project is preparing LAW glass from both simulants and actual waste samples. Glass formulation work with simulants is being performed at the Vitreous State Laboratory (VSL) while confirmatory testing with actual waste samples is being performed at the Battelle - Pacific Northwest Division (PNWD) and at the Savannah River Technology Center (SRTC). Development and testing of LAW glass formulations with simulants are necessary to identify target glass compositions and qualified composition regions for the ILAW products and to demonstrate the ability to meet ILAW product requirements set forth in the WTP contract [7]. Testing with actual waste samples and comparison of the results from the simulant and actual waste testing are used to validate the use of simulants in glass development work and to confirm that the LAW glasses will meet the ILAW product requirements.

During Part B1, fully compliant target glass compositions were identified for eight of the tank wastes based on available characterization data. The selection of the tanks was made on the basis of a schedule of feed delivery [8] that has since been changed several times [9, 10, 11]. Selected glass formulations developed for the Part B1 tanks at VSL have subsequently been employed to support vitrification testing with actual wastes at PNWD [12] and SRTC [13, 14]. These include LAW Sub-Envelope A1 (LAWA44 glass) for AN-103, LAW Sub-Envelope A2 (LAWA88 glass) for AW-101, LAW Sub-Envelope C1 (LAWC22 glass) for AN-107, and LAW Sub-Envelope C2 (LAWC21 glass) for AN-102. Vitrification tests of AP-101 (at PNWD) and AZ-102 (at SRTC) actual waste samples are currently in progress. The purpose of the work described in this report was to develop and test the LAW glass formulations for the vitrification of the AP-101 actual waste sample at PNWD.

As part of the ILAW waste form qualification strategy and to demonstrate compliance with contract specifications for ILAW products [15], the WTP Project has continued testing using actual waste samples. As described in the Interface Control

Document 23 [16], actual waste samples were provided by Office of River Protection (ORP) for testing to confirm the results of flow-sheet calculations and testing with simulated wastes. Waste samples received from tanks AP-101 and AZ-102 have been pretreated, analyzed, and have already been tested or are scheduled for vitrification testing at PNWD and SRTC, respectively. Testing of actual waste samples of tanks SY-101/AP-104, AZ-101, and AN-104 will follow. The compositions and properties of the resulting glasses will be characterized to demonstrate compliance with contract requirements and to provide comparison with the results obtained with simulants. During Part B1, ILAW product testing with actual wastes showed that the glasses prepared based on target formulations developed at the VSL met or exceeded contract specifications for waste loading, chemical composition, radionuclide concentration limits, and ILAW performance [12-14].

The results from glass development and testing conducted at VSL to support vitrification of the LAW AP-101 actual waste sample at PNWD are presented in this report. The development work led to the selection of LAWA126 as the target glass composition for the vitrification of LAW AP-101 actual waste.

1.2 Test Objectives

The principal objectives of this work were:

- Review data from the characterization of the pretreated actual waste sample of LAW AP-101 and define a simulant for the waste;
- Compare actual waste composition data to the compositions published in the TFCOUP [9, 10, 11] and determine the suitability of one of the glass compositions developed during Part B1 for this actual waste;
- Develop compliant LAW glass formulations for each simulant composition, as needed;
- Prepare crucible melts of the LAW glass formulations and test the resulting glasses with respect to chemical composition, melt properties, and glass properties to ensure that the formulations meet all contract and process requirements;
- Recommend the LAW glass formulation and associated glass formers. Provide batching instructions for testing with actual waste at PNWD.
- Evaluate the ability to achieve the waste loading limits required by the RPP-WTP contract for the waste sample selected for actual waste testing.

- Test selected glass formulations to ensure their compatibility with melter materials of construction.

- Compare properties of glasses made from samples of actual waste and simulated waste. Investigate any differences in results between simulated and actual sample testing to understand, and if possible, eliminate the reasons for the difference;

- Identify glass formulation issues with respect to contract specifications or process requirements.

Comparison of the properties of the simulated and actual wastes, melter feeds, and glasses is one of the major objectives of this task. However, since data from actual waste testing at PNWD is not yet available, this comparison will be presented in the PNWD report on LAW AP-101 actual waste testing.

1.3 Test Overview

After receiving and reviewing Project-approved analytical data on the compositions of the LAW AP-101 waste samples from PNWD [4], a determination was made that the existing glass formulations (from Part B1 and Part A) would not be suitable for vitrification testing of this actual waste sample. Formulations developed during Part B1 for LAW Sub-Envelope A2 wastes were based on a much lower ratio of potassium to sodium in the waste based on TFCOUP, Rev. 2.

Past experience, as well as data and property-composition models were used to guide the new formulation effort. Glass samples were prepared in small crucible melts for each new formulation and then characterized to obtain the required composition-property information. An iterative process was employed in that the property characterization data were fed back and used in the selection of subsequent formulations for testing until a target formulation for the waste sample was identified.

The glass formulation parameters and constraints imposed by contract specifications and process requirements were identical to those found in Part B1; these are given in Table 1. Glass samples prepared with simulants were subjected to the tests listed in the Test Specification [5].

Note that for consistency, and since a revised LAW container cooling profile is not yet available, the same profile as was used for the earlier actual waste melts [2] was used in the entirety of the present work.

SECTION 2
EVALUATION OF ACTUAL WASTE SAMPLE DATA

The actual sample composition based on characterization reports transmitted by the Project [4] was compared to the estimated composition published in the current flow-sheet projections [9] and best basis inventory [8] (the sample analysis is given in Table 2). The review included evaluation of the sulfate-to-sodium ratio, which is critical to the design of LAW glasses, and the potassium to sodium ratio, which is important for AP-101 waste stream. The major difference in the waste composition between the earlier information [8, 9] and the actual waste data [4] is the increase in the potassium concentration. On an oxide basis, the K_2O concentration increases from 8.83 wt% to 15.72 wt% when the waste composition basis is changed from the flow sheet projections based on TFCOUP Rev. 2 [9] to the actual waste data [4]: also, the SO_3 concentration increases from 1.17 wt% to 1.41 wt%. Changes in the concentrations of the other components are inconsequential.

The sulfate-to-sodium molar ratio for this sample is 0.0075, which is within the contractual limits defined in contract Specification 7 [15]. The target glass composition for this sample of LAW AP-101 was developed to be in compliance with the contract waste-loading limit of greater than 14 wt% Na_2O. LAW AP-101 has the highest potassium concentration among all the Phase I LAW waste streams. In addition, the sulfate to sodium ratio is low and, as a result, the waste loading is limited by the total alkali content (Na_2O + K_2O) in the resulting glass, as discussed in the following sections. Glasses were prepared and tested at three waste loadings corresponding to waste contributions of sodium oxide in the glass of 19.5 wt%, 18 wt% and 15.9 wt%. The Na_2O concentration in the glass was increased by 2.56% (relative) to account for sodium additions in pretreatment.

The measured [4] total organic carbon (TOC) content of LAW AP-101 actual waste at a concentration of 5.62 molar sodium is 1960 mg/L. Characterization of the organic constituents provided information only on oxalate.

SECTION 3
DEVELOPMENT AND PREPARATION OF SIMULANT GLASSES

As noted earlier, the potassium concentration in LAW AP-101 is the highest among all of the early LAW waste streams and the sulfate concentration is relatively low. Consequently, the waste loading in the glass is limited by the total alkali content (Na_2O + K_2O). The total alkali content of LAW glasses is limited primarily by refractory corrosion and performance on the vapor hydration test (VHT). LAWA88 was initially identified as a suitable glass composition for LAW Sub-Envelope A2 (LAW AP-101) waste based on the concentration information in TFCOUP, Rev. 2 [9]. However, the actual waste composition showed a much higher potassium concentration. Therefore, additional glass formulation work was conducted to identify a glass composition with a higher concentration of potassium. Five glass formulations were developed and tested to identify a suitable composition for the vitrification of LAW AP-101 actual waste sample at PNWD.

The formulations developed and tested for this waste sample are designated LAWA125 to LAWA129. Three of these formulations were prepared with the same mixture of glass former additives as were previously used for LAWA88 glass, but with different waste loadings corresponding to approximately 27, 25, and 22 wt% for LAWA125, LAWA126 and LAWA127, respectively. The other two formulations (LAWA128 and LAWA129) were prepared with different glass former mixtures but with the same waste loading of approximately 25%. Waste contributions to the glass on an oxide basis for the three waste loadings, both with and without the effect of sodium additions in pretreatment, are given in Table 3. The formulations are given in Tables 4 to 6. The organic species and carbonate in the simulants are destroyed in the melting process, leaving principally the inorganic oxides. In these glasses rhenium (Re) was added as a simulant for technetium (Tc). However, unless requested by WTP no special analyses for Re are planned. Some glasses were spiked with Cesium (Cs) to facilitate comparison with the LAW glasses used for melter testing.

A total of eleven simulant melts from dry batches and one melt from a slurry (PNLA126CC) were prepared (see Tables 7 and 8). The waste simulant recipe for LAW AP-101 testing was developed at VSL using the information provided in the analytical report [4]. To account for pretreatment processes, the reported sodium in the waste was increased 2.56% (equivalent to 5.768 M). For the simulant slurry, all constituents were then increased to an equivalent sodium concentration of 8 M (see Appendix A1). A simulant concentration of 8 molar sodium was used in the preparation of the slurry feed in order to produce a feed with the desired rheological properties [3]. Glass formers were added (Appendix A2) to match the batching recipe provided to PNWD (Appendix A3). The formulations were melted at 1150°C and either quenched or container cooled. Container cooling curves from Part B1 were used because new cooling curves from the WTP are not yet available. The cooling curve that was used in this work is given in Figure 1. If cooling curves supplied by the WTP in future differ substantially from that used, these heat treatments may have to be repeated. A summary of the preparation, heat treatment, and characterization of the glasses is given in Table 7.

SECTION 4
CHARACTERIZATION DATA ON SIMULANT GLASSES

All of the crucible melt glasses were tested with respect to the properties specified in the Test Specification [5], and with respect to the properties of importance for the selected vitrification process. Details of the characterization are given below.

4.1 Chemical Composition

All glasses were analyzed by x-ray fluorescence (XRF). Results of the analyses are given in Table 8. The target and analyzed glass compositions are generally in good agreement.

4.2 Glass Melt Properties

The melt viscosity and electrical conductivity were measured between 950°C and 1250°C. The liquidus temperatures were determined to be below 850°C based on heat treatments of 20 hours. Additional heat treatments were performed at 700°C for 20 hours on selected samples as a further test to determine crystallization tendency. Per the requirements given in Table 1 LAW glasses are required to have a liquidus temperature of less than 950°C. However, since these glasses show little or no crystallization at 950°C the heat treatments were done at lower temperatures of 850°C and 700°C.

The results from characterization of the heat-treated glasses are given in Table 7. All of the as-melted and heat-treated glass samples were homogeneous in appearance. The melt viscosity and electrical conductivity at standard temperatures are given in Table 9. The viscosity and electrical conductivity of LAWA126 are within the required ranges given in Table 1. As expected, the viscosity of the glasses decrease as the alkali content is increased. The melt viscosity of glass LAWA126 as a function of temperature is plotted in Figure 2.

4.3 Glass Density and Glass Transition Temperature

The density and glass transition temperature of glasses LAWA126 and LAWA127 are presented in Tables 10 and 11, respectively. As previously observed, for most LAW glasses [2] these properties show relatively little change with glass composition.

4.4 Product Consistency Test (PCT)

The PCT results for the five glass formulations, as measured per ASTM C 1285 Method A (7 day leach at 90°C) are presented in Table 12. All five glasses show normalized mass losses that are far lower than the required limit of 2 g/m^2. Since the PCT results of all of the glasses were much better than the ILAW requirements, amongst these five compositions, this was not a criterion in the selection of a target composition for actual waste vitrification.

4.5 Vapor Hydration Test (VHT)

The VHT was performed on a total of eight glass samples, using the contract requirement of 7-day exposure as well as 24-day exposure for comparison to data collected during Part B1 [2], both at 200°C. The results are presented in Table 13 for five different samples of LAWA126 and one sample each of LAWA125, LAWA127, and LAWA129. The results show that the glass composition selected for actual waste testing (LAWA126) performs much better than the ILAW contract requirement. As expected, the highest VHT alteration rates are observed for LAWA125, which has the highest alkali content.

4.6 Crystalline Phases

The crystallization characteristics of these glasses were determined using SEM/EDS, XRD, and Optical Microscopy/Image Analysis on both quenched and Container Centerline Cooled (CCC) samples. No secondary phases were observed in any of the samples (Figure 3).

4.7 Refractory Corrosion

Two samples of LAWA126 glass were tested for their compatibility with K-3 refractory using methods described previously [17]. Refractory corrosion was an important criterion limiting waste loading in LAW Sub-Envelope A2 glasses. Results of 6-day K3 coupon tests at 1208°C are given in Table 14. Results of K-3 coupon corrosion tests on glass compositions used at West Valley for waste treatment and at the LAW Pilot Melter are included for comparison.

4.8 Toxicity Characteristic Leaching Procedure (TCLP)

The glasses were subjected to the TCLP protocol at VSL to determine their leaching behavior; however, these glasses contain only very small amounts of the RCRA metals. Table 15 provides the TCLP results for all of the RCRA metals and glass constituents. As expected, TCLP results for all RCRA metals are below the Universal Treatment Standard (UTS) limit and most are below the detection limits.

4.9 Other Requirements

Construction of Time-Temperature-Transformation (TTT) diagrams for the target LAW glass formulations (Contract Specification 2.2.2.6.3); leachability index per ANSI/ANS-16.1 (Contract Specification 2.2.2.17.1); compressive strength (Contract Specification 2.2.2.18); and compressive strength after thermal, radiation, biodegradation, and immersion per ANSI/ANS 16.1 (Contract Specification 2.2.2.19), were not part of the current test scope [5, 6].

4.10 Compliance with ILAW Product Specifications

Based on the characterization data listed above, no issues related to compliance with contract specifications [7] or process requirements, as summarized in Table 1, were identified. The selected LAWA126 formulation meets the Envelope A waste loading requirements of incorporating at least 14.0 wt% waste sodium oxide and meets all of the product quality requirements and imposed processability constraints.

4.11 Comparison Between Actual and Simulant Glasses

Data from testing of actual waste samples are not presently available. The comparison of results from simulant and actual waste testing will be included in the PNWD report on LAW AP-101 actual waste testing.

SECTION 5
BATCHING RECIPE FOR ACTUAL WASTE VITRIFICATION

The composition of the actual waste sample and the VSL developed simulant (see Appendix A1) were used to develop a feed batching recipe for actual waste vitrification. The objective was to produce a viable feed slurry for subsequent vitrification in a crucible melt.

A simulant concentration of 8.0 molar sodium was recommended for use based on previously tested feeds of similar waste loading [3]. The LAW AP-101 waste simulant was prepared according to the recipe provided in Appendix A1 and tested. After overnight mixing, the pH was measured to be 13.56 and the density was 1.39 g/ml, both at 25°C. After addition of the glass formers, the resulting feed for LAW126 had a density of 1.76 g/ml and a pH of 11.39 at 25°C. The yield stress at 25°C was very difficult to measure and was estimated to be below 0.1 Pa. The feed recipe is given in Appendix A2. The viscosity of the feed as a function of shear rate is shown in Appendix B1. The procedure used for rheological characterization of the feed is given in Section 7.12. The measurements were conducted at 25°C.

Based on these results, a feed batching recipe and vitrification instructions for the actual sample were prepared and submitted to the Project. The formulation was transmitted with the recommended waste concentration of 8.0 molar sodium for vitrification testing, a list of glass former chemicals, and batching instructions, as shown in Appendix A3. The Project reviewed and approved the proposed target formulation and glass former mix and recipe before their use in actual waste testing at PNWD.

SECTION 6
SUMMARY AND CONCLUSIONS

The glass formulation LAWA126 was developed, characterized, and recommended for actual waste vitrification of LAW AP-101 at PNWD. The formulation meets all contract specifications and contract requirements for the properties that were measured. Batching instructions for actual waste vitrification using this formulation were prepared and provided to the Project and were approved for use at PNWD. The VSL/Duratek part of the overall LAW AP-101 actual waste testing task, which was to develop and provide a compliant glass formulation for actual waste vitrification at PNWD, was successfully completed. The next part of the overall activity, which is the testing with actual waste, will be conducted at PNWD based on the formulation developed in the present work. Comparison of the simulant and actual waste testing will be presented in the PNWD report on LAW AP-101 actual waste testing.

All of the glass samples prepared in this work were examined for homogeneity and secondary phases. Chemical analyses of the glass samples were performed to confirm their composition. Further characterization was done in two stages with limited characterization of key properties performed on all glasses and complete characterization performed on the selected glass. All glasses were characterized with respect to melt viscosity, melt electrical conductivity, Product Consistency Test (PCT) at 90°C, and effect of heat treatment. Glass compositions selected for active waste testing were subjected to additional analyses after preparation by melting a simulated waste feed slurry (liquid waste simulant mixed with glass former chemicals) followed by heat treatment according to the container cooling profile previously provided by the Project. These include Vapor Hydration Test (VHT), determination of glass transition temperature (T_g), measurement of density, and characterization for crystal content by scanning electron microscopy/energy dispersive x-ray spectroscopy (SEM/EDS), x-ray diffraction (XRD), and Optical Microscopy/Image Analysis (vol%) on both quenched and Container Centerline Cooled (CCC) samples.

SECTION 7
EXPERIMENTAL METHODS

A short description of the experimental methods and instrumentation that were used for this work is provided below. The techniques are described in detail in controlled technical procedures [18] that form part of the VSL QA program.

7.1 Glass Preparation

The glasses were prepared from reagent grade or higher purity chemicals to produce a batch size of approximately 400 g according to VSL standard operating procedures. Crucible melts were prepared by melting the appropriate combination of well-mixed chemicals at 1200ºC for 75 minutes in a platinum, platinum-gold, or platinum-rhodium crucible. In addition, glass compositions selected for active waste testing were also prepared by melting a simulated waste feed slurry (liquid waste simulant mixed with glass former chemicals). Mixing of the melt was accomplished with a platinum stirrer beginning 15 minutes after the start of melting and continuing for the next 60 minutes, until the end of melting. The molten glass was poured onto a graphite plate to cool, and the resulting glass was then distributed for analyses. When simulating the LAW centerline container cooling heat treatment, the glass was remelted at 1200ºC and then cooled in the crucible according to the prescribed cooling profile. A computer that is interfaced to the furnace controlled the entire cooling process and also provided for regular acquisition of key parameters during the cooling process.

7.2 Compositional Analysis

Glass samples for analysis were subjected to microwave-assisted total acid dissolution in Teflon vessels according to VSL standard operating procedures. Twenty milliliters of a 1:5 mixture of concentrated $HF:HNO_3$ were diluted to 50 ml and used for the dissolution. This procedure is similar to the ASTM Test Method C1412-99, which also employs a mixture of concentrated HF and HNO_3 in microwave digestion of pulverized glass samples; supplemental use of HCl/H_3BO_3 is not included in the VSL procedure since boron is normally one of the analytes. The resulting solutions were analyzed by direct current plasma atomic emission spectroscopy (DCP-AES) for all constituents except sulfur, for which Dionex Ion Chromatography was used. However, the primary method used for glass composition analysis was x-ray fluorescence (XRF) on powdered glass samples. An ARL 9400 wavelength dispersive XRF spectrometer was used for this purpose.

7.3 Viscosity

The melt viscosity, η, was measured using a Brookfield viscometer. Measurements were performed in the temperature range of 950-1250ºC, and the data interpolated to standard temperatures using the Vogel-Fulcher equation: $\ln \eta = [A/(T-T_o)]+B$, where A, B, and T_o are fitting parameters. The equipment was calibrated at room temperature using standard oils of known viscosity, and then checked at 950-1250ºC using a National Institute of Standards and Technology (NIST) standard reference glass (SRM 711). Both precision and accuracy of the viscosity measurements are estimated to be within ±15 relative%.

7.4 Electrical Conductivity

The electrical conductivity, σ, of each glass was determined by measuring the resistance of the glass melt as a function of frequency using a calibrated platinum/rhodium electrode probe attached to a Hewlett-Packard model 4194A impedance analyzer. Measurements were performed over similar temperature ranges to those employed for the melt viscosity measurements. The results were extrapolated to zero frequency to obtain the DC conductivity. The conductivity data were then interpolated to standard temperatures using the Vogel-Fulcher equation: $\ln \sigma = [A/(T-T_o)] + B$, where A, B and T_o are fitting parameters. Estimated uncertainties in the conductivity measurements are ± 20 relative%.

7.5 Liquidus Temperature and Percent Crystallinity

The bound on the liquidus temperature of the glass was determined by heat treatment followed by analysis using optical and/or electron microscopy. Samples of about 500 mg were heated in a platinum, platinum-gold, or platinum-rhodium crucible at a pre-melt temperature of 1200ºC for one hour to destroy any pre-existing nuclei, followed by heat treatment at 850ºC or 700°C; heat treatment times of 20 hours and above were used. The heat treated glass was then quenched by submerging the outside of the crucible in cold water. This quenching freezes in the phase assemblage in equilibrium with the melt at the heat treatment temperature. If no crystals are observed in the cooled glass, then it is inferred that no crystals are present at the heat treatment temperature and that the liquidus temperature is below the heat treatment temperature. Some additional heat treatments were also performed at 700°C; however, given the generally low liquidus temperatures for the LAW glasses [2] no attempts were made to further bound the liquidus temperature. Per the requirements given in Table 1, LAW glasses are required to have a liquidus temperature of less than 950°C. However, since these glasses show little or no crystallization at 950°C, the heat treatments were done at lower temperatures of 850°C and 700°C.

7.6 Secondary Phases

Scanning electron microscopy (SEM) was used to characterize the microstructure of the glasses and energy dispersive x-ray spectroscopy (EDS) was used to analyze the glassy and crystalline phases that were observed. Typical magnifications used ranged from 25× to 5,000×.

Digital imaging and analyses was used to determine the volume fractions of crystalline phases in both as-melted and heat-treated glasses. The accuracy of the volume percentage determinations is limited by uniformity of distribution and imaging characteristics of the particular phase. The accuracy of the volume percentage determination is estimated to be within 20% relative.

Optical microscopy with image analysis and quantitative x-ray diffraction were used in combination with the SEM/EDS measurements.

7.7 Product Consistency Test (PCT)

The product consistency test (PCT; ASTM C 1285) is used to evaluate the relative chemical durability of glasses by measuring the concentrations of the chemical species released from 100-200 mesh crushed glass (75-149 µm) to the test solution (de-ionized water in this case). PCT tests on the LAW glasses were performed at 90ºC, in accordance with the current RPP-WTP contract requirement. The ratio of the glass surface area to the solution volume for this test is about 2000 m^{-1} (10 g of 100-200 mesh glass is immersed in 100 ml deionized water). All tests were conducted in triplicate, in 304L stainless steel vessels, and in parallel with a standard glass included in each test set. The internal standard was the ANL-LRM reference glass [19], which has undergone round-robin testing. The leachates are sampled at predetermined times, the first of which is seven days. One milliliter of sampled leachate is mixed with 20 ml of 1M HNO_3 and the resulting solution is analyzed by DCP-AES; another 3 ml of sampled leachate is used for pH measurement.

7.8 Vapor Hydration Test (VHT)

The vapor hydration tests are run in Parr series 4700 screw-cap pressure vessels made of 304L stainless steel and having either 22 or 45 ml capacity in accordance with the procedure given in Appendix A of the PSWP [20]. Glass coupons are fashioned about 5 to 10 mm square, about 2 mm thick, and with one cut and one fracture surface face. A hole approximately 1.6 mm in diameter is drilled near one corner of the coupon to allow it to be suspended from a hanger made of 24 gauge stainless steel wire. Dimensional measurements are made to permit calculation of the area, and the coupon is weighed before and after the VHT on a balance having a resolution of 100 µg. The coupon is suspended vertically from the hanger in the pressure vessel, and enough deionized water added to the vessel to saturate the volume at the test temperature, 200ºC, and to allow for a non-dripping layer covering the coupon. The pressure vessels are flushed with argon, sealed, weighed, and placed in an oven held at 200ºC. The temperature is monitored continuously with an independent thermocouple. At the completion of the test, the pressure vessels are removed and immediately partially immersed in an ice water bath to condense the water vapor near the bottom of the vessel. Once cool, the vessels are weighed and opened, and then the coupons are removed and weighed. If the difference in the mass of the sealed pressure vessel before and after the test indicated a water loss in excess of 50% of the original amount, the test results are discarded. The coupons are then examined using low power optical microscopy and an X-ray diffraction pattern is taken directly off the surface of the coupon. Next, the coupons are sectioned and the pieces mounted separately to allow for SEM examination both of the cross section of the leached coupon and the leached surface itself. For consistency with existing data, the nominal test duration was 24 days; however, shorter duration (7 days) was also used for selected glasses.

7.9 Glass Density

Density measurements were made on 5 to 15 grams of crushed glass using the pycnometric method described in ASTMD 854-83. Densities of two NIST glasses (Lead Silicate Glass #1827a and Soda Lime Glass #1826b) were measured as calibration checks. To confirm reproducibility of the results, triplicate analyses of ten percent of the samples was also performed, as required by the procedure.

7.10 Glass Transition Temperature

The glass transition temperature was measured by differential thermal analysis (DTA) with respect to a reference material that has no transitions in the region of interest (e.g., alumina). About 30 mg of powdered glass sample (< 75 μm particle size) were analyzed with a Perkin Elmer differential thermal analyzer DTA7 coupled to a TAC 7/DX controller. The sample is heated from 300°C to 700°C at a rate of 10°C/min while the temperatures of the sample and reference are monitored. During the heating, any endothermic or exothermic transitions in the sample are reflected as a simultaneous temperature difference between the sample and the reference. The glass transition temperature was determined as the extrapolated onset temperature in a manner similar to that described in ASTM E 1356-91 (Standard Test Method for Glass Transition Temperatures by Differential Scanning Calorimetry or Differential Thermal Analysis). Extrapolations from the first baseline deviation temperature and from the midpoint temperature, for which the point of inflection is used, are drawn. The glass transition temperature is taken as the temperature at the intersection of the two extrapolations.

7.11 Toxicity Characteristic Leaching Procedure (TCLP)

The EPA TCLP leach test (SW-846 Method 1311) was used to measure the leach resistance of crushed glass (< 3/8”) in a sodium acetate buffer solution after 18 hours at 22°C with constant end-over-end agitation. A mass of about 100 grams of glass was leached in 2 liters of TCLP extract, according to the extraction method for non-volatiles. The surface area to volume ratio for this test is about 20 m^{-1}, which is about two orders of magnitude lower than that in PCT. The leachate concentrations were measured by DCP-AES. The overall uncertainty associated with this test is estimated to be ± 20% based on repeat analyses of glass samples at the VSL.

TCLP analyses to support environmental and regulatory requirements need to be conducted according to the requirements of the WTP QAPjP [21]. Separate measurement by an external SW-846 laboratory was not requested by the Project and therefore was not performed. Consequently, the TCLP data provided in this report do not fully meet the requirements of the QAPjP [21].

7.12 Rheological Characterization of the Feed

Rheological properties of the feed were measured using a Haake RS75 rheometer, which was equipped with either a Z40DIN or a FL22-SZ40 sensor. A typical set of measurements consists of identifying the flow characteristics of the slurry by measuring the shear stress on the slurry at controlled shear rates and temperatures. In these measurements, the shear rate values are preset and are increased stepwise from 0.01 s^{-1} to 200 s^{-1} with a sufficient delay (typically 15 to 30 seconds) between steps to ensure that shear stress is allowed to fully relax and therefore is measured at equilibrium. It should be noted that this approach is somewhat different than the "flow curve" approach in which the shear rate is ramped up to some maximum value and then ramped back down to produce a hysteresis curve that is dependent on the selected ramp rate. In contrast, the present measurements are equilibrium values of the shear stress at each measured shear rate. The viscosity of the sample as a function of the shear rate is then calculated as the ratio of the shear stress to the shear rate.

The yield stress data for the melter feeds were measured using a controlled-stress mode in which the torque on the rotor was slowly increased while the resulting deformation of the fluid was monitored. The discontinuity in the measured deformation-torque curve was identified as the yield stress. It should be noted that this direct measurement of the "true" yield stress can be quite different from the value that is often reported as the yield stress, which is instead obtained by extrapolation of the shear stress-shear rate curve to zero shear rate.

SECTION 8
QUALITY ASSURANCE

This work was conducted under an NQA-1 (1989) and NQA-2a (1990) Part 2.7 based quality assurance program that is in place at the VSL. This program is supplemented by a Quality Assurance Project Plan for RPP-WTP work [22] that is conducted at VSL. Test and procedure requirements by which the testing activities are planned and controlled are also defined in this plan. The program is supported by VSL standard operating procedures that were used for this work [18]. This work was not subject to DOE/RW-0333P, Rev. 10.

TCLP data were collected at the VSL. The TCLP data provided in this report (Table 15) were not collected by an external SW-846 laboratory and do not fully meet the requirements of the QAPjP [21].

SECTION 9
REFERENCES

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[13] "Crucible-Scale Active Vitrification Testing of Envelope A, Tank 241-AN-103 (U)," C.L. Crawford, D.M. Ferrara, R.F. Schumacher, and N.E. Bibler, WSRC-TR-2000-00322, SRT-RPP-2000-0021, Rev. 1, June 15, 2001.

[14] "Crucible-Scale Active Vitrification Testing of Envelope C, Tank 241-AN-102 (U)," C.L. Crawford, D.M. Ferrara, R.F. Schumacher, and N.E. Bibler, WSRC-TR-2000-00371, SRT-RPP-2000-0022, Rev. 0, June 15, 2001.

[15] Contract No. DE-AC27-01RV14136; Section C: Statement of Work, Specification 7 Low-Activity Waste Envelope Definition, December 2000.

[16] "ICD23 - Interface Control Document for Waste Treatability Samples," 24590-WTP-ICD-MG-01-023, Rev 1, 15 August 2002.

[17] "Corrosion of K-3 Refractory and Metal Alloys in RPP-WTP LAW Glasses," H. Gan, X. Lu, I. Vidensky, M. C. Paul, and I.L. Pegg, VSL-01R3540-1, Final Report, Vitreous State Laboratory, Washington, D.C., March 16, 2001.

[18] "Master List of Controlled VSL Manuals and Standard Operating Procedures in Use," QA-MLCP, Rev. 0, August 14, 2001.

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[20] "Products and Secondary Wastes Plan (PSWP)," RPP-WTP Project, PL-W375-TE00001, Rev. 0, 4/12/2000.

[21] "Quality Assurance Project Plan for Testing Programs Generating Environmental Regulatory Data", RPP-WTP Project, PL-24590-QA00001, July 2001.

[22] "Quality Assurance Project Plan for RPP-WTP Support Activities Conducted by VSL," Vitreous State Laboratory, QAPP, Rev. 4, 8/2/2001.

Table 1. Summary of ILAW Requirements.

Specification Source in contract [15] unless specified	Contracting Requirements	Required limit of given specification		
2.2.2.2	Waste Loading (based on Na_2O wt%)	>14% LAWA	>5% LAWB*	>10% LAWC
2.2.2.3 2.2.2.4 2.2.2.5	Cylindrical package is 1.22m in diameter and 2.3 m high; mass shall not exceed 10,000 kg. Void shall not exceed 10%.	Density of glass < 3.7		
2.2.2.6.1 2.2.2.6.2	Chemical composition of glass	Report all components greater than 0.5% and all regulated elements		
2.2.2.6.3	Crystalline Phase Identification	Report amount of crystals and provide TTT		
LAW SPECIFICATION IID SP-W375LV-M0001	Liquidus	< 950 ^{o}C		
ILAW WFQ	Subjected to LAW Centerline Canister Cooling	Report amount of crystals		
2.2.2.17.1	Leachability Index per ANSI/ANS-16.1	>6.0 after 90 days		
2.2.2.17.2	PCT per ASTM C1285-98. 7 days at 90°C B (g/m^2) Na (g/m^2) Si (g/m^2)	< 2.0 g/m^2		
2.2.2.17.3	VHT at 200 ^{o}C (g/m^2/day) for seven days	< 50 g/m^2/day		
2.2.2.18	Compressive strength	> 3.45 MPa		
2.2.2.19	Compressive strength after Thermal, Radiation, Biodegradation and Immersion per ANSI/ANS 16.1	> 75% of 3.45 MPa		
2.2.2.20	ILAW package acceptable for land disposal under State of Washington WAC 173-303 and RCRA LDR Biotoxicity	Meets UTS limits		
LAW SPECIFICATION IID SP-W375LV-M0001**	Viscosity Data (poise) at 1100^{o}C	10 to 150 P		
LAW SPECIFICATION IID SP-W375LV-M0001**	Conductivity (S/cm) at 1100^{o}C	0.2 to 0.7 S/cm		
ILAW WFQ	T_G (onset) Package Storage	Required for canister cooling modeling		

* For LAW AZ-102 this requirement was changed to "reduced waste loading as necessary to avoid excessive K-3 corrosion and other negative effects caused by the high sulfate to sodium ratio" via WTP Test Exception 24590-LAW-TEF-RT-02-002 dated 10/21/02.

** Since the present work was completed, this reference was superceded by*: Clark, K. 2003. Engineering Specification for Low Activity Waste Melters, 24590-LAW-3PS-AE00-T0001, River Protection Project – Waste Treatment Plant, Richland, Washington, 99352,*which revises the lower conductivity limit to 0.1 S/cm.

Table 2. Composition of AP-101 Waste as Reported in Reference [4][1].

Waste Constituents	AP-101 as given in [4] 5.62 M Na		Constituents in Glass	Wt.%
	Moles/l	mg/l	Oxides	-
Al	0.257	6940	Al_2O_3	5.49
B	0.001	15	B_2O_3	0.02
Ba	0.000	0.33	-	-
Ca	0.000	7.7	CaO	-
Cd	0.000	2	-	-
Cr	0.003	143	Cr_2O_3	0.09
Cs	0.000	5.1	-	-
Cu	0.000	1.6	-	-
Fe	0.000	2.5	Fe_2O_3	-
K	0.798	31200	K_2O	15.72
Mo	0.000	11	-	-
Na	5.624	129300	Na_2O	74.79
Ni	0.000	7.9	NiO	-
Pb	0.000	15	PbO	-
Si	0.005	137	SiO_2	0.12
Zn	0.000	5.6	-	-
Zr	0.000	1.4	-	-
Cl	0.056	1980	Cl	0.83
F	0.153	2900	F	1.21
PO_4	0.011	1020	P_2O_5	0.32
SO_4	0.042	4030	SO_3	1.41
NO_2	0.911	41900	-	-
NO_3	2.129	132000	-	-
CO_3	0.538	6460	-	-
OH	2.429	41300	-	-
Organic Carbon	0.163	1960	-	-
SUM	-	-	-	100.00

[1] Li, Mg, Mn, Sr, and Ti were reported as less-than numbers and are not included.

Table 3. Waste Contribution in Glass for AP-101 LAW for the Three Waste Loadings Tested, Including Contribution of Sodium from Pretreatment.

Glass Oxides	AP-101 waste contribution to glass (wt%)	AP-101waste contribution including sodium addition in pretreatment (wt%)	AP-101 waste contribution to glass (wt%)	AP-101waste contribution including sodium addition in pretreatment (wt%)	AP-101 waste contribution to glass (wt%)	AP-101waste contribution including sodium addition in pretreatment (wt%)
Waste Loading	26.42%	26.92%	24.39 %	24.85%	21.54%	21.95%
Al_2O_3	1.47	1.47	1.35	1.35	1.20	1.20
B_2O_3	0.01	0.01	0.01	0.01	0.00	0.00
Cr_2O_3	0.02	0.02	0.02	0.02	0.02	0.02
K_2O	4.21	4.21	3.88	3.88	3.43	3.43
Na_2O	19.50	**20.00**	18.00	**18.46**	15.90	**16.31**
SiO_2	0.03	0.03	0.03	0.03	0.03	0.03
Cl	0.22	0.22	0.20	0.20	0.18	0.18
F	0.32	0.32	0.30	0.30	0.27	0.27
P_2O_5	0.09	0.09	0.08	0.08	0.07	0.07
SO_3	0.38	0.38	0.35	0.35	0.31	0.31
SUM	26.42	26.92	24.39	24.85	21.54	21.95

Table 4. Glasses Formulated for AP-101 LAW at 20 and 18.5 wt% Na_2O.

Glass Oxides	LAW AP101 pretreated	**LAWA125** Additives		**LAWA125**	LAW AP101 pretreated	**LAWA126** Additives		**LAWA126 and LAWA126R1** (Repeat)	**LAWA126R3** (No cesium spike)
Loading	26.92%	100.00%	73.08%	26.92%	24.85%	100.00%	75.15%	24.85%	24.68%
Al_2O_3	1.47	5.71	4.17	5.64	1.35	5.71	4.29	5.64	5.65
B_2O_3	0.01	13.07	9.55	9.55	0.01	13.07	9.82	9.82	9.85
CaO	-	2.65	1.94	1.94	-	2.65	1.99	1.99	2.00
Cr_2O_3	0.02	-	-	0.02	0.02	-	-	0.02	0.02
Cs_2O spike	0.18	-	-	0.18	0.16	-	-	0.16	0.00
Fe_2O_3	-	7.38	5.39	5.39	-	7.38	5.54	5.54	5.56
K_2O	4.21	-	-	4.21	3.88	-	-	3.88	3.88
MgO	-	1.97	1.44	1.44	-	1.97	1.48	1.48	1.48
Na_2O	**20.00**	-	-	**20.00**	**18.46**	-	-	**18.46**	**18.46**
SiO_2	0.03	58.67	42.87	42.91	0.03	58.67	44.09	44.12	44.22
TiO_2	-	2.66	1.94	1.94	-	2.66	2.00	2.00	2.00
ZnO	-	3.93	2.88	2.88	-	3.93	2.96	2.96	2.96
ZrO_2	-	3.98	2.91	2.91	-	3.98	2.99	2.99	3.00
Cl	0.22	-	-	0.22	0.20	-	-	0.20	0.20
F	0.32	-	-	0.32	0.30	-	-	0.30	0.30
P_2O_5	0.09	-	-	0.09	0.08	-	-	0.08	0.08
SO_3	0.38	-	-	0.38	0.35	-	-	0.35	0.35
Re_2O_7 spike	-	-	-	0.10	-	-	-	0.10	0.10
SUM	26.92	100.00	73.08	100.10	24.85	100.00	-	100.10	100.10

Note: Two spikes were added: cesium was added in some of the glass formulations at the level suitable for tracking in a melter off-gas system; rhenium was added at a fixed concentration of 0.1 wt% as a substitute for technetium.

Table 5. Glasses Formulated for AP-101 LAW at 16.3 wt% Na_2O.

Glass Oxides	AP-101 pretreated	**LAWA127** Additives		**LAWA127 and LAWA127R1** (repeat)	**LAWA127R2** No cesium spike
Loading	21.95%	100.00%	78.05%	21.95%	21.80%
Al_2O_3	1.20	5.71	4.45	5.65	5.66
B_2O_3	0.00	13.07	10.20	10.20	10.22
CaO	-	2.65	2.07	2.07	2.07
Cr_2O_3	0.02	-	-	0.02	0.02
Cs_2O spike	0.15	-	-	0.15	0.00
Fe_2O_3	-	7.38	5.76	5.76	5.77
K_2O	3.43	-	-	3.43	3.43
MgO	-	1.97	1.54	1.54	1.54
Na_2O	**16.31**	-	-	**16.31**	**16.31**
SiO_2	0.03	58.67	45.79	45.82	45.90
TiO_2	-	2.66	2.07	2.07	2.08
ZnO	-	3.93	3.07	3.07	3.08
ZrO_2	-	3.98	3.11	3.11	3.12
Cl	0.18	-	-	0.18	0.18
F	0.27	-	-	0.27	0.27
P_2O_5	0.07	-	-	0.07	0.07
SO_3	0.31	-	-	0.31	0.31
Re_2O_7 spike	-	-	-	0.10	0.10
SUM	21.95	100.00	-	100.10	100.10

Note: Two spikes were added: cesium was added in some of the glass formulations at the level suitable for tracking in a melter off-gas system; rhenium was added at a fixed concentration of 0.1 wt% as a substitute for technetium.

Table 6. Glass Formulated for AP-101 LAW (wt%) for a Sodium Level of 18.5 wt% Na_2O: Varying Additives.

Glass Oxides	AP-101 pretreated	**LAWA128** Additives		**LAWA128 and LAWA128R1** (repeat)	Comparison to LAWA126	**LAWA129** Additives		**LAWA129 and LAWA129R1** (repeat)	Comparison to LAWA126
Loading	24.85%	100.00%	75.15%	24.85%	-	100.00%	75.15%	24.85%	-
Al_2O_3	1.35	6.22	4.67	6.03	0.38	8.14	6.12	7.47	1.83
B_2O_3	0.01	9.40	7.07	7.07	-2.75	11.33	8.52	8.52	-1.30
CaO	-	2.77	2.08	2.08	0.09	4.69	3.53	3.53	1.54
Cr_2O_3	0.02	-	-	0.02	-	-	-	0.02	-
Cs_2O spike	0.16	-	-	0.16	-	-	-	0.16	-
Fe_2O_3	-	7.71	5.79	5.79	0.25	0.00	0.00	0.00	-5.54
K_2O	3.88	-	-	3.88	-	-	-	3.88	-
MgO	-	1.57	1.18	1.18	-0.30	1.57	1.18	1.18	-0.30
Na_2O	**18.46**	-	-	**18.46**	-	-	-	**18.46**	-
SiO_2	0.03	61.29	46.06	46.09	1.98	63.22	47.51	47.54	3.42
TiO_2	-	2.77	2.09	2.09	0.09	2.77	2.09	2.09	0.09
ZnO	-	4.11	3.09	3.09	0.13	4.11	3.09	3.09	0.13
ZrO_2	-	4.16	3.13	3.13	0.13	4.16	3.13	3.13	0.13
Cl	0.20	-	-	0.20	-	-	-	0.20	-
F	0.30	-	-	0.30	-	-	-	0.30	-
P_2O_5	0.08	-	-	0.08	-	-	-	0.08	-
SO_3	0.35	-	-	0.35	-	-	-	0.35	-
Re_2O_7 spike	-	-	-	0.10	-	-	-	0.10	-
SUM	24.85	100.00	-	100.10	-	100.00	-	100.10	-

Note: Two spikes were added: cesium was added in some of the glass formulations at the level suitable for tracking in a melter off-gas system; rhenium was added at a fixed concentration of 0.1 wt% as a substitute for technetium.

Table 7. Preparation and Testing of Glasses Made for AP-101 LAW Actual Sample.

Sample ID		Preparation Summary	Comments
LAWA125	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Amber brown homogenous glass. Used for viscosity, electrical conductivity, PCT, VHT.
	LAWA125C85H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 850°C and quenched.	Medium yellow-brown homogenous glass.
LAWA126	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Amber brown homogenous glass. Used for viscosity, electrical conductivity, density, PCT, VHT, refractory corrosion.
	LAWA126C85H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 850°C and quenched.	Medium brown homogenous glass.
	LAWA126VHT	-	Sample of LAWA126 analyzed and used for VHT.
LAWA126R1	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Replicate of LAWA126.
LAWA126R3	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	No cesium spike. Amber brown homogenous glass. Used for glass transition temperature, VHT, refractory corrosion.
	LAWA126R3C85H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 850°C and quenched.	Medium brown homogenous glass.
	LAWA126R3C70H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 700°C and quenched.	Light brown glass, moderately fractured.
PNLA126CC (LAWA126 prepared from slurry feed)		A feed slurry was prepared from waste simulant (Appendix A1) and glass formers (Appendix A2) and placed in a Pt/Au crucible in a 110°C oven to dry, then heated to 1150°C for four hours. The furnace temperature was reduced to 1020°C, and the sample was subjected to the container cooling profile.	Reddish brown translucent homogeneous, with bubbles on the bottom of the glass. Used for density, PCT, VHT, TCLP (PNLA126CCTC).
LAWA127R1	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Amber brown homogenous glass. Used for viscosity, electrical conductivity, PCT, VHT.
	LAWA127C85H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 850°C and quenched.	Medium brown glass – highly fractured.

Table 7. Preparation and Testing of Glasses Made for AP-101 LAW Actual Sample (continued).

LAWA127R2	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Dark brown homogenous glass. Used for glass transition temperature, PCT.
	LAWA127R2C85H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 850°C and quenched.	No cesium spike. Medium brown glass, moderately fractured.
	LAWA127R2C70H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 700°C and quenched.	Medium brown glass, moderately fractured.
LAWA128	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Amber brown homogenous glass. Used for PCT.
	LAWA128C85H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 850°C and quenched.	Medium dark brown homogenous glass.
LAWA128R1	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Replicate of LAWA128. Used for viscosity, electrical conductivity.
LAWA129	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Used for PCT.
LAWA129R1	As melted	Dry batch melted at 1200°C for 75 minutes in Pt/5%Au crucible, stirred and quenched.	Replicate of LAWA129. Green homogenous glass.
	LAWA129R1C85H20	Previous glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 850°C and quenched.	Clear light green homogenous glass. Used for viscosity, electrical conductivity, VHT.

Table 8. XRF Analyses of Glasses Formulated for AP-101 LAW (wt%).

Oxide	LAWA125		LAWA126		LAWA126R1		LAWA126R3		LAWA126VHT#		PNLA126CC	
	Target	XRF	Target	XRF	Target	XRF	Target	XRF	Target	XRF	Target	XRF
Al_2O_3	5.64	5.35	5.64	5.58	5.64	5.67	5.65	5.72	5.64	5.85	5.65	5.69
B_2O_3*	9.55	9.55	9.82	9.82	9.82	9.82	9.82	9.82	9.82	9.82	9.82	9.82
CaO	1.94	2.07	1.99	2.03	1.99	2.10	2.00	2.01	1.99	1.96	2.00	1.96
Cl	0.22	0.19	0.20	0.17	0.20	0.15	0.20	0.20	0.20	0.09	0.20	0.12
Cr_2O_3	0.02	0.03	0.02	0.02	0.02	0.03	0.02	0.03	0.02	0.02	0.02	0.04
Cs_2O	0.18	0.18	0.16	0.16	0.16	0.16	0.00	0.00	0.16	0.13	0.00	0.00
F	0.32	N.A	0.30	N.A	0.30	N.A	0.30	N.A	0.30	N.A	0.30	N.A
Fe_2O_3	5.39	5.95	5.54	5.83	5.54	6.05	5.56	5.77	5.54	5.45	5.56	5.74
K_2O	4.21	3.91	3.88	3.84	3.88	3.53	3.88	3.48	3.88	3.61	3.88	3.60
MgO	1.44	1.32	1.48	1.39	1.48	1.32	1.48	1.37	1.48	1.39	1.48	1.53
Na_2O	20.00	18.53	18.46	17.71	18.46	17.04	18.46	17.51	18.46	18.28	18.46	17.93
P_2O_5	0.09	0.10	0.08	0.10	0.08	0.10	0.08	0.10	0.08	0.11	0.08	0.10
Re_2O_7	0.10	0.05	0.10	0.06	0.10	0.06	0.10	0.07	0.10	0.02	0.00	0.00
SiO_2	42.91	44.30	44.12	45.29	44.12	45.49	44.22	45.76	44.12	45.91	44.21	45.33
SO_3	0.38	0.31	0.35	0.31	0.35	0.31	0.35	0.33	0.35	0.27	0.35	0.29
TiO_2	1.94	2.13	2.00	2.05	2.00	2.12	2.00	2.03	2.00	1.94	2.00	2.25
ZnO	2.88	3.03	2.96	2.87	2.96	3.04	2.96	2.88	2.96	2.66	2.96	2.92
ZrO_2	2.91	3.65	2.99	3.42	2.99	3.69	3.00	3.44	2.99	3.14	3.00	3.31
Sum	100.10	100.75	100.10	100.75	100.10	100.78	100.10	100.77	100.10	100.74	100.00	100.64

*Boron was not measured; target values shown. # LAWA126 sample used for VHT. N.A. = Not Analyzed

Table 8. XRF Analyses of Glasses Formulated for AP-101 LAW (wt%) (continued).

Oxide	LAWA127R1		LAWA127R2		LAWA128		LAWA128R1		LAWA129		LAWA129R1	
	Target	XRF	Target	XRF	Target	XRF	Target	XRF	Target	XRF	Target	XRF
Al_2O_3	5.65	5.45	5.66	5.48	6.03	5.75	6.03	5.83	7.47	7.20	7.47	7.23
B_2O_3	10.20	10.02	10.22	10.22	7.07	7.07	7.07	7.07	8.52	8.52	8.52	8.52
CaO	2.07	2.13	2.07	2.14	2.08	2.13	2.08	2.20	3.53	3.64	3.53	3.58
Cl	0.18	0.11	0.18	0.13	0.20	0.15	0.20	0.18	0.20	0.17	0.20	0.17
Cr_2O_3	0.02	0.02	0.02	0.02	0.02	0.03	0.02	0.03	0.02	0.02	0.02	0.02
Cs_2O	0.15	0.12	0.00	0.000	0.16	0.15	0.16	0.12	0.16	0.16	0.16	0.14
F	0.27	N.A	0.27	N.A	0.30	N.A	0.30	N.A	0.30	N.A	0.30	N.A
Fe_2O_3	5.76	6.27	5.77	5.79	5.79	6.14	5.79	6.39	0.00	0.10	0.00	0.11
K_2O	3.43	3.10	3.43	3.09	3.88	3.84	3.88	3.60	3.88	3.88	3.88	3.53
MgO	1.54	1.38	1.54	1.51	1.18	1.06	1.18	1.08	1.18	1.09	1.18	1.07
Na_2O	16.31	15.87	16.31	16.32	18.46	18.35	18.46	17.35	18.46	18.16	18.46	18.46
P_2O_5	0.07	0.08	0.07	0.08	0.08	0.09	0.08	0.09	0.08	0.10	0.08	0.09
Re_2O_7	0.10	0.07	0.10	0.04	0.10	0.06	0.10	0.05	0.10	0.05	0.10	0.05
SiO_2	45.82	46.72	45.90	46.62	46.09	46.51	46.09	47.03	47.54	48.16	47.54	48.50
SO_3	0.31	0.18	0.31	0.21	0.35	0.30	0.35	0.32	0.35	0.31	0.35	0.31
TiO_2	2.07	2.31	2.08	2.26	2.09	2.18	2.09	2.28	2.09	2.24	2.09	2.23
ZnO	3.07	3.04	3.08	3.04	3.09	3.09	3.09	3.23	3.09	3.13	3.09	3.11
ZrO_2	3.11	3.72	3.12	3.67	3.13	3.62	3.13	3.66	3.13	3.71	3.13	3.56
Sum	100.10	100.72	100.10	100.70	100.10	100.58	100.10	100.60	100.10	100.80	100.10	100.68

*Boron was not measured; target values shown. N.A. = Not Analyzed

Table 9. Viscosity and Conductivity Data for Glasses Formulated for LAW AP-101.

Glass	LAWA125	LAWA126	LAWA127R1	LAWA128R1	LAWA129R1
Viscosity (poise)					
900ºC	866	1558	2852	3031	3026
950ºC	406	696	1153	1256	1316
1000ºC	208	343	526	581	631
1050ºC	115	182	265	295	329
1100ºC	68	104	145	161	183
1150ºC	42	63	85	94	108
1200ºC	27	40	52	58	67
1250ºC	18	26	34	37	44
Conductivity (S/cm)					
950ºC	0.199	0.170	0.131	0.169	0.129
1000ºC	0.253	0.215	0.165	0.214	0.167
1050ºC	0.314	0.267	0.203	0.267	0.211
1100ºC	0.385	0.327	0.246	0.326	0.263
1150ºC	0.464	0.395	0.295	0.393	0.323
1200ºC	0.553	0.471	0.349	0.468	0.391
1250ºC	0.651	0.555	0.407	0.551	0.467

Table 10. Density of Glasses Formulated for LAW AP-101 Actual Sample.

Glass Name	Density (g/cc at 20°C)
LAWA126 Measured on dry batch LAWA126	2.687
LAWA126 Measured on PNLA126CC	2.679
NIST Soda Lime Glass #1826b Standard density = 2.549 at 20°C	2.550

Table 11. Glass Transition Temperatures Measured by Differential Thermal Analysis.

Glass Name	T_G (ºC)
LAWA126R3	496
LAWA127R2	508

Table 12. Results from the PCT Procedure (7 Days, 90°C).

	LAWA125	LAWA126	PNLA126CC	LAWA127R1	LAWA127R2	LAWA128	LAWA129
Concentration (mg/l)							
B	57.18	36.47	27.40	21.13	23.17	13.80	14.30
Na	239.90	143.50	107.50	82.72	85.36	118.9	101.70
Si	88.74	68.28	64.91	58.43	62.08	75.55	62.28
Normalized Concentrations (g/l)							
B	1.927	1.196	0.896	0.667	0.730	0.628	0.541
Na	1.617	1.048	0.785	0.684	0.706	0.868	0.743
Si	0.442	0.331	0.314	0.273	0.289	0.351	0.280
pH	11.21	10.74	10.56	10.46	10.33	11.03	10.85
Normalized Mass Loss (g/m^2)							
B	0.964	0.598	0.448	0.334	0.365	0.314	0.270
Na	0.808	0.524	0.392	0.342	0.353	0.434	0.371
Si	0.221	0.166	0.157	0.136	0.145	0.175	0.140
Normalized Loss Rate ($g/d/m^2$)							
B	0.138	0.085	0.064	0.048	0.052	0.045	0.039
Na	0.115	0.075	0.056	0.049	0.050	0.062	0.053
Si	0.032	0.024	0.022	0.019	0.021	0.025	0.020

Table 13. Results from Vapor Hydration Tests (VHT) at 200°C for 7 Days and 24- 25 Days (g/m^2/day).

Glass Name	Alteration depth (µm)	Days	Rate (g/m^2/d) Calculated for the measured density of 2.68 g/cc for LAWA126 and average density of 2.655 g/cc for other samples	Comparison to limit of 50 g/m^2/d
Contract limit [15]	-	-	50	-
LAWA125	343	24	37.9	76%
LAWA126	22	24	2.5	5%
LAWA126R3 glass not annealed	6	7	2.3	5%
LAWA126R3 glass not annealed	22	24	2.5	5%
PNLA126CC	3	7	1.1	2%
PNLA126CC	13	25	1.4	3%
LAWA127R2	4	24	0.4	1%
LAWA128	8	24	0.9	2%
LAWA129R1	21.9	24	2.4	5%

Table 14. Results of K3 Refractory Corrosion Testing.

Glass name	Neck Corrosion (inches)	Half-Down Corrosion (inches)	Penetration Depth (inches)
LAWA126	0.01	NA*	0.02
LAWA126R3	0.02	0.00	0.02
WVDP Ref 6[a]	0.01	0.00	0.03
LAWA102[b]	0.02	0.00	0.02
LAWB45[c]	0.01	0.00	0.03

* Some coupon swelling is evident.

a Simulant sample of glass composition used for waste treatment at West Valley

b LAW Sub-Envelope A3 glass successfully tested at the LAW Pilot Melter

c LAW Sub-Envelope B1 glass successfully tested at the LAW Pilot Melter

Table 15. Results (mg/l) from TCLP Testing (Performed at VSL; Analysis by DCP).

Sample ID	UTS Limit	Method Blank EX1T110502TC	Glass PNLA126CCTC
Ag	**0.14**	**<0.003**	**<0.003**
As	**5.00**	**<0.05**	**<0.05**
Ba	**21.00**	**<0.002**	**<0.7***
Cd	**0.11**	**<0.003**	**<0.003**
Cr	**0.60**	**<0.006**	**0.007**
Ni	**11.00**	**<0.008**	**<0.008**
Pb	**0.75**	**<0.024**	**<0.024**
Se	**5.70**	**<0.053**	**<0.053**
Zn	**4.30**	**<0.003**	**1.64**
Al	-	<0.005	0.432
B	-	<0.026	2.062
Ca	-	<0.003	1.126
Fe	-	<0.007	0.414
K	-	<0.014	2.405
Li	-	<0.023	<0.023
Mg	-	<0.003	0.572
Mn	-	<0.007	0.011
P	-	<0.074	<0.074
Si	-	<0.01	2.69
Sr	-	<0.001	0.011
Ti	-	<0.002	0.082
Zr	-	<0.005	0.133
pH	-	4.93	4.94

* Ba concentration in glass TCLP extracts is relatively high due to a suspected interference.

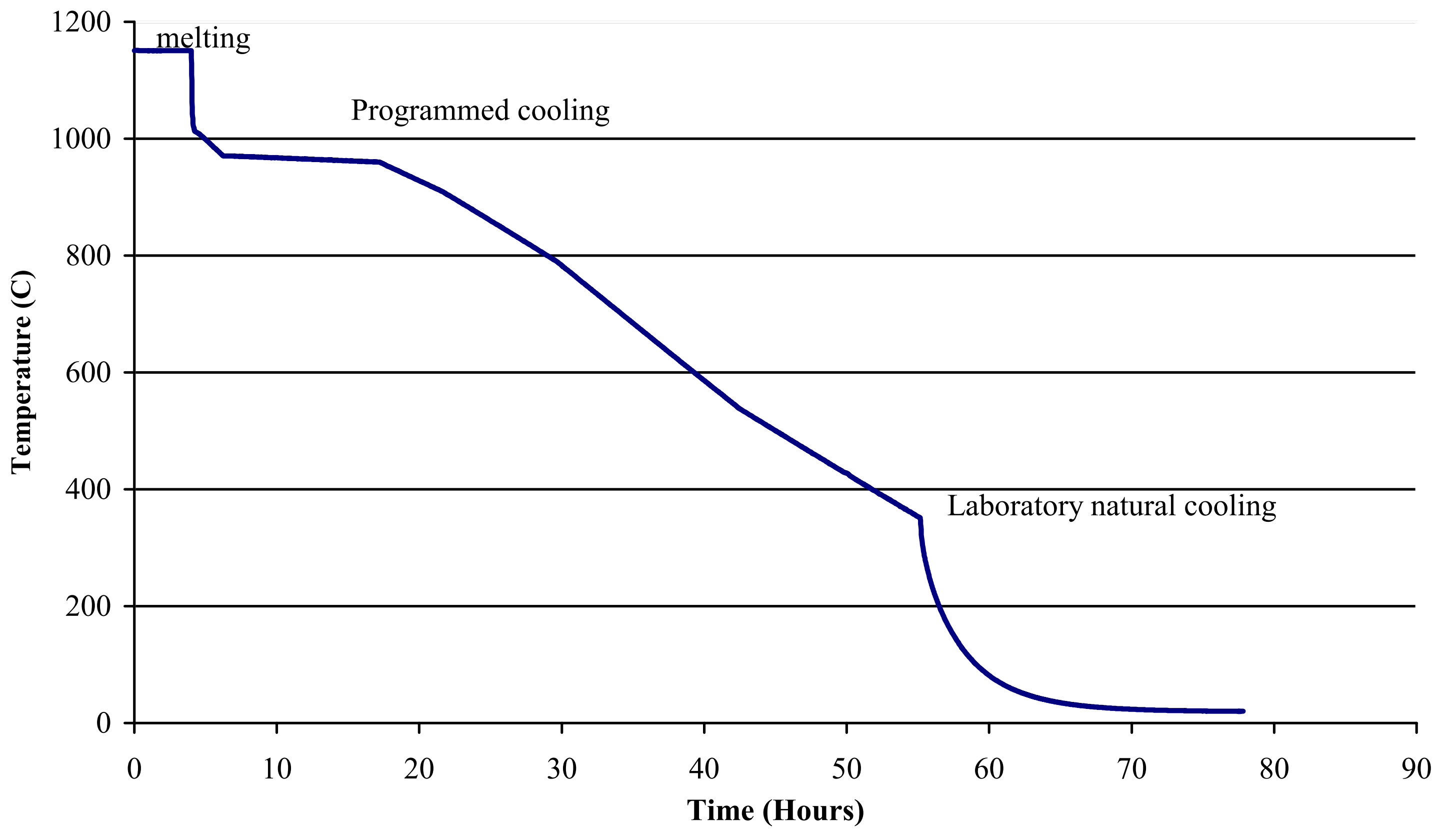


Figure 1. Centerline Container Cooling Curve Used for Heat Treatment in This Work.

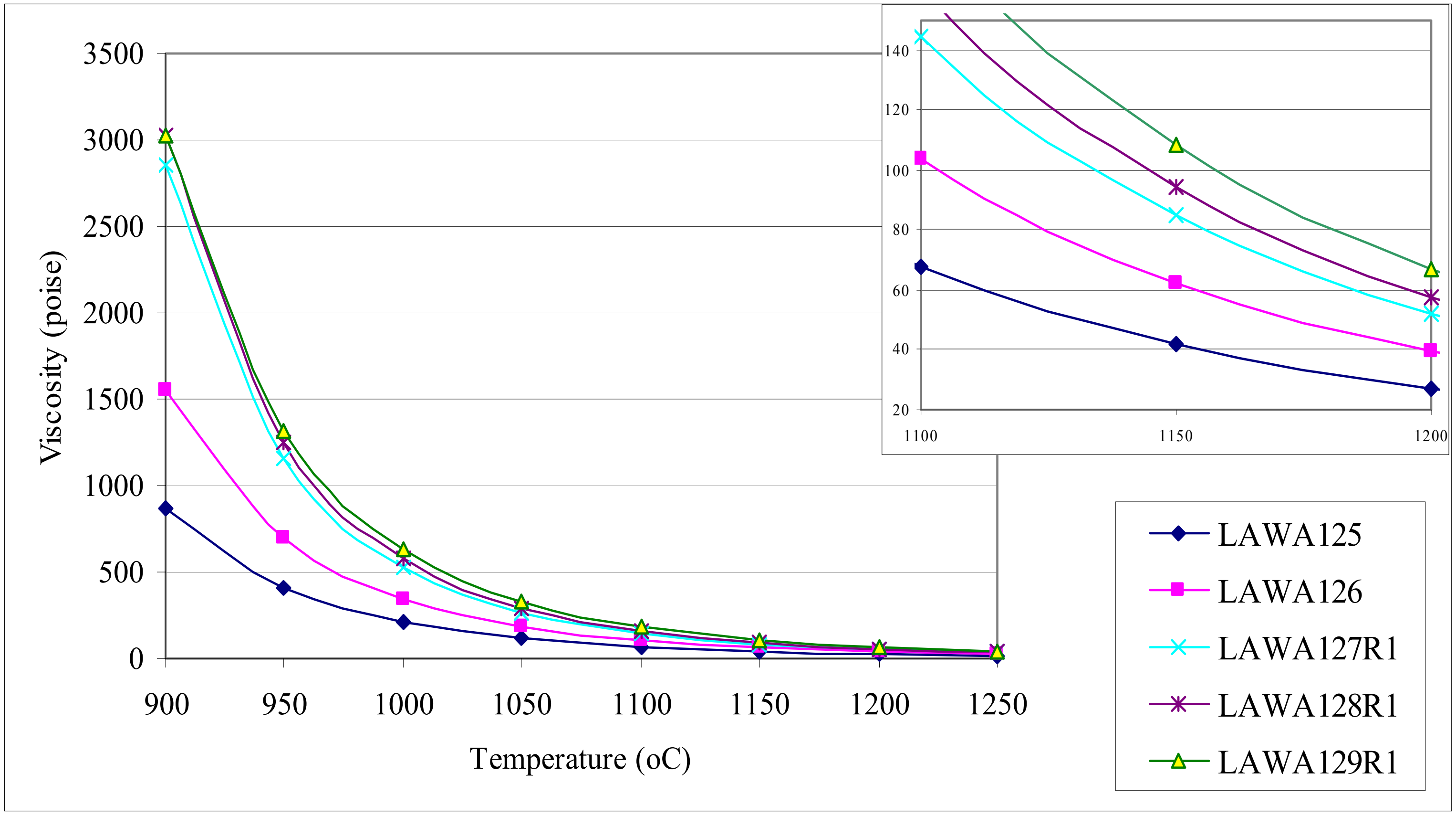


Figure 2. Melt Viscosity as a Function of Temperature for the Five Formulations Tested.

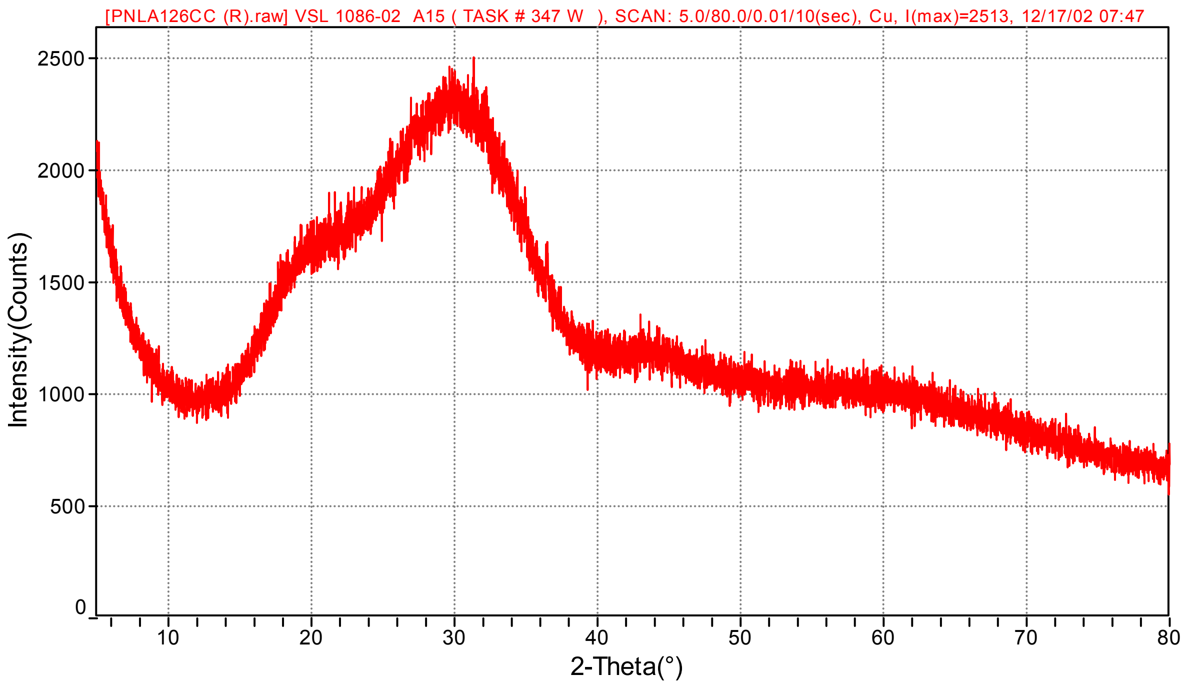


Figure 3. XRD Plot of LAWA126 Glass Prepared from Slurry Feed (PNLA126CC) and Heat Treated According to the LAW Container Cooling Profile, Indicating Absence of Crystalline Phases.

Appendix A1. Recipe Developed at VSL for 1 Liter of AP-101 Simulant Using PNNL-Supplied Analytical Data [4] and Applying a Sodium Addition of 2.5% in Pretreatment.

Envelope Constituents	AP-101 as given in PNNL 5.62 M Na		Simulant AP-101 including pretreatment		GLASS Oxides	AP-101 Wt% pretreated	AP101 wt% in glass pretreated	Source in Simulant	Order for Addition	Formula Weight	Assay[3]	Target Weight (g)
Loading	mg/L	Moles/L	mg/L	Molarity	Loading	100 %	24.68%	In 512 ml water add following compounds in the order listed.				
Al	6940	0.257	9,625	0.357	Al2O3	5.486	1.354	Al(NO3)3.9H2O, 60% sol	5	375.14	0.6069	220.51
B	15	0.001	21	0.002	B2O3	0.020	0.005	H3BO3	8	61.83	0.99	0.12
Ca	7.7	0.000	11	0.000	CaO	-	-	-	-	-	-	-
Cr	143	0.003	198	0.004	Cr2O3	0.087	0.022	Na2CrO4*4H2O	3	234.04	0.985	0.91
Cs	5.1	0.000	7	0.000	Cs2O	0.002	0.001	CsNO3	6	194.91	0.998	0.01
Fe	2.5	0.000	3	0.000	Fe2O3	-	-	-	-	-	-	-
K	31200	0.798	43,270	1.107	K2O	15.724	3.882	KOH	2	56.098	0.908	68.37
Na	129300	5.624	183,920	8.000	Na2O	74.791	18.462	NaOH, 50% sol. d=1.53	1	40	0.5013	220.06
Si	137	0.005	190	0.007	SiO2	0.123	0.030	SiO2	7	60.09	0.99	0.41
Cl	1980	0.056	2,746	0.077	Cl	0.828	0.204	NaCl	9	58.45	0.99	4.57
F	2900	0.153	4,022	0.212	F	1.213	0.300	NaF	10	42	0.99	8.98
PO4	1020	0.011	1,415	0.015	P2O5	0.319	0.079	Na3PO4.12H2O	4	380.12	0.99	5.72
SO4	4030	0.042	5,589	0.058	SO3	1.405	0.347	Na2SO4	11	142.06	0.99	8.35
NO2	41900	0.911	58,110	1.263	NO2	-	-	NaNO2	16	69	0.995	87.60
NO3	132000	2.129	183,067	2.953	NO3	-	-	NaNO3	17	84.99	0.99	161.60
CO3	32300 [2]	0.538	44,830	0.747	CO3	-	-	Na2CO3	18	105.99	1	79.13
OH	41300	2.429	57,278	3.369	OH	-	-	KOH+NaOH	-	-	-	-
Org.Carbon	1960	0.163	2,718	0.227	C as:	-	-		-	-	-	-
Acetate	1640	0.028	2502 [1]	0.042	Acetate	-	-	Sodium Acetate (C2)	11	136.08	0.99	5.82
Formate	1200	0.027	1831 [1]	0.041	Formate	-	-	Sodium Formate (C1)	12	68.01	0.99	2.79
Oxalate	1800	0.020	2746 [1]	0.031	Oxalate	-	-	Sodium Oxalate (C2)	13	134	0.99	4.22
Citric	<890	<0.006	1234	0.006	Citric	-	-	Citric Acid (C6)	14	192.12	0.99	1.25
-	-	-	-	-	-	-	-	Target Glass				1342.87
-	-	-	-	-	SUM	100	24.685	Total simulant wt.				1392.44

(1) Reported concentrations for acetate, formate and oxalate [4] were increased by 10% in order to match the TOC concentration of 0.227 Moles/L.
(2) Reference [4] reports only TIC at 6460 µg/mL from which this carbonate concentration was calculated.
(3) Assay provides the purity of the raw material specified by the vendor.

Appendix A2. Recipe for Feed LAWA126 Used in Feed Testing and Simulant Glasses (and Previous AP-101 Simulant A1).

Envelope Constituents	LAWA126 for this AP101	LAWA126 for AP101 Additives	Additives	Source inAdditives	Assay[1]	Ratio[2]	Target Weight (g) Additives	Other oxides present	
Loading	24.68 %	100 %	75.32 %					% Fe2O3	%SiO2
Al_2O_3	5.651	5.706	4.297	Kyanite (Al2SiO5) 325 Mesh	0.99	0.54	107.93	0.0116	0.437
B_2O_3	9.844	13.065	9.839	H3BO3	0.986	0.563	238.01		
CaO	1.995	2.649	1.995	Wollanstonite NYAD 325 Mesh	0.993	0.475	56.80	0.004	0.51
Cr_2O_3	0.022	-	-	-	-	-	-	-	-
Cs_2O	0.001	-	-	-	-	-	-	-	-
Fe_2O_3	5.555	7.376	5.555	Fe2O3 (Prince manufacturing)	0.998	1	69.84	-	-
K_2O	3.882	-	-	-	-	-	-	-	-
MgO	1.481	1.967	1.481	Olivine (Mg2SiO4) 325 Mesh (#180)	0.99	0.48	41.85	0.0768	0.4252
Na_2O	18.462	-	-	-	-	-	-	-	-
SiO_2	44.210	58.666	44.180	SiO2 (Sil-co-Sil 75)	0.997	1	480.81	-	-
TiO_2	1.999	2.654	1.999	Rutile Sand Premium Airfloated	0.954	1	28.14	0.0071	0.0091
ZnO	2.963	3.935	2.963	ZnO	0.997	1	39.91	-	-
ZrO_2	2.999	3.982	2.999	Zircon ZrSiO4 (Flour) Mesh 325	0.99	0.673	60.49	-	0.326
Cl	0.204	-	-	Sugar	-	-	83.66	-	-
F	0.300	-	-	+Na2SO4 (from recycled off-gas) g	-	-	2.09	-	-
PO_4	0.079	-	-	Simulant weight (simulant AP101)	-	-	1392.44	g for vol. 1 (L)	-
SO_4	0.347	-	-	Expected Glass yield	-	-	1342.87	g	-
-	-	-	-	Sum of Additives (g)	-	-	1123.78	g	-
SUM	100	100	75.307	Sum of complete batch (g)	-	-	2516.21	g	-
-	-	-	-	density measured	-	-	1.76	g/ml	-
-	-	-	-	Final Volume (L, for above density)	-	-	1.43	L	-

(1) Assay provides the purity of the raw material specified by the vendor.
(2) Ratio provides the factor to convert the glass former additive into the corresponding oxide in the glass

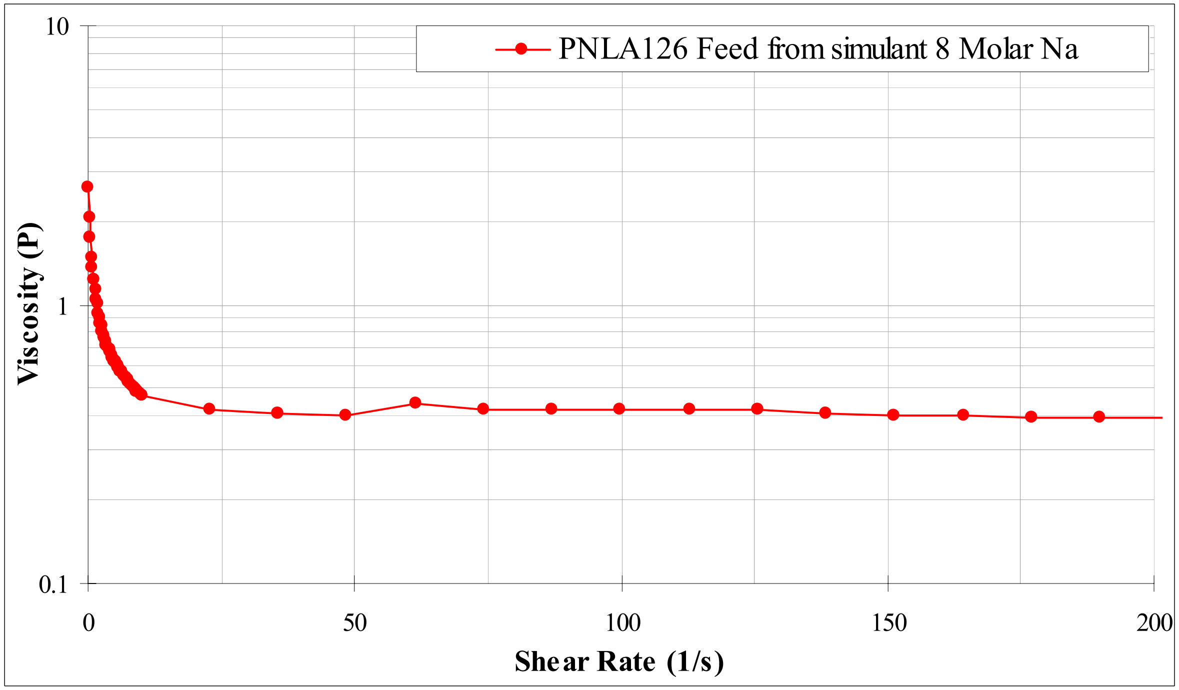
Appendix A3. Batching Recipe for Feed LAWA126 Submitted for Actual Waste Testing

(Various dilutions may be tested; the recipe is based on a total number of sodium moles, here for 8 moles, or 1 liter of 8 molar concentrate).

Envelope Constituents	AP-101 as given in PNNL 5.62 M Na		GLASS Oxides	AP-101 Wt% pretreated	AP101 wt% in glass pretreated	LAWA126 for this AP101	LAWA126 for AP101 Additives	Additives	Source in Additives	Assay [(1)]	Ratio [(2)]	Target Weight (g)	Other oxides present	
Loading	Moles/L	mg/L ICP	Loading	100 %	24.70%	24.70%	100.00%	75.30%	-	-	-	Additives	% Fe2O3	%SiO2
Al	0.257	6,940	Al2O3	5.483	1.354	5.651	5.71	4.30	Kyanite (Al2SiO5) 325 Mesh	0.99	0.54	107.90	1.16%	43.70%
As	0.000	1	As2O3	0.001	0.000	0.000	-	-	-	-	-	-	-	-
B	0.001	15	B2O3	0.020	0.005	9.844	13.07	9.84	H3BO3	0.986	0.563	237.94		
Be	0.000	1	BeO	0.002	0.000	0.000								
Ca	0.000	8	CaO	0.005	0.001	1.995	2.65	1.99	Wollanstonite NYAD 325 Mesh	0.993	0.475	56.75	0.40%	51.00%
Cd	0.000	2	CdO	0.001	0.000	0.000	-	-	-	-	-	-	-	-
Cr	0.003	143	Cr2O3	0.087	0.022	0.022	-	-	-	-	-	-	-	-
Cs	0.000	5	Cs2O	0.002	0.001	0.001	-	-	-	-	-	-	-	-
Cu	0.000	2	CuO	0.001	0.000	0.000	-	-	-	-	-	-	-	-
Fe	0.000	3	Fe2O3	0.001	0.000	5.555	7.38	5.55	Fe2O3	0.998	1	69.81		
K	0.798	31,200	K2O	15.715	3.881	3.881	-	-	-	-	-	-	-	-
Mg	-	-	MgO	-	-	1.481	1.97	1.48	Olivine (Mg2SiO4) 325 Mesh (#180)	0.99	0.48	41.84	7.68%	42.52%
Mo	0.000	15	MoO3	0.009	0.002	0.002	-	-	-	-	-	-	-	-
Na	5.624	129,300	Na2O	74.747	18.462	18.462	-	-	-	-	-	-	-	-
Ni	0.000	8	NiO	0.004	0.001	0.001	-	-	-	-	-	-	-	-
Pb	0.000	15	PbO	0.007	0.002	0.002	-	-	-	-	-	-	-	-
Si	0.005	137	SiO2	0.123	0.030	44.206	58.67	44.18	SiO2 (Sil-co-Sil 75)	0.997	1	480.63		
Ti	-	-	TiO2	-	-	1.999	2.66	2.00	Rutile Sand Premium Airfloated	0.954	1	28.13	0.71%	0.91%
U	0.000	51	UO2	0.024	0.006	0.006	-	-	-	-	-	-	-	-
Zn	0.000	6	ZnO	0.003	0.001	2.963	3.93	2.96	ZnO	0.997	1	39.89	-	-
Zr	0.000	1	ZrO2	0.001	0.000	2.999	3.98	3.00	Zircon ZrSiO4 (Flour) Mesh 325	0.99	0.67	60.47		32.60%
Cl	0.056	1,980	Cl	0.828	0.204	0.204	-	-	Sugar			85.78		
F	0.153	2,900	F	1.213	0.299	0.299	-	-	-	-	-	-	-	-
PO4	0.011	1,020	P2O5	0.319	0.079	0.079	-	-	-	-	-	-	-	-
SO4	0.042	4,030	SO3	1.404	0.347	0.347	-	-	-	-	-	-	-	-
NO2	0.911	41,900	NO2	-	-	-	-	-	Sum of Additives (g)	-	-	1123.37	g	-
NO3	2.129	132,000	NO3	-	-	-	-	-		-	-			-
CO3 (TIC)	0.538	6,460	CO3	-	-	-	-	-	Expected Glass yield (g)	-	-	1342.47	g	-
NH3	0.000	2	NH3	-	-	-	-	-	-	-	-	-	-	-
OH	2.429	41,300	OH	-	-	-	-	-	Number of moles of sodium used for this batch:			8	Moles	-
TIC	0.538	6,460		-	-	-	-	-	-	-	-	-	-	-
Org.Carbon	0.163	1,960		-	-	-	-	-	-	-	-	-	-	-
-	-	-	SUM	100.00	24.70	100.00	100.00	75.30	-	-	-	-	-	-

[(1)] Assay provides the vendor's specified purity
[(2)] Ratio provides the factor to convert the glass former additive into the corresponding oxide in the glass

Appendix B1. Rheology (Viscosity vs. Shear Rate) of Slurry Feed for LAWA126 Measured at 25°C.



Appendix B2. Rheology Data (Viscosity vs. Shear Rate) of Slurry Feed for LAWA126 Measured at 25°C.

VISCOSITY [P]	SHEAR RATE [1/s]	VISCOSITY [P]	SHEAR RATE [1/s]
2.637	0.123	0.469	9.992
2.041	0.328	0.422	22.85
1.731	0.519	0.404	35.71
1.481	0.730	0.398	48.56
1.347	0.932	0.441	61.42
1.237	1.123	0.422	74.28
1.134	1.334	0.422	87.14
1.044	1.539	0.420	99.99
1.005	1.731	0.421	112.9
0.929	1.938	0.419	125.7
0.899	2.147	0.404	138.6
0.851	2.335	0.401	151.4
0.840	2.543	0.399	164.3
0.802	2.736	0.396	177.1
0.776	2.941	0.395	190.0
0.764	3.148	0.393	202.9
0.740	3.343	0.392	215.7
0.717	3.548	0.391	228.6
0.698	3.754	0.390	241.4
0.685	3.944	0.390	254.3
0.673	4.154	0.389	267.1
0.661	4.360	0.389	280.0
0.648	4.551	0.389	292.9
0.636	4.755	0.389	305.7
0.629	4.960	0.389	318.6
0.623	5.157	0.390	331.4
0.616	5.360	0.391	344.3
0.602	5.568	0.392	357.1
0.591	5.760	0.394	370.0
0.588	5.966	0.396	382.9
0.574	6.171	0.398	395.7
0.572	6.366	0.400	408.6
0.561	6.572	0.402	421.4
0.558	6.777	0.405	434.3
0.547	6.968	0.407	447.1
0.545	7.175	0.410	460.0
0.538	7.379	0.413	472.8
0.529	7.572	0.417	485.7
0.522	7.783	0.420	498.6
0.520	7.976	0.424	511.4
0.515	8.183	0.428	524.3
0.510	8.384	0.432	537.1
0.505	8.579	0.443	550.0
0.500	8.786	0.446	562.9
0.496	8.990	0.448	575.7
0.490	9.183	0.450	588.6
0.484	9.390	0.452	601.4
0.480	9.593	0.454	614.3
0.478	9.787	0.456	627.1
0.475	9.997	0.458	640.0