

# **Waste Loading Enhancements for Hanford LAW Glasses**

**VSL-10R1790-1, Final Report, Rev. 0;  
12/1/10**

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

**Office of River Protection**

P.O. Box 450  
Richland, Washington 99352

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I. S. Muller  
Vitreous State Laboratory  
The Catholic University of America

K. S. Matlack  
H. Gan  
I. L. Pegg  
Vitreous State Laboratory  
The Catholic University of America

I. Joseph  
EnergySolutions, Inc.


A. A. Kruger  
Department of Energy – Office of River Protection

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**Final Report**

**Waste Loading Enhancements for Hanford LAW Glasses**

*prepared by*

**Isabelle S. Muller, Keith S. Matlack, Hao Gan, Innocent Joseph,  
and Ian L. Pegg**

**Vitreous State Laboratory  
The Catholic University of America  
Washington, DC 20064**

*for*

**EnergySolutions Federal EPC, Inc.**

*and*

**Department of Energy  
Office of River Protection**

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*The Catholic University of America  
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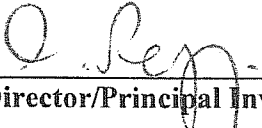
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This report describes the results of testing specified by the above Test Plan. The work was performed in compliance with the quality assurance requirements specified in the Test Plan. Results required by the Test Plan are reported. The test results and this report have been reviewed for correctness, technical adequacy, completeness, and accuracy.

**I.L. Pegg:**   
VSL Program Director/Principal Investigator

**Date:** 12/1/10

**I. Joseph:**   
EnergySolutions Sub-Contract Manager

**Date:** 12/1/10

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## **List of Abbreviations**

ANL-LRM	Argonne National Laboratory – Low-Activity Waste Reference Material
CCC	Canister Centerline Cooling
CUA	Catholic University of America
DCP-AES	Direct Current Plasma - Atomic Emission Spectroscopy
DM	DuraMelter
DOE	Department of Energy
EDS	Energy Dispersive X-ray Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
GFC	Glass Forming Chemical
IHLW	Immobilized High Level Waste
ILAW	Immobilized Low Activity Waste
LAW	Low Activity Waste
M	Molarity
NQA	Nuclear Quality Assurance
ORP	Office of River Protection
PCT	Product Consistency Test
QAPP	Quality Assurance Project Plan
SEM	Scanning Electron Microscope
SOP	Standard Operating Procedure
TFCOUP	Tank Farm Contractor Operation and Utilization Plan
Tg	Glass Transition Temperature
VHT	Vapor Hydration Test
VSL	Vitreous State Laboratory
WTP	Hanford Tank Waste Treatment and Immobilization Plant
XRF	X-Ray Fluorescence Spectroscopy

## **SECTION 1.0 INTRODUCTION**

About 50 million gallons of high-level mixed waste is currently stored in underground tanks at The United States Department of Energy's (DOE's) Hanford site in the State of Washington. The Hanford Tank Waste Treatment and Immobilization Plant (WTP) will provide DOE's Office of River Protection (ORP) with a means of treating this waste by vitrification for subsequent disposal. The tank waste will be separated into low- and high-activity waste fractions, which will then be vitrified respectively into Immobilized Low Activity Waste (ILAW) and Immobilized High Level Waste (IHLW) products. The ILAW product will be disposed in an engineered facility on the Hanford site while the IHLW product will likely be directed to a national deep geological disposal facility for high-level nuclear waste. The ILAW and IHLW products must meet a variety of requirements with respect to protection of the environment before they can be accepted for disposal.

The Office of River Protection is currently examining options to optimize the Low Activity Waste (LAW) Facility and LAW glass waste form. One option under evaluation is to enhance the waste processing rate of the vitrification plant currently under construction. It is likely that the capacity of the LAW vitrification plant can be increased incrementally by implementation of a variety of low-risk, high-probability changes, either separately or in combination. These changes include:

- Operating at the higher processing rates demonstrated at the LAW pilot melter
- Increasing the glass pool surface area within the existing external melter envelope
- Increasing the glass waste loading
- Operating the melter at a slightly higher temperature

The Vitreous State Laboratory (VSL) of The Catholic University of America (CUA) and EnergySolutions, Inc. have evaluated several of these potential incremental improvements for ORP in support of its evaluation of WTP LAW facility optimization [1]. Some of these incremental improvements have been tested at VSL including increasing the waste loading, increasing the processing temperature, and increasing the fraction of the sulfur in the feed that is partitioned to the off-gas (in the event that a decision is made to break the present WTP recycle loop) [2-4]. These approaches successfully demonstrated increases in glass production rates and significant increases in sulfate incorporation at the nominal melter operating temperature of 1150°C and at slightly higher than nominal glass processing temperatures. Subsequent tests demonstrated further enhancement of glass formulations for all of the LAW waste envelopes, thereby reducing the amount of glass to be produced by the WTP for the same amount of waste processed [5, 6]. The next phase of testing determined the applicability of these improvements over the expected range of sodium and sulfur concentrations for Hanford LAW [7]. This approach was subsequently applied to an even wider range of LAW wastes types, including those with high potassium concentration [8]. The feasibility of formulating higher waste loading

glasses using  $\text{SnO}_2$  and  $\text{V}_2\text{O}_5$  in place of  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$  as glass former additives was also evaluated [8].

The present report provides data from investigation of the effects of magnesium content (up to ~10 wt%) on LAW glass properties and from work to identify improved high waste loading glass formulations that meets all processing and product quality requirements for two waste compositions [9]. The scope of testing is detailed in the Test Plan for this work [10]. A glass composition previously developed and tested at VSL for LAW from tank AN-105 (LAWA187 [6]) was varied by substituting Mg for other glass former additives such as Ca, B and Si in an attempt to formulate a glass with improved properties, such as higher waste loading and greater sulfur tolerance. The results were used to reformulate another glass (ORPLG9 [8]) developed for LAW from tank AP-101 that contains high concentrations of alkalis (Na and K). Glass formulation goals for this waste were to increase the sulfur tolerance of the glass as well as to decrease refractory corrosion. Each of the two final glass compositions was evaluated to determine the maximum amount of sulfur that can be incorporated into the glass through melter testing. The results from these tests together with earlier work for ORP were evaluated to develop recommendations for the work scope necessary to modify and update the WTP LAW glass formulation correlation algorithm.

For a large number of Hanford LAW streams, sulfur is the main component that limits waste loading in glass. However, for some LAW streams with low sulfate contents, the alkali concentration becomes the waste loading limiting factor. In general, waste loading is limited by sulfur for wastes with a high sulfur-to-sodium ratio, while those with a low sulfur-to-sodium ratio are limited by sodium (or more specifically, total alkali (sodium plus potassium)). Minimizing overall glass volume across the entire LAW inventory, which is clearly of economic benefit, therefore, entails addressing both the sulfur limitation and the alkali limitation, depending on the waste type.

While processing melter feeds with very high sulfate concentrations, a molten sulfate salt phase forms in the cold-cap region during processing. This phase may exist as transient droplets or may be sufficiently extensive to produce a separate salt phase that becomes mechanically disengaged from the rest of the cold cap. Once formed, the salt phase is slow to dissolve into the underlying glass melt; consequently, the salt phase typically forms before the underlying glass melt is saturated with sulfate [11-14]. If the feed rate is sufficiently low (which is clearly undesirable), the equilibrium sulfate saturation concentration in the glass can be approached more closely before a separate salt phase forms. However, in general, as the feed rate is increased, for the same sulfate concentration in the feed, the salt phase appears progressively earlier. Thus, in practice, the formation of a sulfate phase is governed by both thermodynamic and kinetic factors and, therefore, the effects of both must be considered in order to avoid the formation of such phases during operations. The presence of the corrosive, low-melting, electrically conductive salt phase is undesirable from the perspectives of melter operation, melter lifetime, safety, and product quality. Accordingly, the WTP plans to control the composition of the LAW melter feed such that formation of a separate salt phase is avoided. Clearly, the control bounds that are imposed will determine the achievable waste loading limits and, therefore, will determine the waste processing rate for a given glass production rate (i.e., melter capacity).



For waste with low sulfur-to-sodium ratio, waste loading is instead limited by the total alkali content in the glass. At high alkali contents, glass leach resistance (PCT and VHT) decreases and the refractory corrosion rate in the glass melt increases. In addition, the melt viscosity may become too low and the electrical conductivity may become too high. Typically, however, the product leach resistance and the refractory corrosion properties are the first to be compromised as the alkali content in the glass is increased. The present work addresses LAW streams with the objective of determining the maximum achievable waste loadings for sodium-limited and sodium + potassium limited formulations. As noted above, the broader intent is to develop a basis for estimation of the potential maximum waste loadings and corresponding glass volumes for the entire LAW inventory.

Under a separate contract to support the WTP Project, the VSL has developed and tested glass formulations for WTP to provide data to meet the WTP contract requirements and to support system design activities [15-20]. That work is based upon small-scale batch melts (“crucible melts”) using waste simulants. Selected formulations have also been tested in small-scale, continuously-fed, joule-heated melters (DM10 and DM100 systems) [13, 14, 21-30] and, ultimately, in the LAW Pilot Melter [31-42]. Such melter tests provide information on key process factors such as feed processing behavior, dynamic effects during processing, sulfate incorporation, processing rates, off-gas amounts and compositions, foaming control, etc., that cannot be reliably obtained from crucible melts. This sequential scale-up approach in the vitrification testing program ensures that maximum benefit is obtained from the more costly melter tests and that the most effective use is made of those resources.

Under the WTP support effort, VSL and EnergySolutions have developed and identified glass compositions for processing the Phase I LAW tank streams for the WTP. These compositions have been tested for processing and product quality requirements at various scales ranging from crucible melts of about 400 g up to the LAW Pilot Melter at processing rates in excess of 6600 kg/day (2000 kg/m<sup>2</sup>/day). The testing included the nominal feed compositions and those with  $\pm 15\%$  variations in the waste simulants added to the melter feeds. The melter testing provided high confidence that the selected WTP compositions are unlikely to cause accumulation of a separate sulfate phase in the melter even at high feed processing rates. Feed processing characteristics and off-gas characteristics have been determined at various melter scales and data have been collected to support engineering and permitting requirements. Furthermore, statistically designed composition matrices were generated and crucible melts of the resulting glass compositions were prepared and characterized to qualify the glass composition region covering these LAW glass compositions selected for WTP waste processing [19, 20]. The selected WTP compositions have also been tested to ensure their compatibility with melter materials of construction. The glass formulation development and melter testing work for the selected WTP compositions have reached a level of maturity where the compositions can be used for waste processing at the WTP with relatively high confidence.

To capture the body of information collected in support of the WTP baseline, VSL/EnergySolutions have developed a LAW glass formulation correlation [43] that can be used to calculate glass compositions for waste processing, given the composition of an LAW stream. The correlation can be used to calculate compliant glass compositions for waste processing based on the ratios of sulfate to sodium (S/Na) and potassium to sodium (K/Na) in the LAW stream.

The correlation uses a set of empirical relationships derived from the above array of test data to define waste loadings and the concentrations of glass former additives for a given LAW composition.

The glass formulation and melter testing work reported herein was aimed at identifying glass compositions that have higher waste loadings than those developed for the WTP baseline. To achieve this goal, it is necessary to modify the glass formulations to mitigate the effects of increased waste loadings on sulfur salt formation and corrosion of refractory components. Two LAW compositions were specified by ORP for this work [9, 44]. Two ORP LAW glass compositions, LAWA187 [6] and ORPLG9 [8], were used as the starting points for development of the new formulations. These glasses are based on the compositions of LAW in tanks AN-105 and AP-101, which have amongst the highest sodium and potassium concentrations expected in Hanford LAW. This information will provide ORP with a basis for evaluation of the likely potential for future enhancements of the WTP over and above the present well-developed baseline. In this regard, the present work is complementary to, and necessarily of a more exploratory nature than the work in support of the current WTP baseline.

## **1.1 Test Objectives**

As described in the Test Plan [10], the principal objective of this work was to extend the glass formulation methodology developed in the earlier work [2, 5-8, 43] by development of acceptable glass compositions for two LAW waste compositions in an attempt to increase waste loading and sulfur tolerance as well as reduce refractory corrosion. Per the ORP scope of work [9], the effects and potential benefits of increased magnesium concentrations were investigated in the reformulation process. Initial tests were conducted to determine the effect of magnesium on glass properties and the preferred concentrations. These objectives were accomplished through a combination of crucible-scale work and tests on the DM10 melter system. The DM10 was used for several previous tests on LAW compositions [2-8, 13, 14] to determine the maximum feed sulfur concentrations that can be processed without forming secondary sulfate phases on the surface of the melt pool. This melter is the most efficient melter platform for screening glass compositions over a wide range of sulfate concentrations and therefore was selected for the present tests. The melter tests provide information on melter processing characteristics and off-gas data, including sulfur incorporation and partitioning.

The two waste types selected for testing, and their respective starting glass compositions are:

- LAWA187 with a waste loading of 30.5 wt% for LAW from tank AN-105 [6].
- ORPLG9 with a waste loading of 29.1 wt% for LAW from tank AP-101 [8].

The objectives of the work are [10]:

- Develop glass formulations that are compliant with processing and product quality requirements for the above two waste streams, that exceed previously attained waste loadings, sulfur tolerance, and are less corrosive to refractory materials.

- Evaluate the effect of higher MgO concentrations on the properties of LAW glasses formulated for tanks AN-105 and/or AP-101.
- Based on the results of the above evaluation and previous work for WTP and ORP, formulate higher waste loading glasses for AN-105 and AP-101.
- Determine the maximum feed sulfur concentrations that can be processed on the DM10 melter without the formation of secondary sulfate phases for each of the two LAW glass formulations.
- Develop the work scope needed to update and modify the LAW correlation algorithm [43] using the new data on high waste loading LAW glasses developed for ORP.

## **1.2 Test Overview**

Glass formulation development and testing was conducted to identify higher waste loading compositions than those given in Table 1.1 for each of the LAW AP-101 and AN-105 tank compositions. Magnesium as a glass former additive was tested at varying concentrations in order to evaluate its effect on glass properties. Initial glass formulation work began with a glass previously formulated for the LAW AN-105 waste, LAW A187 [6], by progressively replacing glass forming additives with magnesium to determine the effect of MgO content (up to ~10 wt%) on LAW glass properties. Based on the results of this evaluation and previous LAW glass formulation work for ORP and WTP, a high waste loading glass composition that meets all processing and product quality requirements was developed for this waste composition. Once a suitable glass was formulated for the LAW AN-105 waste, a similar strategy was used to reformulate ORPLG9 glass [8] in an attempt to reduce K-3 refractory corrosion and increase sulfur tolerance for glasses formulated for the LAW AP-101 waste stream. Crucible melts were prepared and the glass samples characterized with respect to properties affecting processing (viscosity, electrical conductivity, crystallization, and refractory corrosion) and product quality (PCT and VHT) to determine whether they are compliant with WTP requirements as specified in Table 1.2. Initial characterization of the glass samples was limited to the properties that were expected to be most challenging so that further characterization could be limited to only those samples that passed the initial property requirements. Based on the crucible melt results, a glass formulation was selected for DM10 melter testing for each of the two LAW compositions. For each waste composition, the crucible melt work identified suitable formulations that maximize waste loading. DM10 tests were conducted with the selected glass compositions to determine the processing characteristics of the glass composition and the corresponding melter feed. These tests were performed at 1150°C and with a target glass production rate of ~2,250 kg/(m<sup>2</sup>-day). Each test segment was approximately 14 hours in duration, which corresponds to about three melter turnovers at the above glass production rate. In each test sequence, composed of about 3 to 4 test segments, the sulfate content was progressively increased to the point at which a sulfate salt phase developed, indicating the limit of sulfate incorporation for that particular formulation.

The glass formulation development work relied heavily on previous work performed for ORP [2, 5-7] and relevant WTP LAW glass formulation work [12-20]. Existing property-composition models were used to guide glass formulation development. However, since the existing models are not expected to be reliable in the new composition regions that were explored in this work, glass science knowledge and experience and information about the effect of various additives on glass structure and properties were used as additional tools to guide glass formulation development.

### **1.3 Quality Assurance**

This work was conducted under a quality assurance program compliant with 10 CFR 830 Subpart A, Nuclear Quality Assurance (NQA)-1 (2004), and DOE Order 414.1C. This program was supplemented by a Quality Assurance Project Plan (QAPP) for ORP work [45] that is conducted at VSL. Test and procedure requirements by which the testing activities are planned and controlled are defined in this plan in combination with the Test Plan [10]. The program is supported by VSL standard operating procedures that were used for this work [46]. The requirements of DOE/RW-0333P are not applicable to this work.

## **SECTION 2.0**

### **WASTE SIMULANTS, GLASS FORMULATIONS AND FEED ANALYSIS**

Glass formulation development and melter testing were conducted to identify compliant high waste loading glass formulations for Hanford LAW streams, starting with LAW Hanford tank AN-105 waste simulant and the previously tested LAWA187 as the base glass composition [6]. Per the ORP Scope of Work [9], special attention was given to determine the extent to which this glass formulation can be improved (increased waste loading and higher sulfur tolerance) by using magnesium as a glass former additive. Once the optimum MgO concentration was established, other additive concentrations and the waste loading were modified to achieve the optimum glass formulation. Formulating a glass for the LAW AP-101 waste using the ORPLG9 glass as a starting point followed a similar approach.

#### **2.1 Hanford Tank AN-105 Waste Simulant and ORPLA Glass Formulations**

Glass formulation development and testing for the Hanford LAW tank AN-105 waste simulant began with the previously tested LAWA187 as the base glass composition [6]. Details of the waste simulant and glass formulation development and testing are given below.

##### **2.1.1 Hanford Tank AN-105 Waste Simulant**

The waste simulant was the same as that used in the earlier studies on AN-105, which was based on the composition data for tank AN-105 as given in a WTP Test Specification [47]; however, the sulfate concentration was increased from 0.6 to 2.7 wt% SO<sub>3</sub> in order to meet the requirements of the present tests. The base waste composition incorporates Tank Farm Contractor Operation and Utilization Plan (TFCOUP) [48] data, actual waste analysis data, and WTP flow sheet information. The sodium concentration in the simulant includes a 2.5 % increase to account for sodium additions in pretreatment [15, 49]. The nominal concentration, expressed in terms of the sodium molarity, was determined on the basis of melter feed rheology tests on similar formulations [50, 51]. The results of those tests led to the selection of 8.0 molar sodium as the nominal simulant concentration for the LAW AN-105 waste. This is the same concentration that was used for previous WTP melter tests for LAW AN-105 waste [21, 28].

The nominal simulant formulation is given in Table 2.1. The LAW AN-105 simulant is a solution of predominantly sodium, aluminum, nitrate, nitrite, and sulfate. Since the simulant was similar to those tested previously at the VSL, no new laboratory feed tests were needed. For the melter tests, the waste simulant was prepared at VSL and the glass forming chemicals were added to produce a single large batch. Sugar as a reductant and the requisite combinations of sodium hydroxide and sodium sulfate to adjust the sodium and sulfur contents were added to the feed for each melter test segment.

### 2.1.2 ORPLA Glass Formulation Development

LAWA187, a glass formulation previously developed and tested for ORP [6], based on the composition of the LAW AN-105 waste stream, was used as the starting point for the current work. The objective was to develop a glass composition that improves upon LAWA187, which was used in previous melter tests, with a nominal waste loading of 30.5 wt% and 23 wt% Na<sub>2</sub>O. The MgO concentration in this glass is 0.9 wt%. DM10 melter tests with this glass formulation showed that it can tolerate up to 0.95 wt% SO<sub>3</sub> in the feed on a glass basis. K-3 refractory corrosion and VHT alteration rate were the major constraints that limited waste loading in this glass formulation.

Based on test results from previous sets of crucible melts with Na<sub>2</sub>O concentrations in the range of 23 to 25 wt% [6, 7, 8], the Na<sub>2</sub>O concentrations in the new formulations selected for testing were set between 23 and 24.15 wt%. Fifteen new crucible melts were prepared and characterized in an effort to identify a new glass formulation that meets all of the processing and product quality requirements [52, 53]. The target and analyzed compositions of the crucible melts are given in Table 2.2. Observations of heat treated ORPLA glass samples are presented in Table 2.3 and measured sulfate solubilities are presented in Tables 2.4. Results from the Product Consistency Test (PCT) and Vapor Hydration Test (VHT) are given in Table 2.5. Measured viscosities and electrical conductivities of the ORPLA glasses are given in Table 2.6 and results of K-3 refractory corrosion tests are given in Table 2.7.

The Na<sub>2</sub>O concentration in the glass was initially set at 23 wt%, which is the same as that in LAWA187 [6]. One of the new glasses, ORPLA26, replicates LAWA187 with a target SO<sub>3</sub> concentration of 0.95 wt%, the highest SO<sub>3</sub> concentration from melter tests that did not result in the formation of a secondary phase. Glasses ORPLA27 to ORPLA32 were formulated with higher concentrations of MgO (4, 7, and 10 wt%), compensated by decreases in the concentrations of Al<sub>2</sub>O<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, and SiO<sub>2</sub> in ORPLA27, ORPLA28 and ORPLA29, and decreases in the amounts of Al<sub>2</sub>O<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, and CaO in ORPLA30, ORPLA31 and ORPLA32. As can be seen in Table 2.5, all of the above glasses with high MgO concentrations showed high leach rates, both in PCT and VHT.

Since the above glasses with high MgO concentrations showed high leach rates, the next set of glasses, ORPLA33 to ORPLA35, were formulated at lower MgO concentrations varying from 1 to 3 wt%. The concentrations of SnO<sub>2</sub> and ZrO<sub>2</sub>, two additives that have been shown to improve leach resistance [54-57], were increased in these glasses. ZrO<sub>2</sub>, which is added primarily to improve the chemical durability of the glass, was kept at a concentration of about 6 wt% or less to avoid crystallization of zircon [8]. Of these glasses, ORPLA33, ORPLA33-1 and ORPLA34 showed acceptable leach rates, whereas ORPLA35 with the lowest SnO<sub>2</sub> and ZrO<sub>2</sub> concentrations failed the PCT and VHT acceptance criteria.

Additional glasses (ORPLA36 to ORPLA38-1) were prepared with a target Na<sub>2</sub>O concentration of about 24 wt% and comparatively high SnO<sub>2</sub> (~2.5 wt%) and ZrO<sub>2</sub> (~6 wt%) concentrations. Al<sub>2</sub>O<sub>3</sub>, B<sub>2</sub>O<sub>3</sub> and CaO concentrations were decreased along with an increase in SiO<sub>2</sub> concentration, which is predicted to decrease alteration rate on the VHT and increase

sulfate solubility. One of the glasses from this set, ORPLA38-1, showed the highest sulfate solubility and acceptable performance on PCT and VHT.

For the very high  $\text{Na}_2\text{O}$  glasses, the properties of most concern are VHT alteration rate and K-3 refractory corrosion. In order to reduce K-3 refractory corrosion,  $\text{Cr}_2\text{O}_3$  was added to all of the glasses at a concentration of about 0.5 wt% in addition to maintaining  $\text{SiO}_2$ ,  $\text{SnO}_2$ , and  $\text{ZrO}_2$  concentrations at high levels [7, 8].

Target and analyzed compositions of the ORPLA glasses are given in Table 2.2. Glass compositions were determined by x-ray fluorescence spectroscopy (XRF) on powdered glass samples, except for  $\text{B}_2\text{O}_3$ , which was measured by direct current plasma – atomic emission spectroscopy (DCP-AES) after acid dissolution. As is evident from the table, the target and analyzed compositions generally show good agreement. Testing of all formulations started with glass preparation and optical microscopic evaluation of the as-melted sample. Glass samples were heat treated for 20 hours at  $950^\circ\text{C}$  and then evaluated for secondary phases. Observations of the as-melted and heat treated glasses are given in Table 2.3. Large sodalite crystals representing 0.3 to 4 volume percent of the heat treated glass samples were observed in the six glass samples with the highest  $\text{MgO}$  concentrations. A scanning electron microscopy (SEM) image of the heat treated ORPLA28 glass is given in Figure 2.1. Energy dispersive x-ray spectroscopy (EDS) spectra of two crystals (spinel and sodalite) from the SEM image are given in Figure 2.2. X-ray diffraction (XRD) analysis of the sample identified the presence of lazurite, a blue sulfate sodalite. While magnesium was not identified in these large alumina-silicate crystals, it was abundant in the small chromium rich spinels that represent a much smaller volume fraction of the crystals. The glasses with lower  $\text{MgO}$  concentrations appeared optically clear with small amounts ( $< 0.1$  vol%) of spinels rich in chromium and zinc. As melted samples of ORPLA27 to ORPLA32 with high  $\text{MgO}$  concentrations appeared opaque, while the lower  $\text{MgO}$  glasses appeared clear. The opaque appearance of the higher  $\text{MgO}$  glasses, an example of which is given in Figure 2.3, is probably due to undissolved sulfate, which was also seen as a separate sulfate layer in some of the samples.

The sulfate solubilities of the ORPLA glasses were assessed by batch saturation tests. This is a crucible-scale screening test that is used to provide an indication of the extent of sulfur incorporation that will be obtained under actual melter operating conditions, which is the measure that is of practical importance. The results of these screening tests are then used to guide the range of sulfur concentrations over which the melter tests are performed. The batch saturation tests were performed by remelting finely ground samples of the glasses with an excess of sulfate amounting to 4 wt%  $\text{SO}_3$  if all of it were retained in the glass. The remelted glass samples are identified with an S4 at the end of the sample name. Results of sulfate batch saturation tests are given in Table 2.4 and Figure 2.4. The results identified as “after acid wash” are analyses of glass samples remelted with 4 wt%  $\text{SO}_3$  after grinding and washing to remove any interstitial sulfate phases to ensure that only the  $\text{SO}_3$  that is dissolved in the glass is measured. The sulfate retentions in the glasses (“after acid wash”) varied from about 0.44 wt%  $\text{SO}_3$  for ORPLA29 to 0.88 wt%  $\text{SO}_3$  for ORPLA26.

VHT results are summarized in Table 2.5 and illustrated in Figures 2.5. The results show that all of the new ORPLA glasses with more than 3 wt%  $\text{MgO}$  exceeded the VHT alteration rate

requirement of 50 g/m<sup>2</sup>/day. Increases in SiO<sub>2</sub> and ZrO<sub>2</sub> concentrations with decreases in the Al<sub>2</sub>O<sub>3</sub>, B<sub>2</sub>O<sub>3</sub>, CaO and MgO concentrations, lead to decreases in the VHT alteration rate. PCT releases for the glasses are given in Table 2.5 and Figure 2.6 and show trends similar to those observed in VHT results. The results indicate that the immobilized low activity waste (ILAW) product quality requirement of normalized mass loss of less than 2 g/m<sup>2</sup> for B, Na, and Si is exceeded for boron in all glasses containing 3 wt% MgO or more. In many cases, the normalized mass loss also exceeds the limit for sodium, even for glass compositions with a lower Na<sub>2</sub>O content of 23 wt%. Also notable and indicative of the detrimental effect of magnesium is the fact that sodium release is lower than boron release (30 to 35% lower); this deviation from congruence is indicative of the role of a previously documented magnesium phyllosilicate phase which accelerates glass leaching [54, 58].

The viscosities and electrical conductivities of glasses at select temperatures are given in Table 2.6. Viscosities and electrical conductivities of the remaining glasses were not measured because they failed one of the leaching or K-3 corrosion criteria, or showed low sulfate solubility. All of the measured viscosity and electrical conductivity values are in the acceptable range for melt processing [52] at WTP.

K-3 refractory corrosion is a concern due to the high alkali content of the ORPLA glass formulations. The glass with the highest alkali contents and sulfate solubility and showing the best leaching performance, ORPLA38-1, was tested for its K-3 corrosion characteristics. K-3 refractory corrosion test results for this glass are given in Table 2.7 and Figure 2.7, where they are compared to the results for some of the previously tested ORP LAW glasses [7]. Acceptability of the corrosion characteristics of a glass composition is somewhat subjective because a glass composition that shows slightly higher K-3 corrosion, but allows higher waste loading, may be a more economic choice than one with lower K-3 corrosion and lower waste loading. However, for WTP LAW glass formulation development, a neck corrosion of 0.035 inches on the 6-day K-3 coupon corrosion test at 1208°C has been used as an acceptance limit. For the current LAW glass formulation development work for ORP, since higher waste loading compositions are being explored, a slightly higher neck corrosion value of 0.040 inches has been used as a guide for acceptable corrosion characteristics. ORPLA38-1 shows neck corrosion value of 0.0435 inches.

ORPLA38-1 met all of the product quality and processing criteria except for slightly high K-3 neck corrosion, and showed reasonably high sulfate solubility. This glass composition was, therefore, selected for DM10 melter tests. The measured properties of the glass ORPLA38-1 are compared to the ILAW performance requirements [52, 53] in Table 2.8. The density measured on crucible glass ORPLA38-1 was 2.69 g/cc; the density was also measured for a glass sample from the DM10 melter test and showed a value of 2.60 g/cc, which is well within the acceptable limit. In addition, none of the measured densities of LAW glasses prepared for WTP or ORP exceeded or even approached the limit of 3.7 g/cc. Glass transition temperature ( $T_g$ ) measurements and canister centerline cooling (CCC) heat treatments were not conducted on ORPLA38-1 samples because the glass is expected to be acceptable with respect to these properties. The only requirement for  $T_g$  is that it be measured and reported. Since the sample heat treated at 950°C for 20 hours showed only minute amounts ( $<<0.1$  vol%) of crystals, CCC treatment is not expected to cause extensive crystallization. Although cooling of the glass



discharged from the DM10 melter occurs faster than in a WTP LAW canister, examination of cooled ORPLA38-1 glass samples from the DM10 melter corroborated this expectation in that very few crystals were present in the discharge glass samples.

The composition of the ORPLA38-1 glass used in melter tests is given in Table 2.9 along with the oxide contributions from the LAW AN-105 waste simulant and from the glass former additives. The simulant was prepared with no  $\text{SO}_3$  and the sulfur concentration was increased in steps during the melter tests by adding the appropriate amounts of  $\text{Na}_2\text{SO}_4$  and  $\text{NaOH}$  to the feed. The melter feed was prepared at a  $\text{Na}_2\text{O}$  concentration of 23.3 wt% (for zero sulfate) in order to accommodate  $\text{Na}_2\text{SO}_4$  and  $\text{NaOH}$  additions, without increasing the  $\text{Na}_2\text{O}$  concentration above 24.0 wt% while accommodating up to 0.90 wt%  $\text{SO}_3$ . The types and amounts of glass former additives used to prepare the melter feed along with the target feed properties are given in Table 2.10a. The glass former additives are the same as those planned for use at the WTP, with the exception of chromium, tin, and vanadium, which would be new additives. The amounts of  $\text{Na}_2\text{SO}_4$  and  $\text{NaOH}$  added to the feed to obtain 24 wt%  $\text{Na}_2\text{O}$  and 0.50 to 0.90 wt%  $\text{SO}_3$  are given in Table 2.10b.

## 2.2 Hanford Tank AP-101 Waste Simulant and ORPLG Glass Formulations

Glass formulation development and testing for the LAW Hanford tank AP-101 waste simulant began with the previously tested ORPLG9 as the base glass composition [8]. Details of the waste simulant and glass formulation development and testing are given below.

### 2.2.1 Hanford Tank AP-101 Waste Simulant

The waste simulant for the present study was based on the composition data for Hanford LAW tank AP-101, as given in a WTP Test Specification [47]. The base waste composition incorporates TFCOUP [48] data, actual waste analysis data, and WTP flow sheet information. The sodium concentration in the simulant includes a 2.5 wt% increase to account for sodium additions in pretreatment [14, 49]. The nominal concentration, expressed in terms of the sodium molarity, was estimated on the basis of melter feed rheology tests on similar formulations [50, 51]. The concentration of the simulant used in DM10 melter tests was 8 molar sodium. The nominal simulant formulation is given in Table 2.11. The LAW AP-101 simulant is a solution of predominantly aluminum, sodium, potassium, nitrate, nitrite, carbonate, and sulfate.

### 2.2.2 ORPLG Glass Formulation Development

The starting point for the current ORPLG glass formulation development work was ORPLG9, a glass based on the composition of the LAW AP-101 waste stream. The objective was to develop a glass formulation that can improve on ORPLG9 in terms of sulfate solubility and refractory corrosion while maintaining the target waste loading of 29.10 wt% with 21 wt%  $\text{Na}_2\text{O}$  and 5.75 wt%  $\text{K}_2\text{O}$ . Another objective was to determine the effect of higher  $\text{MgO}$

concentrations than the starting concentration of 0.96 wt% on the properties of the glasses. DM10 melter tests with ORPLG9 showed very little tolerance for  $\text{SO}_3$  in the feed (0.2 wt%  $\text{SO}_3$  or less). K-3 refractory corrosion and VHT alteration rate were the major constraints that limited waste loading in the ORPLG9 glass formulation. The high alkali content of the LAW AP-101 waste from a combination of  $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$  is the primary challenge for glass formulation development in this composition region.

Target and analyzed compositions of the ORPLG glasses are given in Table 2.12. In order to reduce K-3 refractory corrosion,  $\text{Cr}_2\text{O}_3$  was kept at a concentration of ~0.6 wt% in all of the glasses, as was previously used [8]. The new ORPLG glass development work started with ORPLG13, ORPLG14 and ORPLG15, all with about 4 wt% MgO and with increasing waste loadings. The waste loading increases result in increases in the  $\text{Na}_2\text{O}$  concentrations from 20 to 20.5 to 21 wt% with corresponding increases in the  $\text{K}_2\text{O}$  concentrations from 5.47 to 5.61 to 5.75 wt%. The additive blend was kept identical to that used for ORPLG9, changing all of components together as the waste loading was changed. Similar to the ORPLA glasses with high MgO contents, all three of the ORPLG glasses failed both VHT and PCT leach rate limits. The next set of glasses, ORPLG16 to ORPLG19, was formulated with 2 wt% MgO. These glasses contained reduced concentrations of ZnO and  $\text{Al}_2\text{O}_3$ , along with high concentrations of  $\text{SnO}_2$ ,  $\text{ZrO}_2$  and  $\text{SiO}_2$ .  $\text{Na}_2\text{O}$  concentrations in these glasses varied from 20.6 to 21.1 wt%. Viscosity models were used to select additive combinations yielding similar or slightly higher melt viscosity than ORPLG9 in an attempt to reduce K-3 refractory corrosion. However, all of the glasses failed PCT and VHT leach rate criteria. Finally, MgO concentrations were reduced to about 1 wt% or further reduced to 0.5 wt% in the remaining glass formulations, which tested two waste loadings at about 20.5 and 21 wt%  $\text{Na}_2\text{O}$ . In these glasses, the amounts of  $\text{SiO}_2$ ,  $\text{SnO}_2$  and  $\text{ZrO}_2$  were varied while maintaining the remaining glass former additives fairly constant.

Target and analyzed compositions of the ORPLG glasses are given in Table 2.12. Testing and analysis of the glasses followed the same methods used for ORPLA glasses, which are described in Section 2.1.2. As is evident from the table, the target and analyzed compositions show good agreement. Glass samples were heat treated for 20 hours at 950°C and evaluated for secondary phases. Observations of the as-melted, and heat treated glasses are given in Table 2.13. The first three, higher magnesium glasses appeared slightly opaque while all other as-melted glasses appeared clear. The heat treated glasses also showed little crystallization (0.3 to 0.7 vol%).

The sulfate solubilities of the ORPLG glass compositions were assessed by batch saturation tests. The batch saturation tests are described in Section 2.1.2 and the results of the tests are given in Figure 2.4 and Table 2.14. The sulfate solubilities of the ORPLG glasses varied from 0.42 to 0.52 wt% by batch saturation tests and generally increased with increased waste loading. In addition to batch saturation tests, the sulfate solubility of the ORPLG27 glass composition was determined by gas bubbling. Results of these tests are given in Figure 2.8 along with results from the earlier tests on ORPLG9 [8]. ORPLG27 showed a sulfate solubility of 0.58 wt% and the onset of a secondary sulfate layer occurred at an  $\text{SO}_3$  concentration of about 0.33 wt%.

VHT and PCT results are summarized in Table 2.15 and illustrated in Figures 2.5 and 2.6. Glasses containing high concentrations of MgO do not meet either the PCT release limit or the VHT alteration rate limit. Among the glasses with high waste loading (21 wt% Na<sub>2</sub>O and 5.75 wt% K<sub>2</sub>O), only ORPLG26 and ORPLG27 meet both leaching limits. The viscosities and electrical conductivities of seven of the ORPLG glasses at select temperatures are given in Table 2.16. All of the viscosity and electrical conductivity values are in the acceptable range for processing. Similar to ORPLA glasses, the ORPLG glasses were designed to have viscosities towards the higher end of the acceptable range in order to reduce refractory corrosion. Three ORPLG glasses with high waste loadings (>29 wt%) were tested for their K-3 corrosion characteristics. K-3 refractory corrosion test results for these glasses are given in Table 2.17. The three have somewhat comparable K-3 corrosion characteristics with a neck loss near 0.03 inches. The depth of the reaction zone into the K-3 coupon is lower in these three samples than it was in the previously tested glass ORPLG9 of similar waste loading.

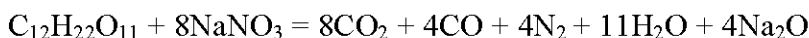
Of the fifteen ORPLG glass formulations tested, ORPLG26 and ORPLG27 are two similar formulations with high waste loading that also meet all processing and product quality requirements. Formulation ORPLG27, with slightly higher sulfate solubility and lower viscosity was recommended by VSL/EnergySolutions for DM10 melter tests. The measured properties of the glass ORPLG27 are compared to the ILAW performance requirements [52, 53] in Table 2.18. Measured densities of the crucible and melter glasses were 2.71 and 2.67 g/cc, respectively, both well below the limit of 3.7 g/cc. Glass transition temperature measurements and CCC heat treatments were not conducted on ORPLG27 glass. Examination of cooled ORPLG27 glass samples from the melter showed little crystallization, indicating that the glass is unlikely to show substantial crystallization on CCC heat treatment.

The composition of the ORPLG27 glass used in melter tests is given in Table 2.19 along with the oxide contributions from the LAW AP-101 waste simulant and from the glass former additives. The simulant was prepared with no SO<sub>3</sub> and the sulfur concentration was increased in steps during the melter tests by adding the appropriate amounts of Na<sub>2</sub>SO<sub>4</sub> and NaOH to the feed. The melter feed was prepared at a Na<sub>2</sub>O concentration of 20.21 wt% in order to accommodate Na<sub>2</sub>SO<sub>4</sub> and NaOH additions without increasing the Na<sub>2</sub>O concentration above 21.0 wt%. The types and amounts of glass former additives used to prepare the melter feed along with the target feed properties are given in Table 2.20a. The glass former additives are the same as those planned for use at the WTP, with the exception of chromium and tin, which would be new additives. The amounts of Na<sub>2</sub>SO<sub>4</sub> and NaOH added to the feed to obtain 21.0 wt% Na<sub>2</sub>O and 0.0 to 0.6 wt% SO<sub>3</sub> are given in Table 2.20b.

## 2.3 Sugar Additions

With high nitrate feeds, the addition of reductants is necessary in order to control melt foaming. Sugar, which was used for this purpose at West Valley, has also been selected as the baseline reductant for the WTP. The amount of sugar required increases with the amount of nitrates present in the feed and decreases with the amount of waste organics present in the feed, which themselves act as reductants. Excessive additions of reductants can be deleterious, leading to over-reduction of the melt and formation of sulfides and molten metals. Consequently, the

oxidants and reductants in the feed must be suitably balanced. The basis for achieving this balance was developed by VSL and EnergySolutions for the vitrification of high-sodium-nitrate feeds at Savannah River Site's M-Area and has been successfully applied to the processing of a wide variety of simulated WTP feeds over the past six years. In developing this approach, we elected to conservatively adopt the most reducing potential reaction as the basis for the *definition* of a "sugar" or stoichiometric ratio of 1.0 as a result of concerns for over-reducing the melt. Such a reaction, using sodium salts as an example, is:



Fundamentally, the basis that is selected is simply a convention, since the precise stoichiometry of the reactions involved is neither known nor constant under the conditions prevailing in the melter. However, with this convention, a sugar ratio of 1.0 corresponds to one mole of sucrose per eight moles of nitrate or, more generally, 1.5 moles of organic carbon per mole of nitrate. It is then expected that significantly less sugar than this will be required in practice. The empirically determined amount required to successfully control melt foaming without significantly reducing the glass melt was found to correspond to a ratio of 0.5 when any nitrites present were counted as nitrates (i.e., 0.75 moles of organic carbon per mole of nitrate + nitrite). This approach has been employed for all WTP melter testing. It is, however, expected that slight variations around the nominal value of 0.5 may be necessary to account for differences in the reducing power of waste organics in comparison to sugar, particularly for LAW streams that are high in organics.

As an example, the calculation of the amount of sugar needed for the present LAW AN-105 (Envelope A) feed to achieve a sugar ratio of 0.5 proceeds as follows:

- One liter of 8 molar sodium simulant contains 1.857 moles of nitrite and 2.048 moles of nitrate, giving a total of 3.905 moles of NO<sub>x</sub> (see Table 2.1)
- The required total amount of organic carbon for a sugar ratio of 0.5 is  $3.905 \times 0.75 = 2.929$  moles
- One liter of simulant contains 0.174 moles of organic carbon (see Table 2.1)
- Therefore,  $2.929 - 0.174 = 2.755$  moles of organic carbon must be added.

Since the molecular weight of sucrose is 342 g,  $2.755 \times 342/12 = 78.5$  g sugar must be added per liter of simulant, as shown in Table 2.10.a.

## 2.4 Analysis of Melter Feed Samples

### 2.4.1 General Properties

Feed samples from melter tests were analyzed to confirm physical properties and chemical composition. Samples were taken from residual composite melter feed and feed prior to adjusting the sulfur content. Sample names, sampling dates, measured properties and comparisons with feed analysis for similar waste streams [6, 8] are provided in Table 2.21. The average measured glass yield for the melter samples differed from the target values by less than

two percent (on a mass per unit mass basis) validating the use of the target value for calculating glass production rates. Measured feed densities, feed solids content, and pH are within narrow ranges for each waste type due to the similarity in waste loading and, in the case of the current tests, the consistency of the feed. The measured pH values for the composite feed samples are higher than the stock feed as a result of the addition of sodium hydroxide and sodium sulfate to adjust the sulfur and sodium content.

## **2.4.2 Chemical Composition**

The chemical compositions of the feed samples were determined by first making a glass from the feed sample via crucible melt. The glass was subsequently crushed and analyzed directly by XRF. The boron oxide target values were used for normalizing the XRF data since boron was not determined by XRF. The XRF-analyzed compositions of the feed samples are provided in Table 2.22. The data generally show good agreement with the target composition for the major components. Of the oxides with target concentrations of one percent or greater in the composite feed samples, the XRF values showed deviations of less than 10%, except for a high tin bias of about twenty five percent in the ORPLA38-1-S0 composition. Deviations of up to about thirteen percent were observed for other oxides in the stock feed batch, reflecting a small bias from sampling the drum. Surpluses of tin were also observed in the product glass while processing the ORPLA38-1-S0 composition (see Section 4.1) as well as in previous tests using tin as an additive [6-8] suggesting that there may be a potential analytical bias or the purity of the additive source is underestimated. Similar observations were made for vanadium, which was 10 to 13 relative percent above target concentrations, similar to previous tests [2, 6, 7, 59].

Corroborative analysis of feed samples was performed by the DCP analysis of solutions generated by microwave aided acid dissolution; results are compared to target and XRF analysis in Table 2.23. Measured boron concentrations were within seven and a half percent of the target, validating the use of the target value for normalizing the XRF data. The analysis also confirms the lack of lithium in the feed. Agreement between the two analytical methods was excellent, except for low sodium values obtained from the DCP analysis; previous experience indicates that the XRF results are more reliable in this regard [6, 8, 59]. Also of note are the lower, and therefore closer to target, concentrations of tin and vanadium for the DCP analysis suggesting a potential high bias for the XRF method. Measured chromium oxide concentrations were about 0.2 weight percent above the target for all but one XRF analysis, whereas the DCP analysis more closely approximates the target concentration.

Volatile minor elements such as sulfur and chlorine are, as expected, below target due to loss during crucible melting. Titanium oxide was measured in the feed samples at levels ranging from 0.05 to 0.15 weight percent, even though it was not included in the target composition. Similar observations were made in previous tests with LAW melter feeds [6-8, 59]; this is due to the presence of titanium as a contaminant in the glass forming additives, most notably kyanite [2]. Iodine was measured in the feed samples at around a tenth of a weight percent as a result of its presence as an impurity in the tin additive used [6-8]. Common elements such as chromium, iron, and phosphorus, which are typical impurities in bulk chemicals, are over-represented when

the constituent is a minor component. Higher measured concentrations for these common elements may also be attributable to measurement uncertainties at these low concentrations.

## **SECTION 3.0**

### **DM10 MELTER TESTS**

Melter tests were conducted on the DM10 with the LAW simulants from 5/6/10 to 5/14/10 to determine the maximum sulfur concentration that can be processed without forming secondary phases for each of the two glass compositions. These tests produced over 200 kg of glass from about 400 kg of feed. Tables 3.1 and 3.2 provide summaries of the DM10 tests, including run times, the amount of sulfur in the feed, the amount of feed processed, the amount of feed sulfur retained in the glass product, observations of secondary phases, key processing parameters, and measured concentrations of gaseous species. The tests, listed in the order in which they were performed, were as follows:

- Test A: Four nominally 14-hour feeding segments with LAW AP-101/Sub-Envelope A2 simulated wastes with a Na<sub>2</sub>O concentration of 21 wt% in the ORPLG27 target glass composition. The test segments employed SO<sub>3</sub> concentrations of 0.2, 0.4, 0.6, and 0.5 wt% in the glass product (assuming total retention).
- Test B: Four nominally 14-hour feeding segments with LAW AN-105/Sub-Envelope A1 simulated wastes with a Na<sub>2</sub>O concentration of 24 wt% in the ORPLA38-1 target glass composition. The test segments employed SO<sub>3</sub> concentrations of 0.5, 0.7, 0.9, and 0.8 wt% in the glass product (assuming total retention).

The principal objective of these tests was to determine, for each feed, the maximum sulfur content in the feed that can be fed into the melter without forming secondary sulfate phases. The bubbling rate was adjusted to maintain target glass production rates between 2250 and 2500 kg/m<sup>2</sup>/day with a complete cold cap. Test segment durations of 12.6 to 15.25 hours were selected since, at the target glass production rate, this provided three melt pool turnovers (24 kg) for each sulfur concentration. Sugar was added to the feed at a stoichiometric carbon ratio of 0.5 for all of the melter tests. At the end of each test segment, dip samples were taken to detect the presence of a separate sulfur phase on the glass pool surface. The melt surface was considered free of a sulfate layer if no visible secondary sulfate phases were observed on any of the three dip samples. If a sulfate layer was detected on the melt surface, the glass pool was bubbled until the dip samples indicated that the sulfate layer had dissipated prior to commencing the subsequent test segment.

### **3.1 DM10 System Description**

#### **3.1.1 Feed System**

The feed container is mounted on a load cell for weight monitoring and is stirred continuously except for periodic, momentary interruptions during which the weight is recorded. The material in the feed container is constantly recirculated, which provides additional mixing.

The recirculation loop extends to the top of the melter where feed is diverted from the recirculation loop through a peristaltic pump into the melter through a Teflon-lined feed line and vertical water-cooled feed tube. A diverter valve permits direction of the feed stream either to the melter or to a sampling vessel.

### **3.1.2 Melter**

The DM10 system used for this work is a ceramic refractory lined melter, which includes two Inconel 690 plate electrodes that are used for joule-heating of the glass pool and a bubbler for agitating the melt. Glass is discharged from the melter using an air-lift system. The melt pool has a surface area of 0.021 m<sup>2</sup> and typically contains about 8 kg of glass. The plenum volume is 19.5 liters at the nominal glass level. Inconel 690 thermowells were custom fabricated and installed in the DM10 for the current tests since in previous tests, thermowells made from Inconel 601 experienced rapid corrosion in tests with high-alkali, high-sulfur feeds [5].

### **3.1.3 Off-Gas System**

For operational simplicity, the DM10 is equipped with a dry off-gas treatment system involving gas filtration operations only. Exhaust gases leave the melter plenum through a film cooler device that minimizes the formation of solid deposits. The film cooler air has constant flow rate and its temperature is thermostatically controlled. The geometry of the transition line (between the melter and the first filtration device) conforms to the requirements of the 40-CFR-60 air sampling techniques. Immediately downstream of the transition line are cyclonic filters followed by conventional pre-filters and HEPA filters. The temperature of the cyclonic filters is maintained above 150°C while the HEPAs are held above 100°C to prevent moisture condensation. The entire train of gas filtration operations is duplicated and each train is used alternately. An induced draft fan completes the system. The sampling location for gaseous species monitored by FTIR is immediately downstream of the draft fan.

## **3.2 DM10 Test Conditions**

Target processing conditions, including bubbling rate adjusted to maintain a target production rate between 2250 and 2500 kg/m<sup>2</sup>/day, a melt pool temperature of 1150°C, and a complete cold cap were achieved throughout the majority of the melter tests. Test segment average production rates ranged from 2068 to 2401 kg/m<sup>2</sup>/day. All test segments had average production rates within the target range except for the initial test segment, which was about eight percent low. Test segment average bubbling rates ranged from 2.4 to 4.1 liters per minute, which is within the range used in previous tests with the same waste streams [8].

The measured test segment average glass temperatures two inches from the melter floor were between 1144 - 1152°C for all test segments, thus indicating that the target glass temperature of 1150°C was achieved. Measured glass temperatures two inches higher in the glass pool averaged 4°C lower during the tests. Each test segment started with the melt pool at the



nominal operating temperature of 1150°C. Typical plots of DM10 melter temperatures are given in Figures 3.1.a and 3.1.b. The plots are for the first two and the last two test segments (A1, A2, B3, and B4). As mentioned above, the data at 2" from the melter bottom are most representative of the bulk glass temperature; these data average very close to the target of 1150°C and vary little during feeding periods. The measurement at 4" from the bottom is closer to the melt surface and varies by about 25°C as the level of the glass changes. Variations in temperatures measured at both locations were observed during idling periods used to take glass dip samples and purge the melt surface of secondary sulfate phases. In keeping with many previous DM10 tests, the electrode temperatures were 50 to 100°C lower than glass pool temperatures [6, 7]. The measured test segment average plenum temperatures were well below 600°C, indicating that a complete cold cap covered the melt pool surface throughout the tests. Typical plots of DM10 plenum temperatures are given in Figures 3.2.a and 3.2.b for the first two and last two test segments conducted. Higher plenum temperatures occurred at the beginning of testing and in between test segments while the melt pool was probed for secondary sulfate phases. Unlike plenum temperature measurements on larger melters, the exposed thermocouple often gave a lower temperature reading than the thermocouple in the thermowell due to variable amounts of feed coating the exposed thermocouple.

### **3.3 DM10 Test Results**

Evaluation of glass pool samples provided an indication of the tolerance of the glass formulations to sulfur under nominal melter conditions. Depictions of the target and measured sulfur contents are provided in Figure 3.3 for the two test series. During Test A with the ORPLG27 target glass composition, secondary sulfate phases were observed while processing feeds targeted at 0.6 wt% SO<sub>3</sub> but not at 0.2, 0.4, and 0.5 wt% SO<sub>3</sub>; thus the sulfur saturation occurs between 0.5 and 0.6 wt% SO<sub>3</sub> with respect to the feed. The highest sulfur concentration in the glass that did not have secondary phases was 0.47 wt% SO<sub>3</sub>. This result is a significant improvement over past glass compositions for this waste: previous tests with same waste composition showed little tolerance for sulfur with secondary sulfate phases observed at only 0.2 wt% SO<sub>3</sub> [8]. During Test B with the ORPLA38-1 target glass composition, secondary sulfate phases were observed while processing feeds targeted at 0.9 wt% SO<sub>3</sub> but not at 0.5, 0.7, and 0.8 wt% SO<sub>3</sub>; thus the sulfur saturation occurs between 0.8 and 0.9 wt% SO<sub>3</sub>. The highest sulfur concentration in the glass that did not have secondary phases was 0.76 wt% SO<sub>3</sub>. In previous tests with this waste stream, sulfur saturation occurred between 0.7 and 0.8 wt% SO<sub>3</sub> in the feed [8]; thus the glass formulation tested here shows a modest improvement over the previously tested glass formulation.

### **3.4 Gases Monitored by FTIR**

Melter emissions were monitored in each test for a variety of gaseous components, most notably carbon monoxide, ammonia, sulfur dioxide and nitrogen species, by Fourier Transform Infra Red Spectroscopy (FTIR). The off-gas system temperature is maintained well above 100°C beyond the sampling port downstream of the HEPA filter in order to prevent analyte loss due to condensation prior to monitoring. Test segment average concentrations of NO, NO<sub>2</sub>, CO, and

NH<sub>3</sub> are provided in Tables 3.1 and 3.2; these analytes are those that were expected to be observed during the test, based on previous work. No HF, HCl, HCN, SO<sub>2</sub> and nitric acid were detected in any of the tests and therefore none is reported in the tabular data. The most abundant nitrogen species monitored was NO, which is consistent with previous tests [2-8, 13, 14, 21-30] in which nitrates and nitrites were abundant in the feed. The two waste simulants have similar concentrations of nitrogen oxides, and therefore similar sugar additions, and the measured concentrations of most monitored components are similar throughout the two tests. The carbon monoxide and ammonia observed in melter emissions as products of incomplete combustion for LAW simulants are typical while processing relatively high concentrations of nitrate, nitrite and sugar.

## **SECTION 4.0**

### **DM10 GLASS PRODUCTS**

Over a fifth of a metric ton of glass was produced in these tests. The glass was discharged from the melter periodically into square steel cans using an airlift system. The discharged product glass was sampled at the end of each test by removing sufficient glass from the top of the cans for total inorganic analysis. Care was exercised during sampling of each can to identify and segregate any potential secondary phases. Minor amounts of secondary sulfate were observed on the exterior of drips from the end of discharged glasses during Test A2, as shown in Figure 4.1. These occurrences of secondary phase were transient and had no correlation with secondary phases on the glass pool surface. Product glass masses, discharge date, and analyses performed are listed in Table 4.1. Glass samples were also obtained by dipping a rod into the glass pool at the beginning and end of each test. These "dip samples" underwent visual examination to detect the presence of a separate sulfate phase on the glass pool surface.

#### **4.1 Compositional Analysis**

Glass discharge samples were crushed and analyzed directly by XRF. The target values for boron and lithium oxides, which are not determined by XRF, were used for normalizing the XRF data to 100 wt%. The XRF-analyzed compositions of all discharged glass samples are provided in Tables 4.2. The majority of the XRF analysis results compare favorably to their corresponding target values and feed sample analysis (see Section 2.4.2). Of the oxides with target concentrations of one percent or greater, the XRF values had deviations of less than 10% except for the initial test segment, which had a high calcium content due to incomplete turnover and high tin bias of about thirteen to seventeen percent throughout most of the tests with the ORPLA38-1 composition. Measured vanadium concentrations were about ten percent above the target concentration during the tests with the ORPLA38-1 composition once the glass was completely turned over. Chromium oxide concentrations were an absolute 0.2 to 0.25 weight percent above the target; most of this surplus was measured in the feed samples; however, a small increase is attributable to corrosion of melter brick and Inconel components. Iron oxide concentrations were above the low target concentrations due to the ubiquity of the element in bulk chemicals. Elements not included in the target glass compositions, including iodine, manganese, nickel, phosphorus, titanium, and vanadium were observed in the product analysis as a result of corrosion of melter components, carryover from previous tests, and trace contamination of additives.

Compositional trends of the major and select oxides during the tests are shown in Figures 4.2. - 4.9. These trends illustrate the differences between the two tested compositions and closeness to target over the course of the tests. Aside from the intended changes in sulfur, the two glass compositions were held relatively constant during each testing sequence and therefore the most significant changes were observed during the first test segment. These changes for additives calcium, magnesium, vanadium, and tin as well as the waste components sodium,

potassium, and aluminum are evident in the plotted data. Both glass compositions have high target sodium concentrations (20 and 24 wt% oxide), therefore the range is not nearly as extensive as previous tests with the LAW waste streams [6, 8]. These depictions also show the sampling and analytical variations attributable to the methods used; for example, potassium, chlorine, and vanadium show minimal variability whereas zirconium and tin can vary by about half a weight percent in sequential glass discharges. The relative volatility of sulfur in Figure 3.3 and chlorine in Figure 4.9 is illustrated as the difference between the target and measured amounts in the glass; about a third of the chlorine and a tenth of the sulfur was volatilized. This chlorine loss is similar to previous tests with these wastes [6, 8] but less than the near-50% loss of chlorine from the glass at target concentrations greater than 0.3 weight percent [7, 28, 60] in previous tests. This difference may be due to the higher concentration of sodium in the target glasses and possibly higher chlorine contamination of feed constituents. Complete sampling and analysis of melter exhaust commonly conducted on larger melters [2-6, 13, 14, 21-30, 60-64] is required for a more accurate assessment of elemental volatility and mass balance calculations.

## 4.2 Secondary Phase Observations

All discharged glass and glass “dip” samples taken directly from the melt pool were closely examined to document the presence or absence of secondary phases. Glass dip samples were obtained from three separate locations in the melt pool at the end of each test segment; these samples were collected to ascertain whether a secondary sulfate layer had formed on the surface of the glass melt in response to the feed sulfur concentration used for that test segment. Samples were also taken to ensure the melt surface was free of secondary phases prior to starting each test segment, as well as after bubbling intended to volatilize sulfur from a previously formed sulfur layer. Table 4.3 provides a listing of all of the dip samples and an indication of whether or not a separate salt phase was evident. Examples of secondary phases observed while processing three of the formulations are shown in Figures 4.10 – 4.12. Notice the yellow material adhering to the rod and on the outside of the glass, both of which are indicative of a sample taken from a melt pool with a sulfate layer on the surface.

## 4.3 Comparison of the Properties of Crucible and Melter Glasses

Samples of DM10 discharge glasses and crucible melts for the two waste compositions were subjected to the PCT leaching procedure. Samples were collected from melter test segments processing feeds with the highest sulfate concentrations that did not result in secondary sulfate phases. The PCT releases of the melter glasses, along with those of crucible glasses with the same target compositions, are given in Table 4.4. PCT releases of all of the melter and crucible glasses are well below the WTP contract limit mass loss of 2.0 g/m<sup>2</sup> for B, Na and Si. The PCT releases of the melter glasses are, in general, higher than those of the corresponding crucible glasses. Some variations in the PCT releases of glasses of the same composition are expected based on round robin PCT testing of an Argonne National Laboratory-Low Activity Reference Material (ANL-LRM) glass sample [65]. However, the results presented in Table 4.4 show PCT releases of the melter glasses to be uniformly higher than those of the corresponding crucible glasses. This is probably due to the differences in analyzed compositions of the glasses given in

Tables 2.2, 2.12 and 4.2. The melter glasses contain slightly higher concentrations of alkalis ( $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$ ) and lower concentrations of  $\text{SiO}_2$  and  $\text{ZrO}_2$  as compared to the corresponding crucible glasses. Alkalis tend to increase PCT release, whereas  $\text{SiO}_2$  and  $\text{ZrO}_2$  decrease PCT release.

VHT results for the melter and corresponding crucible glasses with the same target composition are given in Table 4.5. VHT alteration rates calculated by two different methods are given in Table 4.5. One method involves direct measurement of the alteration layer thickness, while the other involves measurement of the remaining glass. The VSL Standard Operating Procedure (SOP) for VHT measurement specifies that when the alteration layer thickness is greater than 100  $\mu\text{m}$ , it should be determined based on the remaining glass. This is because when the alteration layer thickness is sufficiently large, measuring the dimensions of the remaining glass and subtracting it from the original sample dimensions gives a better estimate of the thickness of the altered layer. A direct measurement of the layer thickness can provide erroneous values because the altered layer may have expanded, thus giving a larger value than the actual thickness of the altered part of the glass sample, particularly for thick layers. The VHT alteration rates for the crucible and melter samples of ORPLA38 are similar, with the melter glass sample showing a slightly higher value. The melter glass sample of ORPLG27 showed substantially higher VHT alteration rate as compared to the corresponding crucible glass sample, with the melter glass sample exceeding the contractual limit of 50  $\text{g/m}^2/\text{day}$  [53]. The major reason for the difference is the presence of bubbles and cracks in the melter glass samples, which are absent in the crucible glass samples. Excessive alteration around bubbles and cracks in melter glass samples as compared to the crucible glasses can be seen in Figures 4.13.a to 4.14.b. Another potential contributor is that the melter glasses prepared from slurry feeds are likely to contain more OH groups in the structure as compared to the crucible melts prepared from dry chemicals, which would also likely lead to higher VHT alteration rates. A third reason is the differences in analyzed compositions between the crucible and melter glasses discussed above. As is evident from Figures 4.14.a and 4.14.b, bubbles and cracks in this melter glass samples clearly increase the VHT alteration and lead to higher variability in the alteration rate measurement. The difference in the VHT alteration rates between the crucible and melter glass samples of ORPLG27 is clearly higher than the previously reported uncertainty associated with VHT measurements of about 31% relative standard deviation [66]. The difference is mostly due to the presence of cracks and bubbles in the melter glass sample that are not present in the crucible glass sample. In future, one way to reduce the difference in the VHT results from crucible and melter glasses may be to remelt the melter glass samples and subject both the crucible and melter glass samples to CCC heat treatment before performing VHT.

The densities of both the crucible and melter glass samples were measured. The measured densities of all glass samples are below the limit of 3.7  $\text{g/cc}$ . In both cases, the melter glass samples showed slightly lower densities than the corresponding crucible glass samples (2.60  $\text{g/cc}$  versus 2.69  $\text{g/cc}$  for ORPLA38 and 2.67  $\text{g/cc}$  versus 2.71  $\text{g/cc}$  for ORPLG27). This is due to the presence of some gas bubbles in the melter glass samples, whereas the crucible glass samples are free of bubbles.

## SECTION 5.0

### SUMMARY AND CONCLUSIONS

Two sets of tests were conducted on the DM10 vitrification system to evaluate newly developed LAW glass formulations intended to improve waste loading and sulfate tolerance. Glass formulations for the melter tests were selected on the basis of a series of crucible melts that were prepared and characterized. Glasses were formulated for two different waste streams, maximizing waste loading while meeting requirements for product quality (PCT and VHT), refractory corrosion characteristics, and processing properties. The results from this work, in combination with our previous work in this area, provides ORP with a broad basis of glass formulation and characterization data from which to assess the likely enhancements in LAW loadings that should be possible in the WTP LAW design with the current product quality and processability constraints. Such enhancements provide the potential for significant cost and schedule savings. Implications of the results from this work for the WTP and recommendations for future work are provided at the end of this section.

The GFC additives that have been used previously in the LAW glasses are  $\text{Al}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Li}_2\text{O}$ ,  $\text{MgO}$ ,  $\text{SiO}_2$ ,  $\text{SnO}_2$ ,  $\text{TiO}_2$ ,  $\text{V}_2\text{O}_5$ ,  $\text{ZnO}$  and  $\text{ZrO}_2$ . For the present work,  $\text{Fe}_2\text{O}_3$  and  $\text{TiO}_2$  were not used as glass former additives.  $\text{Li}_2\text{O}$  also was not used because the two waste streams that were investigated, LAW AN-105 and LAW AP-101, have very high alkali concentrations ( $\text{Na}_2\text{O}$  and  $\text{K}_2\text{O}$ ) and comparatively lower  $\text{SO}_3$  concentrations.  $\text{ZnO}$  is added at concentrations in the range of 2 to 3 wt% mainly to reduce corrosion of K-3 refractory by the glass. It is also beneficial in reducing corrosion of Inconel components. In general, components such as  $\text{SiO}_2$ ,  $\text{SnO}_2$ , and  $\text{ZrO}_2$  improve the chemical durability of the glass, including performance on the PCT and VHT. Of these,  $\text{ZrO}_2$  and  $\text{SnO}_2$  are the most effective in reducing VHT alteration rates. These components usually are also effective in reducing corrosion of both K-3 and Inconel by the glass. Even though  $\text{Al}_2\text{O}_3$ , in general, tends to improve chemical durability, its substitution by other components such as  $\text{SiO}_2$ ,  $\text{SnO}_2$ , and  $\text{ZrO}_2$  can be beneficial in reducing VHT alteration rates. Increases in  $\text{B}_2\text{O}_3$  concentration can have variable effects on chemical durability and typically tend to reduce melt viscosity.  $\text{Li}_2\text{O}$ ,  $\text{CaO}$ , and  $\text{V}_2\text{O}_5$  all are beneficial in increasing sulfate loading. In the present work, since very high alkali compositions were being formulated, components that reduce corrosion and improve chemical durability were increased.  $\text{Cr}_2\text{O}_3$  was added solely to reduce corrosion of K-3 refractory by the glass. The addition of  $\text{Cr}_2\text{O}_3$  has been quite successful in increasing the waste loading in high alkali LAW glasses, where K-3 refractory corrosion can limit waste loading. However, it is also known that sulfate and chromate show synergistic behavior [67] and the presence of chromium can increase the tendency for secondary sulfate/chromate phase formation, which can potentially limit waste loading in LAW glasses that contain sulfur. It should be noted that the effect of the addition of each of these components cannot be taken in isolation. Each has to be considered in combination with the other glass former additives, the overall composition of the glass, and relevant glass melt properties such as viscosity and glass redox state.

One objective of the current work as specified by ORP [9] was to determine the effect of higher MgO concentrations on the properties of Hanford LAW glasses. Accordingly, glasses with high MgO concentrations (up to 10 wt%) were prepared and characterized. Heat treated glass samples with high MgO concentrations ( $> 3$  wt%) showed small Cr-rich spinel crystals with Mg. Larger sodalite crystals that were present in these samples did not contain Mg. Decreasing the MgO concentration to 1 wt% or less eliminated most of the crystals containing Mg. Previous tests with high Mg concentrations ( $> 5$  wt%) in lower alkali, higher sulfate glasses showed augite-aegirine crystals with Mg, along with  $\text{MgAl}_2\text{O}_4$  spinels [19]. All of the glasses from the present work with MgO contents in excess of 3 wt% failed the contract leaching criteria for VHT in all cases and PCT in a number of cases. The leach rates as a function of MgO content are given in Figure 5.1. This is consistent with previous studies that showed MgO to be one of the components that increases leach rate of borosilicate glasses [20, 54, 58, 68]. Increases in MgO concentrations are predicted to increase leaching of these borosilicate glasses as much as increases in  $\text{Li}_2\text{O}$  and greater than increases in  $\text{Na}_2\text{O}$ . In a glass produced at the Marcoule Vitrification Facility with 6 wt% MgO [54], the precipitation of magnesium phyllosilicate was found to control the significant residual rate of glass alteration [68]. The above phase identification was made after 2500 to 3400 days of leaching. Even though XRD of the material from the current short term (7 day) leach test did not identify such phases, similar behavior after longer term leaching cannot be ruled out for the high MgO glasses. For all of these reasons, MgO concentrations were reduced to 1 wt% or less during the latter part of the present glass development work.

For the current work, glass formulation development and testing were designed such that the maximum achievable waste loadings could be determined for two LAW streams specified by ORP. Details of the LAW glasses selected for the current set of melter tests for the two regions, ORPLA38 and ORPLG27, are summarized below.

**ORPLA38-1:** This glass formulation was based on the composition of LAW from Hanford tank AN-105. The developed glass formulation, ORPLA38-1, has a target waste loading of 31.5 wt%, with target  $\text{Na}_2\text{O}$  and  $\text{SO}_3$  loadings of 24.0 and 0.8 wt%, respectively. The waste loading for the previous ORP glass developed for this LAW stream, ORPLA20 [8], had the same  $\text{Na}_2\text{O}$  and overall waste loadings, but a lower  $\text{SO}_3$  loading of 0.7 wt%. The increase in  $\text{SO}_3$  loading was achieved using  $\text{V}_2\text{O}_5$  as a glass former additive with decreases in the concentrations of  $\text{B}_2\text{O}_3$ ,  $\text{CaO}$  and  $\text{SiO}_2$ . The waste loading of 31.5 wt% in ORPLA20 is higher than that in the corresponding WTP LAW correlation glass, LAWE4H [60] of 27.2 wt%, and the WTP LAW baseline glass, LAWA44 [16] of 26.0 wt%. The nominal composition of LAW from tank AN-105 has a  $\text{SO}_4/\text{Na}$  molar ratio of  $3.0 \times 10^{-3}$ , which equates to an  $\text{SO}_3$  concentration of 0.18 wt% at a  $\text{Na}_2\text{O}$  loading of 24.0 wt%. Glass ORPLA38-1, which can accommodate 0.8 wt%  $\text{SO}_3$  at 24.0 wt%  $\text{Na}_2\text{O}$  loading can, therefore, be used to treat LAW AN-105 type streams with  $\text{SO}_4/\text{Na}$  ratios of up to  $1.33 \times 10^{-2}$ .

**ORPLG27:** This glass formulation was based on the composition of LAW from Hanford tank AP-101. In addition to  $\text{Na}_2\text{O}$  and  $\text{SO}_3$ , this waste has a high concentration of  $\text{K}_2\text{O}$ . The developed glass formulation, ORPLG27, has a target waste loading of 29.1 wt%, with target  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$  and  $\text{SO}_3$  loadings of 21.0, 5.8 and 0.5 wt%, respectively. ORPLG9, which was previously selected to treat this waste stream, has the same waste loading and alkali

concentrations, but a much lower sulfate loading of 0.2 wt%. The waste loading of 29.1 wt% in ORPLG27 is higher than that in the corresponding WTP LAW correlation glass, LAWE3 [60] of 25.4 wt%, and the WTP LAW baseline glass, LAWA126 [17], of 24.5 wt%. Due to the high alkali content, the properties of concern for glass formulation were VHT alteration rate and K-3 refractory corrosion. In this glass, in order to minimize K-3 corrosion,  $\text{Cr}_2\text{O}_3$  was kept at a concentration of 0.59 wt%. The measured  $\text{SO}_3$  solubility in the crucible glass was 0.49 wt% by batch saturation tests and 0.58 wt% by bubbling. During DM10 melter tests, a secondary sulfate layer developed at a sulfate concentration of 0.6 wt%.

At the very high  $\text{Na}_2\text{O}$  loadings (23 wt% or higher), VHT response becomes especially challenging due to the rapid increase of VHT alteration rate with increasing alkali content, the increased variability in VHT response at high alkali content, and increased VHT alteration rates due to cracking in melter glasses. Achieving significantly higher  $\text{SO}_3$  loadings in borosilicate glasses would appear to be unlikely without changing the processing conditions or the processing and/or product quality constraints (e.g., it is a simple matter to achieve higher  $\text{SO}_3$  loadings by increasing the contents of Li, Ca, V, etc., but such glasses do not meet VHT and refractory corrosion requirements). Thus, the glasses identified during the current and previous work [7, 8] serve to define the likely limits of possible  $\text{Na}_2\text{O}$  and  $\text{SO}_3$  loadings in Hanford LAW glasses that are compliant with the current product quality and processing requirements. It should be noted, however, that these glasses were tested only at the crucible and DM10 melter scales. Additional testing at larger scales is required to confirm the results from smaller scale testing and the results of such testing may result in refinement of these limits. It should also be noted that because of the bounding nature of the formulations (they are deliberately close to the limits of the requirements), practically viable operating points would fall at somewhat lower waste loadings since nominal glass compositions selected for waste processing need to accommodate process variations without adverse effects on processing or product quality. In addition, the corrosion rate of Inconel in these new glass compositions has not been tested. While experience suggests that the adjustments made to the glass compositions to reduce VHT alteration rate and K-3 corrosion, along with the higher viscosity, will also maintain acceptable Inconel corrosion rates, this needs to be confirmed through testing.

## 5.1 Implications for WTP

VSL and EnergySolutions have previously developed and tested a number of LAW glass formulations for ORP [4-8] and WTP [15-20]. The formulations that were developed to support the WTP baseline, as distinct from those developed for ORP that were directed towards enhancements to that baseline, were tested at the crucible scale and at various melter scales, including the one-third scale LAW Pilot Melter at EnergySolutions. As a result of the considerable testing completed with the WTP baseline formulations, there is high confidence that they can be used to process LAW at Hanford with little additional testing. The recommended glass compositions for LAW processing were selected such that they can tolerate process variations without adverse effects on processing or product quality. Based on these well-tested formulations, VSL developed a LAW glass formulation correlation that is currently being used by the WTP [43]. Compositions produced by this correlation fall along the dotted lines in Figure 5.2. The WTP baseline formulations were developed to comply with the requirements of the



Bechtel contract with ORP [53]. Although these formulations are fully compliant, extensive further optimization with respect to waste loading could not be performed due to the schedule constraints imposed by the LAW Pilot Melter testing program defined by the WTP Project. As a result, while this extensive basis set of formulations provides a solid underpinning of the WTP baseline, there is also potential for improvement of waste loadings. Exploiting this potential has been the subject of the present and previous work for ORP.

LAW testing for ORP at VSL and EnergySolutions was aimed at optimizing the glass formulations and processing parameters in order to minimize the volume of glass produced and to shorten the plant operating schedule. An important objective was to determine the likely limits of LAW loading across the full range of expected LAW compositions. During the current work, increases in sulfate loadings were pursued for two Hanford LAW streams, AN-105 and AP-101. A compilation of the target sodium, potassium, sulfur, and overall waste loading achieved for the two glass compositions from the present work and previous glasses developed for ORP and WTP is given in Table 5.1. An overview of the  $\text{Na}_2\text{O}$  and  $\text{SO}_3$  loadings for the WTP and ORP glasses is given in Figure 5.2. As is evident from Figure 5.2 and Table 5.1, these glasses represent considerable increases in waste loadings over the WTP baseline and therefore considerable potential for reductions in cost and schedule. The principal objective of the present work was to assess the likely limits to the extent of this improvement for two LAW streams specified by ORP. Results from the present work combined with previous work for ORP [5-8], show the potential for a 58% increase in average  $\text{Na}_2\text{O}$  loading in Hanford LAW glasses from the WTP baseline of approximately 13 wt% to about 20.6 wt%. This increase in waste loading has the potential to reduce the amount of LAW glass produced at Hanford by over 230,000 MT depending on the amount of process sodium additions. Importantly, these enhancements should be directly applicable to the planned supplemental treatment facility. Furthermore, since the approach adopted in the work for ORP spans likely extremes in the expected LAW compositions, the results would remain relevant even if various LAW tank wastes are blended, since blending generally has the effect of affording yet further improvements in waste loadings.

## 5.2 Recommendations for Future work

In much of the earlier high-alkali LAW glass development work, VHT response was the most constraining property. However, as the database for VHT response for high-alkali LAW glasses has grown and the performance of the VHT model in the composition region has improved [66], there have been corresponding increases in waste loadings for high-alkali glasses with acceptable VHT response. As a result, recent work indicates that K-3 refractory corrosion is now at least as constraining as VHT response for high-alkali LAW glasses. Consequently, more data on this important property and models for K-3 corrosion would be useful.

As discussed in a previous report [8], the high sulfate LAW formulations (LAWB99, ORPLE12 and ORPLF7) all show sulfate loading of 1.5 wt%, even though the sodium loadings vary from 10 to 16 wt%. Two of the three glasses (ORPLE12 and ORPLF7) contain  $\text{Cr}_2\text{O}_3$  as an additive. It would be useful to determine whether sulfate loading in these glasses can be improved if  $\text{Cr}_2\text{O}_3$  is not used as an additive.

Bubbles and cracks that are present in melter glass samples have significant effect on the measured VHT response, with the result that the measured alteration rate may not necessarily be related solely to the glass composition. This appears to be particularly important for high-alkali glasses with VHT response near the WTP contract limit. Better comparison between crucible and melter glasses may be possible if samples that are remelted and subjected to CCC heat treatment are used to perform the VHT.

The LAW correlation was developed for the WTP by VSL/EnergySolutions to determine the types and amounts of glass forming chemicals (GFCs) to be used at the WTP for LAW processing under the current WTP baseline. This was possible only after the completion of much more extensive testing than has been done for the new ORP glasses and after a set of nominal Sub-Envelope formulations were refined. The data collected so far for the ORP higher waste loading glasses are not yet sufficiently extensive to support a revised LAW correlation algorithm, but does serve to define what ranges of enhanced waste loadings should be possible. However, once sufficient data are collected, a new LAW formulation correlation similar to the one currently being used by the WTP needs to be developed to support the implementation of these higher waste loading glass compositions at the WTP in order to realize the cost and schedule reductions. The steps needed to collect the data to support a new LAW correlation include:

- Define GFC additives to be used at the WTP.
- Refine LAW glass formulations using the specified GFC additives.
- Refine waste loading in LAW glasses to accommodate the effect of halides.
- Specify waste loading limits for LAW formulations to accommodate process variations.
- Perform scale-up testing (DM100 and/or DM1200).

Since the process for producing such a revised correlation would closely parallel the one used successfully to develop the current WTP baseline, there is high confidence in the success of this approach.

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**Table 1.1. Waste Compositions and Corresponding Waste Loadings in Glass.**

<b>ORP Composition Region Designation</b>	<b>Tank / Sub-Envelope Identification</b>	<b>Correlation Formulation Identification</b>	<b>Target Minimum Waste Loading (wt%)</b>
A	AN-105/ Sub-Envelope A1	LAWE4H	27.18
G	AP-101/ Sub-Envelope A2	LAWE3	25.41

**Table 1.2. Glass Processing and Product Quality Requirements.**

Property	Requirement(s)
Density	< 3.7 g/ml
Liquidus Temperature	< 950°C
LAW Canister Centerline Cooling Heat Treatment	Report amount of crystals
PCT per ASTM C1285 Test conducted at glass to water ratio of 1 gram of glass (-100 +200 mesh) per 10 ml of water at 90°C B (g/m <sup>2</sup> ) Na (g/m <sup>2</sup> ) Si (g/m <sup>2</sup> )	< 2.0 g/m <sup>2</sup>
VHT at 200°C (g/m <sup>2</sup> /day) for 24 days	< 50 g/m <sup>2</sup> /day
Viscosity (poise) at 1100°C	10 to 150 P
Electrical Conductivity (S/cm) at 1100°C	0.1 to 0.7 S/cm
Glass Transition T <sub>G</sub> (onset)	Report T <sub>G</sub>
K-3 Refractory Corrosion	For WTP LAW glass formulation development, a neck corrosion of 0.035 inches on 6-day K-3 coupon corrosion test at 1208°C has been used as an acceptance limit. For the current LAW glass formulation development work for ORP, since higher waste loading compositions are being explored, a slightly higher neck corrosion value of 0.040 inches was used as a guide for acceptable corrosion characteristics.

**Table 2.1. LAW Sub-Envelope A1 for AN-105 Waste Simulant Recipe at Nominal 8 Molar Sodium.**

Envelope Constituents	Simulant for AN-105 including pretreatment		Glass Oxides	AN-105 Simulant as Oxides (wt%)	Source in Simulant	Order for Addition	Formula Weight	Assay*	Target Weight (g)
-	mg/L	M	Loading		In 274 ml water add following compounds in the order listed below				
Al	30554	1.132	Al <sub>2</sub> O <sub>3</sub>	17.613	Al(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O, 60% sol.	1	375.14	0.61	422.01
					Al(OH) <sub>3</sub>	7	78.00	1.00	35.56
B	79	0.007	B <sub>2</sub> O <sub>3</sub>	0.077	H <sub>3</sub> BO <sub>3</sub>	2	61.83	0.99	0.45
Cr	149	0.003	Cr <sub>2</sub> O <sub>3</sub>	0.066	Na <sub>2</sub> CrO <sub>4</sub> ·4H <sub>2</sub> O	6	234.04	0.99	0.68
K	4608	0.118	K <sub>2</sub> O	1.694	KOH	5	56.10	0.91	7.28
Na	183920	8.000	Na <sub>2</sub> O	75.638	NaOH, 50% sol. d=1.53	4	40.00	0.50	449.56
Si	157	0.006	SiO <sub>2</sub>	0.102	SiO <sub>2</sub>	3	60.09	0.99	0.34
Cl	6996	0.197	Cl	2.134	NaCl	8	58.45	0.99	11.65
F	35	0.002	F	0.011	NaF	9	42.00	0.99	0.08
SO <sub>4</sub> (Nominal) <sup>§</sup>	10488	0.109	SO <sub>3</sub>	2.667	Na <sub>2</sub> SO <sub>4</sub>	10	142.06	0.99	15.67
NO <sub>2</sub>	85428	1.857	-	-	NaNO <sub>2</sub>	14	69.00	0.97	128.79
NO <sub>3</sub>	126988	2.048	-	-	NaNO <sub>3</sub>	-	84.99	0.99	0.00
TOC	2093	0.174	-	-	-	-	-	-	-
Acetate	2251	0.038	-	-	Sodium Acetate (C2)	11	136.08	0.99	5.24
Formate	2135	0.047	-	-	Sodium Formate (C1)	12	68.01	0.99	3.26
Glycolate	1936	0.025	-	-	Glycolic Acid (C2)	13	76.05	0.71	2.73
-	-	-	SUM	100.00	Total simulant wt.				1358.73

– Empty data field.

\* – Assay refers to the purity of the raw material as specified by the vendor.

**Table 2.2. Target and Analyzed Compositions (wt%) of Fifteen ORPLA Crucible Glasses.**

Glass	ORPLA26		ORPLA27		ORPLA28		ORPLA29		ORPLA30	
Oxides	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*
Al <sub>2</sub> O <sub>3</sub>	10.57	9.77	10.33	9.74	10.10	9.20	9.87	8.82	9.56	8.83
B <sub>2</sub> O <sub>3</sub>	12.77	12.78	12.20	12.28	11.64	11.74	11.08	11.08	11.73	11.73
CaO	6.47	6.91	6.18	6.86	5.90	6.60	5.62	5.94	5.43	5.93
Cr <sub>2</sub> O <sub>3</sub>	0.52	0.70	0.50	0.69	0.48	0.74	0.45	0.79	0.52	0.74
Fe <sub>2</sub> O <sub>3</sub>	0.90	1.03	0.86	1.03	0.82	0.99	0.78	0.87	0.90	1.05
K <sub>2</sub> O	0.51	0.48	0.51	0.48	0.51	0.52	0.51	0.51	0.51	0.49
MgO	0.90	1.03	4.00	4.14	7.00	7.06	10.00	10.07	4.00	4.02
Na <sub>2</sub> O	23.00	23.80	23.00	22.93	23.00	22.26	23.00	23.62	23.00	23.21
SiO <sub>2</sub>	34.80	33.86	33.23	32.94	31.71	30.91	30.19	28.38	34.80	33.84
SnO <sub>2</sub>	1.00	0.97	0.96	0.84	0.91	0.98	0.87	0.89	1.00	1.11
TiO <sub>2</sub>	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.00
V <sub>2</sub> O <sub>5</sub>	0.97	1.03	0.93	1.03	0.89	1.00	0.84	0.91	0.97	1.06
ZnO	2.99	3.16	2.86	3.07	2.72	3.07	2.59	2.70	2.99	3.26
ZrO <sub>2</sub>	2.99	2.94	2.86	2.70	2.72	2.94	2.59	2.58	2.99	3.11
Cl	0.64	0.58	0.64	0.40	0.64	0.55	0.64	0.54	0.64	0.57
P <sub>2</sub> O <sub>5</sub>	0.00	0.01	0.00	0.01	0.00	0.02	0.00	0.01	0.00	0.00
SO <sub>3</sub>	0.95	0.83	0.95	0.82	0.95	1.41	0.95	2.17	0.95	0.88
Sum	100.0	99.9	100.0	100.0	100.0	100.0	100.0	99.9	100.0	99.8

\* – Analyzed by X-ray fluorescence except for boron which was measured by DCP

**Table 2.2. Target and Analyzed Compositions (wt%) of Fifteen ORPLA Crucible Glasses (continued).**

Glass	ORPLA31		ORPLA32		ORPLA33		ORPLA33-1		ORPLA34	
Oxides	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*
Al <sub>2</sub> O <sub>3</sub>	8.65	8.14	7.73	7.14	6.16	5.67	6.73	6.21	7.26	6.67
B <sub>2</sub> O <sub>3</sub>	10.68	10.77	9.64	9.62	8.95	9.14	8.90	8.27	10.07	10.09
CaO	4.39	4.59	3.34	3.52	3.20	3.65	3.18	3.49	4.21	4.57
Cr <sub>2</sub> O <sub>3</sub>	0.52	0.68	0.52	0.71	0.50	0.72	0.50	0.69	0.50	0.68
Fe <sub>2</sub> O <sub>3</sub>	0.90	0.96	0.90	0.96	0.26	0.33	0.26	0.32	0.47	0.54
K <sub>2</sub> O	0.51	0.47	0.51	0.49	0.51	0.53	0.51	0.55	0.51	0.51
MgO	7.00	7.29	10.00	10.32	1.01	0.99	1.00	1.04	2.01	2.11
Na <sub>2</sub> O	23.00	23.35	23.00	23.45	23.14	22.29	23.00	22.37	23.17	23.20
SiO <sub>2</sub>	34.80	34.36	34.80	33.87	42.49	42.39	42.23	41.84	39.52	38.89
SnO <sub>2</sub>	1.00	1.00	1.00	0.99	2.47	2.39	2.46	2.68	1.97	2.07
TiO <sub>2</sub>	0.00	0.01	0.00	0.00	0.00	0.01	0.00	0.02	0.00	0.01
V <sub>2</sub> O <sub>5</sub>	0.97	0.97	0.97	0.97	0.94	1.03	0.93	0.99	0.94	0.97
ZnO	2.99	2.89	2.99	2.91	2.87	3.17	2.86	3.06	2.88	3.02
ZrO <sub>2</sub>	2.99	2.77	2.99	2.77	5.89	6.08	5.86	6.08	4.89	4.98
Cl	0.64	0.55	0.64	0.53	0.64	0.66	0.64	0.63	0.64	0.62
P <sub>2</sub> O <sub>5</sub>	0.00	0.00	0.00	0.02	0.00	0.01	0.00	0.01	0.00	0.00
SO <sub>3</sub>	0.95	1.15	0.95	1.57	0.96	0.82	0.95	0.81	0.96	0.82
Sum	100.0	100.0	100.0	99.8	100.0	99.9	100.0	99.1	100.0	99.8

\* – Analyzed by X-ray fluorescence except for boron which was measured by DCP

**Table 2.2. Target and Analyzed Compositions (wt%) of Fifteen ORPLA Crucible Glasses (continued).**

Glass	ORPLA35		ORPLA36		ORPLA37		ORPLA38		ORPLA38-1	
Oxides	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*
Al <sub>2</sub> O <sub>3</sub>	8.37	7.72	6.35	5.87	6.35	5.88	6.35	5.82	6.94	6.38
B <sub>2</sub> O <sub>3</sub>	11.19	11.52	8.78	9.29	8.26	8.59	8.26	8.76	8.21	7.85
CaO	5.23	5.54	3.14	3.60	3.14	3.44	3.14	3.49	3.12	3.49
Cr <sub>2</sub> O <sub>3</sub>	0.50	0.66	0.49	0.70	0.49	0.67	0.49	0.70	0.49	0.70
Fe <sub>2</sub> O <sub>3</sub>	0.67	0.73	0.26	0.33	0.26	0.32	0.26	0.32	0.26	0.32
K <sub>2</sub> O	0.51	0.50	0.53	0.55	0.53	0.53	0.53	0.53	0.53	0.56
MgO	3.03	3.28	0.99	1.04	1.98	2.22	0.99	1.00	0.98	1.01
Na <sub>2</sub> O	23.19	23.89	24.15	23.42	24.15	24.13	24.15	23.50	24.00	22.94
SiO <sub>2</sub>	36.54	35.94	41.69	41.28	41.22	40.88	41.70	41.18	41.43	41.25
SnO <sub>2</sub>	1.47	1.37	2.42	2.46	2.42	2.28	2.68	2.95	2.66	2.96
TiO <sub>2</sub>	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.02
V <sub>2</sub> O <sub>5</sub>	0.94	0.94	0.92	1.02	0.92	0.98	0.92	0.99	0.91	0.98
ZnO	2.88	2.90	2.82	3.12	2.82	2.99	2.82	3.06	2.80	3.02
ZrO <sub>2</sub>	3.89	3.68	5.78	5.98	5.78	5.59	6.04	6.33	6.00	6.38
Cl	0.65	0.58	0.67	0.70	0.67	0.66	0.67	0.64	0.67	0.62
P <sub>2</sub> O <sub>5</sub>	0.00	0.00	0.00	0.01	0.00	0.01	0.00	0.00	0.00	0.01
SO <sub>3</sub>	0.96	0.88	0.99	0.85	0.99	0.87	0.99	0.88	0.99	0.83
Sum	100.0	100.1	100.0	100.2	100.0	100.1	100.0	100.2	100.0	99.3

\* - Analyzed by X-ray fluorescence except for boron which was measured by DCP

**Table 2.3. Descriptions of Fifteen As-Melted and Heat Treated ORPLA Crucible Glasses.**

<b>Glass ID</b>	<b>As-melted glass</b>	<b>Heat Treatment - Glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 950°C, and quenched.</b>
ORPLA26	Clear glass	Clear glass with few small particles
ORPLA27	green opaque glass	~2.7 vol% large sodalite <sup>s</sup> crystals with Mg, Sn, Zn, & Fe
ORPLA28	partially crystallized brown glass	~3.8 vol% large sodalite crystals with Mg, Sn, Zn, & Fe
ORPLA29	partially crystallized brown glass	~2.5 vol% large sodalite crystals with Mg, Sn, Zn, & Fe
ORPLA30	green opaque glass	~0.3 vol% small sodalite crystals with Mg, Sn, Zn, & Fe
ORPLA31	partially crystallized brown glass	~0.4 vol% sodalite crystals with Mg, Sn, Zn, & Fe
ORPLA32	partially crystallized brown glass	~0.3 vol% sodalite crystals with Mg, Sn, Zn, & Fe
ORPLA33	Clear glass	Clear glass
ORPLA34	Clear glass	Clear glass with few small particles
ORPLA35	Clear glass	Clear glass
ORPLA36	Clear glass	Clear glass with few Cr-containing crystals
ORPLA37	Clear glass	Clear glass
ORPLA38	Clear glass	Clear glass
ORPLA33-1	Clear glass	Clear glass with few Cr-containing crystals Zn- rich crystal clusters
ORPLA38-1	Clear glass	Clear glass with few small Cr-containing crystals

<sup>s</sup>Sodalite formula (Na, Ca)<sub>8</sub>(AlSiO<sub>4</sub>)<sub>6</sub>(SO<sub>4</sub>)<sub>2</sub>



**Table 2.4. Measured Sulfate Solubility in Fifteen ORPLA Crucible Glasses.**

Sample ID	SO <sub>3</sub> Content (wt%)	
	Batch Saturation	
	As-Melted	After Acid Wash
ORPLA26S4	1.05	0.88
ORPLA27S4	1.03	0.79
ORPLA28S4	0.94	0.62
ORPLA29S4	0.89	0.44
ORPLA30S4	0.95	0.72
ORPLA31S4	0.83	0.62
ORPLA32S4	0.71	0.56
ORPLA33S4	0.82	0.68
ORPLA33-1S4	0.88	0.67
ORPLA34S4	0.90	0.65
ORPLA35S4	1.00	0.70
ORPLA36S4	0.93	0.71
ORPLA37S4	1.00	0.68
ORPLA38S4	1.03	0.72
ORPLA38-1S4	0.88	0.71

**Table 2.5. Results of 7-day PCT (at 90°C) and VHT (at 200°C for 24 Days) for Fifteen ORPLA Crucible Glasses.**

Glass ID	ORPLA26	ORPLA27	ORPLA28	ORPLA29	ORPLA30	ORPLA31	ORPLA32	ORPLA33
<b>7-Day PCT, Stainless Steel Vessel; S/V=2000 m<sup>-1</sup> (ppm)</b>								
B	73.15	208.10	336.60	239.10	208.30	515.00	349.60	53.70
Na	264.70	674.10	1195.00	947.50	627.70	1820.00	1402.00	278.30
Si	63.40	101.30	97.55	61.68	118.90	172.60	129.20	107.50
<b>Normalized Concentrations (g/L)</b>								
B	1.84	5.49	9.32	6.95	5.72	15.52	11.68	1.93
Na	1.55	3.95	7.00	5.55	3.68	10.67	8.22	1.62
Si	0.39	0.65	0.66	0.44	0.73	1.06	0.79	0.54
pH	11.71	12.15	12.41	12.44	12.16	12.51	12.54	11.70
<b>7-Day PCT Normalized Mass Loss (g/m<sup>2</sup>)</b>								
B	0.92	2.75	4.66	3.47	2.86	7.76	5.84	0.97
Na	0.78	1.98	3.50	2.78	1.84	5.33	4.11	0.81
Si	0.19	0.33	0.33	0.22	0.37	0.53	0.40	0.27
<b>7-Day PCT Normalized Loss Rate (g/d/m<sup>2</sup>)</b>								
B	0.13	0.39	0.67	0.50	0.41	1.11	0.83	0.14
Na	0.11	0.28	0.50	0.40	0.26	0.76	0.59	0.12
Si	0.03	0.05	0.05	0.03	0.05	0.08	0.06	0.04
<b>VHT Alteration (24 days at 200°C)</b>								
Alteration Depth (μm)	459	1305	1012	633	1320	1360	982	5
Alteration Rate (g/m <sup>2</sup> /day)*	51	144	112	70	146	150	108	1

\* Alteration rates calculated using estimated density of 2.65 g/cc

**Table 2.5. Results of 7-day PCT (at 90°C) and VHT (at 200°C for 24 Days) for Fifteen ORPLA Crucible Glasses (continued).**

Glass ID	ORPLA33-1	ORPLA34	ORPLA35	ORPLA36	ORPLA37	ORPLA38	ORPLA38-1
<b>7-Day PCT, Stainless Steel Vessel; S/V=2000 m<sup>-1</sup> (ppm)</b>							
B	36.90	81.23	191.21	77.82	117.67	42.49	37.11
Na	205.87	354.58	693.53	393.22	570.05	264.88	240.74
Si	83.92	101.53	136.69	132.79	180.49	102.29	88.53
<b>Normalized Concentrations (g/L)</b>							
B	1.34	2.60	5.50	2.85	4.59	1.66	1.46
Na	1.21	2.06	4.03	2.19	3.18	1.48	1.35
Si	0.43	0.55	0.80	0.68	0.94	0.52	0.46
pH	11.78	11.68	11.85	12.10	11.93	12.05	11.78
<b>7-Day PCT Normalized Mass Loss (g/m<sup>2</sup>)</b>							
B	0.67	1.30	2.75	1.43	2.29	0.83	0.73
Na	0.60	1.03	2.02	1.10	1.59	0.74	0.68
Si	0.21	0.27	0.40	0.34	0.47	0.26	0.23
<b>7-Day PCT Normalized Loss Rate (g/d/m<sup>2</sup>)</b>							
B	0.10	0.19	0.39	0.20	0.33	0.12	0.10
Na	0.09	0.15	0.29	0.16	0.23	0.11	0.10
Si	0.03	0.04	0.06	0.05	0.07	0.04	0.03
<b>VHT Alteration (24 days at 200°C)</b>							
Alteration Depth (μm)	8	103	1066	57	125	90	71
Alteration Rate (g/m <sup>2</sup> /day)*	1	11	118	6	14	10	8

\* Alteration rates calculated using estimated density of 2.65 g/cc

**Table 2.6. Viscosities and Electrical Conductivities of Two ORPLA Crucible Glasses.**

Glass ID	ORPLA33-1	ORPLA38-1
<b>Viscosity (poise)</b>		
900°C	2611	2440
950°C	950	905
1000°C	403	388
1050°C	193	186
1100°C	102	98
1150°C	58	56
1200°C	35	34
1250°C	23	22
<b>Electrical Conductivity (S/cm)</b>		
900°C	0.225	0.269
950°C	0.276	0.329
1000°C	0.333	0.396
1050°C	0.397	0.471
1100°C	0.466	0.552
1150°C	0.541	0.641
1200°C	0.623	0.736
1250°C	0.709	0.837

**Table 2.7. Results of K-3 Corrosion Testing for the Selected ORPLA38-1 Crucible Glass.**

Glass ID	Neck loss (inches)	Depth of altered zone (inches)	Half-down loss (inches)
ORPLA38-1	0.0435	0.033	0.000

**Table 2.8. Summary of Test Results for Selected ORPLA Glass Formulation ORPLA38-1 and Comparison to ILAW Requirements.**

Test	Requirement [52, 53]	Test Result for ORPLA38-1
Density of glass	< 3.7 g/cc	2.60 to 2.69 g/cc *
Crystalline Phase	Phase identification	Clear homogeneous glass after heat treatment at 950°C for 20 hours
Liquidus	< 950°C	< 950°C
Centerline Canister Cooling	Phase identification	Not measured
PCT B (g/m <sup>2</sup> )	< 2.0 g/m <sup>2</sup>	0.73 g/m <sup>2</sup>
PCT Na (g/m <sup>2</sup> )	< 2.0 g/m <sup>2</sup>	0.68 g/m <sup>2</sup>
PCT Si (g/m <sup>2</sup> )	< 2.0 g/m <sup>2</sup>	0.23 g/m <sup>2</sup>
VHT at 200°C (g/m <sup>2</sup> /day)	< 50 g/m <sup>2</sup> /day	8 g/m <sup>2</sup> /day
Viscosity (poise) at 1100°C	10 to 150 P	98 P
Conductivity (S/cm) at 1100°C	0.2 to 0.7 S/cm	0.552 S/cm
T <sub>G</sub> (°C)	Report for modeling	Not measured

\* Density measured for melter glass J10-G-24B (2.60 g/cc) and crucible glass ORPLA38-1 (2.69 g/cc).

**Table 2.9. Oxide Composition of LAW AN-105 Simulant and ORPLA38-1 Glass Composition Used in Melter Tests (wt%).**

Component	AN-105 waste contribution	Glass former additives	ORPLA38-1 (for AN-105)
Loading	31.5%	68.5%	-
Al <sub>2</sub> O <sub>3</sub>	5.44	1.51	6.95
B <sub>2</sub> O <sub>3</sub>	0.02	8.20	8.22
CaO	-	3.13	3.13
Cr <sub>2</sub> O <sub>3</sub>	0.02	0.47	0.49
Fe <sub>2</sub> O <sub>3</sub>	-	0.26	0.26
K <sub>2</sub> O	0.52	-	0.52
MgO	-	0.98	0.98
Na <sub>2</sub> O <sup>(a)</sup>	23.35 + 0.62 <sup>(1)</sup> + 0.03 <sup>(2)</sup>	-	24.00
SiO <sub>2</sub>	0.03	41.52	41.55
SnO <sub>2</sub>	-	2.67	2.67
V <sub>2</sub> O <sub>5</sub>	-	0.92	0.92
ZnO	-	2.82	2.82
ZrO <sub>2</sub>	-	6.03	6.03
Cl	0.66	-	0.66
F	0.00	-	0.00
P <sub>2</sub> O <sub>5</sub>	0.00	-	0.00
SO <sub>3</sub> <sup>(b)</sup>	0.80 <sup>(1)</sup>	-	0.80
SUM	31.5	68.5	100.0

(a) Simulant was prepared at a concentration of 23.35 wt% Na<sub>2</sub>O and modified before each melter test with (1) Na<sub>2</sub>SO<sub>4</sub> and (2) NaOH additions to obtain 24 wt% Na<sub>2</sub>O in the glass.

(b) Concentration of SO<sub>3</sub> was increased in steps during the melter tests from 0.5 wt% SO<sub>3</sub> in the glass up to 0.9 wt% and back to 0.8 wt%.

– Empty data field

**Table 2.10a. Glass Former Additives for 1 Liter of AN-105 Simulant (8 M Na) and Corresponding Melter Feed Properties.**

Additives Source	Feed ORPLA38-1
Additives in Glass (wt%)	68.5%
Kyanite ( $\text{Al}_2\text{SiO}_5$ ) 325 Mesh (Kyanite Mining) (g)	23.19
$\text{H}_3\text{BO}_3$ (US Borax – Technical Granular) (g)	150.49
Wollastonite NYAD 325 Mesh (NYCO Minerals) (g)	68.21
$\text{Cr}_2\text{O}_3$ oxide	4.97 <sup>(1)</sup>
$\text{Fe}_2\text{O}_3$ Alfa Aesar (g)	0.67
Olivine ( $\text{Mg}_2\text{SiO}_4$ ) 325 Mesh (#180 Unimin) (g)	21.28
$\text{SiO}_2$ (Sil-co-Sil 75 US Silica) (g)	344.53
$\text{SnO}_2$ - Stannous Oxide - Mason Color <sup>(2)</sup>	27.79
$\text{V}_2\text{O}_5$ - PULVA ground STRATCOR	9.48
ZnO (KADOX – 920 Zinc Corp. of America) (g)	29.09
Zircon $\text{ZrSiO}_4$ (Flour) Mesh 325 (AM. Mineral) (g)	93.47
$\text{Na}_2\text{SO}_4$	Variable – Table 2.10b
Addition of Sucrose as Reductant (g)	78.5
Simulant Weight for 1 liter (g)	1359
Sum of Additives (g)	773.16
Sum of Complete Batch (g)	2115
Target Final Volume (l)	1.38
Estimated Density (g/ml)	1.61
Target Glass Produced (g)	1033
Target Weight % Additives in Slurry	39
Target Glass Yield (g/kg of Feed)	471
Target Glass Yield (g/l of Feed)	758
Target Total Solids (g/l of Feed)	969
Target Additives (g/l of Feed)	625

<sup>(1)</sup> Note that a  $\text{Cr}_2\text{O}_3$  addition was reduced by 50% to account for K3-brick contribution.

<sup>(2)</sup> Note that  $\text{SnO}_2$  from Mason Color was complemented by Alfa product, which included iodine as an impurity

**Table 2.10b. NaOH and  $\text{Na}_2\text{SO}_4$  Additions Required to Obtain 24 wt%  $\text{Na}_2\text{O}$  and Various  $\text{SO}_3$  Concentrations in the ORPLA38-1 Glass.**

Final $\text{SO}_3$ (wt%)	NaOH needed per kg of feed (grams)	$\text{Na}_2\text{SO}_4$ needed per kg of feed (grams)
0.5	3.25	4.22
0.6	2.31	5.07
0.7	1.37	5.91
0.8	0.43	6.73
0.9	0.00	7.60

**Table 2.11. LAW Sub-Envelope A2 (AP-101) Waste Simulant Recipe at 8 Molar Sodium.**

Envelope Constituents	Simulant AP-101 Including Pretreatment		Glass Oxides	Simulant as Oxides (wt%)	Source in Simulant	Order for Addition	Formula Weight	Assay*	Target Weight (g)
-	mg/L	Molarity	Loading	100%	In 494 ml water add following compounds in the order listed below				
Al	9625	0.357	Al <sub>2</sub> O <sub>3</sub>	5.26	Al(NO <sub>3</sub> ) <sub>3</sub> ·9H <sub>2</sub> O, 60% sol.	1	375.14	0.61	220.51
B	12	0.001	B <sub>2</sub> O <sub>3</sub>	0.01	H <sub>3</sub> BO <sub>3</sub>	2	61.83	0.99	0.07
Cr	749	0.014	Cr <sub>2</sub> O <sub>3</sub>	0.32	Na <sub>2</sub> CrO <sub>4</sub> ·4H <sub>2</sub> O	8	234.04	0.99	3.42
K	56301	1.440	K <sub>2</sub> O	19.61	KOH	7	56.10	0.91	88.97
Na	183920	8.000 <sup>\$</sup>	Na <sub>2</sub> O	71.67	NaOH, 50% sol. d=1.53	6	40.00	0.50	226.00
Ni	82	0.001	NiO	0.03	NiO	3	74.69	1.00	0.10
Pb	95	0.0005	PbO	0.03	PbO	4	223.20	1.00	0.10
Si	157	0.006	SiO <sub>2</sub>	0.10	SiO <sub>2</sub>	5	60.09	0.99	0.34
Cl	2730	0.077	Cl	0.79	NaCl	10	58.45	0.99	4.55
F	1083	0.057	F	0.31	NaF	11	42.00	0.99	2.42
PO <sub>4</sub>	2251	0.024	P <sub>2</sub> O <sub>5</sub>	0.49	Na <sub>3</sub> PO <sub>4</sub> ·12H <sub>2</sub> O	9	380.12	0.99	9.10
SO <sub>4</sub> (Nominal) <sup>\$</sup>	5764	0.060	SO <sub>3</sub>	1.39	Na <sub>2</sub> SO <sub>4</sub>	12	142.06	0.99	8.61 <sup>\$</sup>
NO <sub>2</sub>	58110	1.263	-	-	NaNO <sub>2</sub>	16	69.00	1.00	87.60
NO <sub>3</sub>	183067	2.953	-	-	NaNO <sub>3</sub>	17	84.99	0.99	161.61
CO <sub>3</sub>	44775	0.746	-	-	Na <sub>2</sub> CO <sub>3</sub>	18	105.99	1.00	79.08
Org. Carbon	2718	0.227	-	-	-	-	-	-	-
Acetate	3025	0.051	-	-	Sodium Acetate (C2)	13	136.08	0.99	7.04
Formate	2213	0.049	-	-	Sodium Formate (C1)	14	68.01	0.99	3.38
Oxalate	3321	0.038	-	-	Sodium Oxalate (C2)	15	134.00	0.99	5.11
-	-	-	SUM	100.0	Total simulant Weight				1393.4

- Empty data field.

Assay refers to the purity of the raw material as specified by the vendor.

<sup>\$</sup>SO<sub>3</sub> content was varied during each melter run segment. The value given here corresponds to the nominal.



**Table 2.12. Target and Analyzed Compositions (wt%) of Fifteen ORPLG Crucible Glasses.**

Glass	ORPLG13		ORPLG14		ORPLG15		ORPLG16		ORPLG17	
Oxides	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*
Al <sub>2</sub> O <sub>3</sub>	6.54	6.14	6.53	6.18	6.51	6.06	5.52	5.09	5.52	5.05
B <sub>2</sub> O <sub>3</sub>	8.28	8.13	8.20	8.22	8.12	8.03	8.26	8.72	8.26	8.77
CaO	2.62	2.87	2.60	2.70	2.57	2.81	2.73	2.96	2.73	2.95
Cr <sub>2</sub> O <sub>3</sub>	0.57	0.77	0.57	0.72	0.57	0.77	0.59	0.79	0.59	0.79
Fe <sub>2</sub> O <sub>3</sub>	0.28	0.33	0.27	0.31	0.27	0.33	0.29	0.34	0.29	0.34
K <sub>2</sub> O	5.47	5.48	5.61	5.44	5.75	5.61	5.64	5.60	5.64	5.76
MgO	4.04	4.14	4.00	4.20	3.96	4.14	2.05	2.17	2.05	2.12
Na <sub>2</sub> O	20.00	19.50	20.50	20.83	21.00	20.95	20.61	20.77	20.61	20.68
NiO	0.01	0.00	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.00
PbO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
SiO <sub>2</sub>	39.76	39.97	39.38	39.68	39.00	39.05	41.38	40.42	41.38	40.64
SnO <sub>2</sub>	2.76	2.67	2.73	2.57	2.71	2.64	2.87	3.02	3.23	3.33
TiO <sub>2</sub>	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.01
ZnO	3.31	3.43	3.28	3.17	3.25	3.29	3.45	3.63	2.73	2.86
ZrO <sub>2</sub>	5.52	5.36	5.47	4.95	5.41	5.15	5.74	5.85	6.10	6.11
Cl	0.22	0.21	0.23	0.21	0.23	0.21	0.23	0.23	0.23	0.23
F	0.09	NA	0.09	NA	0.09	NA	0.09	NA	0.09	NA
P <sub>2</sub> O <sub>5</sub>	0.14	0.17	0.14	0.17	0.14	0.17	0.14	0.16	0.14	0.16
SO <sub>3</sub>	0.39	0.37	0.40	0.37	0.41	0.39	0.40	0.36	0.40	0.35
Sum	100.0	99.6	100.0	99.8	100.0	99.7	100.0	100.2	100.0	100.2

\* – Analyzed by X-ray fluorescence except for boron which was measured by DCP  
NA – Not analyzed (Sum includes target fluorine value)

**Table 2.12. Target and Analyzed Compositions (wt%) of Fifteen ORPLG Crucible Glasses (continued).**

Glass	ORPLG18		ORPLG19		ORPLG20		ORPLG21		ORPLG22	
Oxides	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*
Al <sub>2</sub> O <sub>3</sub>	5.52	5.07	5.51	5.08	6.04	5.60	6.04	5.58	6.04	5.52
B <sub>2</sub> O <sub>3</sub>	8.04	8.49	7.96	8.35	8.00	7.58	8.00	7.42	8.00	7.75
CaO	2.73	2.98	2.70	2.89	2.71	2.94	2.71	2.93	2.71	2.89
Cr <sub>2</sub> O <sub>3</sub>	0.59	0.80	0.59	0.78	0.59	0.80	0.59	0.80	0.59	0.77
Fe <sub>2</sub> O <sub>3</sub>	0.29	0.35	0.28	0.33	0.29	0.33	0.29	0.35	0.29	0.33
K <sub>2</sub> O	5.64	5.75	5.78	5.71	5.61	5.83	5.61	5.84	5.61	5.83
MgO	1.97	2.00	1.95	2.07	0.96	1.07	0.96	1.12	0.46	0.49
Na <sub>2</sub> O	20.61	20.22	21.12	21.52	20.50	21.35	20.50	20.49	20.50	21.12
NiO	0.01	0.01	0.01	0.01	0.01	0.03	0.01	0.01	0.01	0.00
PbO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02
SiO <sub>2</sub>	42.24	41.71	41.83	41.19	42.01	41.04	43.01	42.50	42.01	41.06
SnO <sub>2</sub>	2.73	2.94	2.70	2.76	3.71	3.36	2.71	2.66	3.46	3.46
TiO <sub>2</sub>	0.00	0.01	0.00	0.01	0.00	0.02	0.00	0.02	0.00	0.02
ZnO	2.73	2.87	2.70	2.78	2.71	2.78	2.71	2.81	2.71	2.78
ZrO <sub>2</sub>	6.03	6.16	5.97	5.87	6.00	5.73	6.00	5.85	6.75	6.59
Cl	0.23	0.24	0.23	0.20	0.23	0.23	0.23	0.23	0.23	0.25
F	0.09	NA	0.09	NA	0.09	NA	0.09	NA	0.09	NA
P <sub>2</sub> O <sub>5</sub>	0.14	0.15	0.14	0.17	0.14	0.17	0.14	0.16	0.14	0.16
SO <sub>3</sub>	0.40	0.36	0.41	0.36	0.40	0.37	0.40	0.35	0.40	0.34
Sum	100.0	100.2	100.0	100.2	100.0	99.3	100.0	99.2	100.0	99.5

\* – Analyzed by X-ray fluorescence except for boron which was measured by DCP

NA – Not analyzed (Sum includes target fluorine value)

**Table 2.12. Target and Analyzed Compositions (wt%) of Fifteen ORPLG Crucible Glasses (continued).**

Glass	ORPLG23		ORPLG24		ORPLG25		ORPLG26		ORPLG27	
Oxides	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*	Target	Analyzed*
Al <sub>2</sub> O <sub>3</sub>	6.04	5.64	6.03	5.54	6.03	5.50	6.03	5.55	6.03	5.55
B <sub>2</sub> O <sub>3</sub>	8.00	7.69	7.92	7.49	7.92	7.42	7.92	7.70	7.92	7.59
CaO	2.71	2.89	2.69	2.94	2.69	2.94	2.69	2.76	2.69	2.84
Cr <sub>2</sub> O <sub>3</sub>	0.59	0.79	0.59	0.81	0.59	0.80	0.59	0.76	0.59	0.77
Fe <sub>2</sub> O <sub>3</sub>	0.29	0.34	0.28	0.35	0.28	0.34	0.28	0.32	0.28	0.33
K <sub>2</sub> O	5.61	5.86	5.75	6.04	5.75	6.04	5.75	5.79	5.75	5.84
MgO	0.46	0.48	0.94	0.97	0.94	0.97	0.44	0.51	0.44	0.47
Na <sub>2</sub> O	20.50	20.21	21.00	20.72	21.00	20.57	21.00	22.43	21.00	21.49
NiO	0.01	0.02	0.01	0.01	0.01	0.02	0.01	0.02	0.01	0.01
PbO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.00
SiO <sub>2</sub>	42.51	42.25	41.60	41.11	42.60	42.07	41.60	40.69	42.10	41.30
SnO <sub>2</sub>	3.21	3.20	3.69	3.71	2.69	2.79	3.44	3.18	3.19	3.30
TiO <sub>2</sub>	0.00	0.02	0.00	0.01	0.00	0.01	0.00	0.02	0.00	0.01
ZnO	2.71	2.75	2.69	2.80	2.69	2.83	2.69	2.66	2.69	2.74
ZrO <sub>2</sub>	6.50	6.47	5.94	5.96	5.94	6.13	6.69	6.31	6.44	6.34
Cl	0.23	0.23	0.23	0.23	0.23	0.23	0.23	0.21	0.23	0.22
F	0.09	NA	0.09	NA	0.09	NA	0.09	NA	0.09	NA
P <sub>2</sub> O <sub>5</sub>	0.14	0.16	0.14	0.16	0.14	0.16	0.14	0.16	0.14	0.16
SO <sub>3</sub>	0.40	0.34	0.41	0.36	0.41	0.35	0.41	0.34	0.41	0.35
Sum	100.0	99.4	100.0	99.3	100.0	99.3	100.0	99.5	100.0	99.4

\* – Analyzed by X-ray fluorescence except for boron which was measured by DCP

NA – Not analyzed (Sum includes target fluorine value)

**Table 2.13. Descriptions of Fifteen As-Melted and Heat Treated ORPLG Crucible Glasses.**

<b>Glass ID</b>	<b>As-melted glass</b>	<b>Heat Treatment - Glass remelted at 1200°C for 1 hour, heat treated for 20 hours at 950°C, and quenched.</b>
ORPLG13	Opaque green glass	Opaque green glass ~0.4 vol% large NaZr silicate with Sn and a few Cr-rich crystals
ORPLG14	Opaque green glass	Opaque green glass ~0.4 vol% large NaZr silicate with Sn and a few Cr-rich crystals
ORPLG15	Opaque green glass	Opaque green glass ~0.4 vol% large NaZr silicate with Sn and a few Cr-rich crystals
ORPLG16	Clear glass	Mostly clear glass ~0.2 vol% small Cr oxide crystals
ORPLG17	Clear glass	Mostly clear glass ~0.6 vol% small Cr-rich crystals with Zn and few large NaZr silicate crystals with Sn
ORPLG18	Clear glass	Mostly clear glass ~0.3 vol% small Cr-rich crystals with Zn and few large NaZr silicate crystals with Sn
ORPLG19	Clear glass	Mostly clear glass ~0.7 vol% small Cr-rich crystals with Zn and few large NaZr silicate crystals with Sn
ORPLG20	Clear glass	Mostly clear glass ~0.3 vol% small Cr-rich crystals with Zn and few large NaZr silicate crystals with Sn
ORPLG21	Clear glass	Mostly clear glass ~0.4 vol% small Cr-rich crystals with Zn, few large NaZr silicate crystals with Sn and Cr-rich crystal cluster
ORPLG22	Clear glass	Mostly clear glass ~0.4 vol% small Cr-rich crystals with Zn, few large NaZr silicate crystals with Sn and Cr-rich crystal cluster
ORPLG23	Clear glass	Mostly clear glass ~0.3 vol% small Cr-rich crystals with Zn few large NaZr silicate crystals with Sn
ORPLG24	Clear glass	Mostly clear glass ~0.3 vol% small Cr-rich crystals with Zn few large NaZr silicate crystals with Sn
ORPLG25	Clear glass	Mostly clear glass ~0.3 vol% small Cr-rich crystals with Zn few large NaZr silicate crystals with Sn
ORPLG26	Clear glass	Foamy region observed ~0.3 vol% small Cr-rich crystals with Zn few large NaZr silicate crystals with Sn
ORPLG27	Clear glass	Mostly clear glass ~0.5 vol% several NaZr silicate crystals with Sn

**Table 2.14. Measured Sulfate Solubility in Fifteen ORPLG Crucible Glasses.**

Sample ID	SO <sub>3</sub> Content (wt%)		
	Batch Saturation		Bubbling*
	As-Melted	After Acid Wash	
ORPLG13S4	0.49	0.42	-
ORPLG14S4	0.47	0.42	-
ORPLG15S4	0.51	0.44	-
ORPLG16S4	0.51	0.45	-
ORPLG17S4	0.52	0.48	-
ORPLG18S4	0.53	0.48	-
ORPLG19S4	0.52	0.48	-
ORPLG20S4	0.51	0.49	-
ORPLG21S4	0.49	0.46	-
ORPLG22S4	0.50	0.49	-
ORPLG23S4	0.49	0.47	-
ORPLG24S4	0.54	0.52	-
ORPLG25S4	0.54	0.52	-
ORPLG26S4	0.51	0.46	-
ORPLG27S4	0.54	0.49	0.58 (sulfate layer onset at 0.34 wt% SO <sub>3</sub> )

- Empty data field

\* Starting glass for bubbling tests contained no SO<sub>3</sub>.

**Table 2.15. Results of 7-day PCT (at 90°C) and VHT (at 200°C for 24 Days) for Fifteen ORPLG Crucible Glasses.**

Glass ID	ORPLG13	ORPLG14	ORPLG15	ORPLG16	ORPLG17	ORPLG18	ORPLG19
<b>7-Day PCT, Stainless Steel Vessel; S/V=2000 m<sup>-1</sup> (ppm)</b>							
B	249.82	262.46	272.99	139.27	117.10	123.09	172.23
Na	987.40	1,068.17	1,145.83	610.39	507.18	544.07	753.51
Si	234.65	245.66	259.18	200.70	175.13	196.74	251.29
<b>Normalized Concentrations (g/L)</b>							
B	9.72	10.31	10.83	5.43	4.57	4.93	6.96
Na	6.65	7.02	7.35	3.99	3.32	3.56	4.81
Si	1.26	1.33	1.42	1.04	0.91	1.00	1.29
pH	12.41	12.45	12.54	12.12	12.11	12.10	12.32
<b>7-Day PCT Normalized Mass Loss (g/m<sup>2</sup>)</b>							
B	4.86	5.15	5.41	2.72	2.28	2.46	3.48
Na	3.33	3.51	3.68	2.00	1.66	1.78	2.40
Si	0.63	0.67	0.71	0.52	0.45	0.50	0.64
<b>7-Day PCT Normalized Loss Rate (g/d/m<sup>2</sup>)</b>							
B	0.69	0.74	0.77	0.39	0.33	0.35	0.50
Na	0.48	0.50	0.53	0.29	0.24	0.25	0.34
Si	0.09	0.10	0.10	0.07	0.06	0.07	0.09
<b>VHT Alteration (24 days at 200°C)</b>							
Alteration Depth (μm)	1236	1138	1081	Coupon fully reacted	Coupon fully reacted	Coupon fully reacted	Coupon fully reacted
Alteration Rate (g/m <sup>2</sup> /day)*	136	126	119	>>100	>>100	>>100	>>100

\* Alteration rates calculated using estimated density of 2.65 g/cc

**Table 2.15. Results of 7-day PCT (at 90°C) and VHT (at 200°C for 24 Days) for Fifteen ORPLG Crucible Glasses (continued).**

Glass ID	ORPLG20	ORPLG21	ORPLG22	ORPLG23	ORPLG24	ORPLG25	ORPLG26	ORPLG27
<b>7-Day PCT, Stainless Steel Vessel; S/V=2000 m<sup>-1</sup> (ppm)</b>								
B	30.45	34.49	23.33	22.66	33.85	34.24	31.17	32.65
Na	224.00	233.30	218.50	213.80	262.60	248.90	237.80	240.00
Si	83.28	91.20	73.93	74.54	86.20	97.03	80.73	84.69
<b>Normalized Concentrations (g/L)</b>								
B	1.23	1.39	0.94	0.91	1.38	1.39	1.27	1.33
Na	1.47	1.53	1.44	1.41	1.69	1.60	1.53	1.54
Si	0.42	0.45	0.38	0.38	0.44	0.49	0.42	0.43
pH	11.90	11.91	11.91	11.90	11.98	11.92	11.94	11.95
<b>7-Day PCT Normalized Mass Loss (g/m<sup>2</sup>)</b>								
B	0.61	0.69	0.47	0.46	0.69	0.70	0.63	0.66
Na	0.74	0.77	0.72	0.70	0.84	0.80	0.76	0.77
Si	0.21	0.23	0.19	0.19	0.22	0.24	0.21	0.22
<b>7-Day PCT Normalized Loss Rate (g/d/m<sup>2</sup>)</b>								
B	0.09	0.10	0.07	0.07	0.10	0.10	0.09	0.09
Na	0.11	0.11	0.10	0.10	0.12	0.11	0.11	0.11
Si	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
<b>VHT Alteration (24 days at 200°C)</b>								
Alteration Depth (μm)	31	415	32	52	648	722	32	34
Alteration Rate (g/m <sup>2</sup> /day)*	3	46	4	6	72	80	4	4

\* Alteration rates calculated using estimated density of 2.65 g/cc

**Table 2.16. Viscosities and Electrical Conductivities of Seven ORPLG Crucible Glasses.**

Glass ID	ORPLG21	ORPLG22	ORPLG23	ORPLG24	ORPLG25	ORPLG26	ORPLG27
<b>Viscosity (poise)</b>							
900°C	2748	3226	3202	2429	2598	2924	2364
950°C	1062	1198	1193	914	993	1070	908
1000°C	465	509	510	394	433	451	397
1050°C	225	241	244	189	210	213	192
1100°C	119	125	128	100	111	110	102
1150°C	67	69	72	56	63	62	58
1200°C	40	41	44	34	38	37	35
1250°C	25	26	28	22	24	23	22
<b>Electrical Conductivity (S/cm)</b>							
900°C	0.195	0.181	0.189	0.119	0.198	0.174	0.197
950°C	0.242	0.230	0.238	0.198	0.248	0.253	0.250
1000°C	0.296	0.285	0.294	0.284	0.305	0.334	0.310
1050°C	0.356	0.348	0.358	0.371	0.368	0.416	0.377
1100°C	0.423	0.420	0.430	0.457	0.440	0.495	0.451
1150°C	0.496	0.499	0.510	0.538	0.518	0.571	0.531
1200°C	0.575	0.586	0.598	0.616	0.604	0.643	0.617
1250°C	0.661	0.681	0.693	0.688	0.696	0.711	0.709



**Table 2.17. Results of K-3 Corrosion Testing for Three ORPLG Crucible Glasses.**

<b>Glass ID</b>	<b>Neck loss (inches)</b>	<b>Depth of altered zone (inches)</b>	<b>Half-down loss (inches)</b>
ORPLG24	0.0320	0.0230	Coupon expanded – no measurable loss
ORPLG26	0.0285	0.0255	Coupon expanded – no measurable loss
ORPLG27	0.0340	0.0290	Coupon expanded – no measurable loss

**Table 2.18. Summary of Test Results for Selected ORPLG Glass Formulation ORPLG27 and Comparison to ILAW Requirements.**

Test	Requirement [52, 53]	Test Result for ORPLG27
Density of glass	< 3.7 g/cc	2.67 to 2.71 g/cc*
Crystalline Phase	Phase identification	~0.5 vol% of sodium zirconium silicate
Liquidus	< 950°C	T <sub>1%</sub> below 950°C
Centerline Canister Cooling	Phase identification	Not measured
PCT B (g/m <sup>2</sup> )	< 2.0 g/m <sup>2</sup>	0.66 g/m <sup>2</sup>
PCT Na (g/m <sup>2</sup> )	< 2.0 g/m <sup>2</sup>	0.77 g/m <sup>2</sup>
PCT Si (g/m <sup>2</sup> )	< 2.0 g/m <sup>2</sup>	0.22 g/m <sup>2</sup>
VHT at 200°C (g/m <sup>2</sup> /day)	< 50 g/m <sup>2</sup> /day	4 g/m <sup>2</sup> /day
Viscosity (poise) at 1100°C	10 to 150 P	102 P
Conductivity (S/cm) at 1100°C	0.2 to 0.7 S/cm	0.451 S/cm
T <sub>G</sub> (°C)	Report for modeling	Not measured

\*Density measured for melter glass I10-G-135A (2.67) and crucible glass ORPLG27 (2.71).

**Table 2.19. Oxide Composition of AP-101 Simulant and ORPLG27 Glass Composition Used in Melter Tests (wt%).**

Component	AP-101 waste contribution	Glass former additives	ORPLG27 (for AP-101)
Loading	29.10%	70.90%	-
Al <sub>2</sub> O <sub>3</sub>	1.54	4.48	6.02
B <sub>2</sub> O <sub>3</sub>	0.00	7.91	7.91
CaO	-	2.68	2.68
Cr <sub>2</sub> O <sub>3</sub>	0.09	0.50	0.59
Fe <sub>2</sub> O <sub>3</sub>	-	0.28	0.28
K <sub>2</sub> O	5.74	-	5.74
MgO	-	0.44	0.44
Na <sub>2</sub> O <sup>(a)</sup>	20.21 + 0.39 <sup>(1)</sup> + 0.40 <sup>(2)</sup>	-	21.00
NiO	0.01	-	0.01
PbO	0.01	-	0.01
SiO <sub>2</sub>	0.03	42.02	42.05
SnO <sub>2</sub>	-	3.18	3.18
ZnO	-	2.68	2.68
ZrO <sub>2</sub>	-	6.43	6.43
Cl	0.22	-	0.23
F	0.09	-	0.09
P <sub>2</sub> O <sub>5</sub>	0.14	-	0.14
SO <sub>3</sub> <sup>(b)</sup>	0.50 <sup>(1)</sup>	-	0.50
SUM	29.4	70.6	100.0

(a) Simulant was ordered at a concentration of 20.21 wt% Na<sub>2</sub>O and modified before each melter test with (1) Na<sub>2</sub>SO<sub>4</sub> and (2) NaOH additions to obtain 21.0 wt% Na<sub>2</sub>O in the glass.

(b) Concentration of SO<sub>3</sub> was varied in steps during the melter tests.

– Empty data field

**Table 2.20a. Glass Former Additives for 1 Liter of AP-101 Simulant (8 M Na) and Corresponding Melter Feed Properties.**

Additives Source	Feed ORPLG27
Additives in Glass (wt%)	70.9%
Kyanite ( $\text{Al}_2\text{SiO}_5$ ) 325 Mesh (Kyanite Mining) (g)	97.66
$\text{H}_3\text{BO}_3$ (US Borax – Technical Granular) (g)	166.01
Wollastonite NYAD 325 Mesh (NYCO Minerals) (g)	69.87
$\text{Cr}_2\text{O}_3$ oxide – Alfa Aesar	5.95
$\text{Fe}_2\text{O}_3$ oxide – Alfa Aesar	1.87
Olivine ( $\text{Mg}_2\text{SiO}_4$ ) 325 Mesh (#180 Unimin) (g)	9.73
$\text{SiO}_2$ (Sil-co-Sil 75 US Silica) (g)	375.39
$\text{SnO}_2$ - Stannous Oxide - Mason Color	37.81
ZnO (KADOX – 920 Zinc Corp. of America) (g)	31.76
Zircon $\text{ZrSiO}_4$ (Flour) Mesh 325 (AM. Mineral) (g)	114.67
$\text{Na}_2\text{SO}_4$	Variable – Table 2.20b
Addition of Sucrose as Reductant (g)	83.66
Simulant Weight for 1 liter (g)	1393
Sum of Additives (g)	911
Sum of Complete Batch (g)	2388
Target Final Volume (l)	1.33
Estimated Density (g/ml)	1.74
Target Glass Produced (g)	1181
Target Weight % Additives in Slurry	39
Target Glass Yield (g/kg of Feed)	510
Target Glass Yield (g/l of Feed)	888
Target Total Solids (g/l of Feed)	1126
Target Additives (g/l of Feed)	685

**Table 2.20b. NaOH and  $\text{Na}_2\text{SO}_4$  Additions Required to Obtain 21.0 wt%  $\text{Na}_2\text{O}$  and Various  $\text{SO}_3$  Concentrations in the ORPLG27 Glass.**

Final $\text{SO}_3$ wt%	NaOH needed per kg of feed (grams)	$\text{Na}_2\text{SO}_4$ needed per kg of feed (grams)
0.0	10.53	-
0.1	9.50	0.93
0.2	8.47	1.86
0.3	7.43	2.79
0.4	6.40	3.71
0.5	5.37	4.64
0.6	4.34	5.57

**Table 2.21. Characteristics of Melter Feed Samples During DM10 ORP LAW Tests.**

	Formulation	Date	Sample Name	% Water	pH	Density (g/ml)	Glass Yield			
							(g/l)	Measured	Target	%Dev.
								(kg/kg)	(kg/kg)	
AP-101/ Sub-Envelope A2	Stock Feed	5/6/2010	I10-F-82A	36.34	12.85	1.73	878	0.508	0.500	1.60
	Consolidated feed from Tests A1 - A4	5/11/2010	I10-F-131A	34.67	13.03	1.72	865	0.503	0.500	0.60
	ORPLG9 [8]			36.45	12.82	1.74	862	0.497	0.509	-2.42
AN-105/ Sub-Envelope A1	Stock Feed	5/11/2010	I10-F-130A	39.22	11.30	1.64	782	0.477	0.470	1.49
	Consolidated feed from Tests B1 - B4	5/14/2010	J10-F-24A	37.79	12.91	1.65	773	0.468	0.470	-0.43
	ORPLA15 [6]			42.08	13.59	1.64	725	0.442	0.465	-5.0

**Table 2.22. Target and XRF Analyzed Composition of Vitrified Melter Feed Samples (wt%).**

Test	A					
Sample Info	ORPLG27-S0			Combined A1, A2, A3 and A4		
Constituent	Target	I10-F-82A	%Dev.	Target	I10-F-131A	%Dev.
Al <sub>2</sub> O <sub>3</sub>	6.06	5.71	-5.87	6.02	5.91	-1.84
B <sub>2</sub> O <sub>3</sub> *	8.04	8.04	NC	7.91	7.91	NC
CaO	2.73	3.04	11.46	2.68	2.83	5.32
Cl	0.23	0.16	NC	0.23	0.15	NC
Cr <sub>2</sub> O <sub>3</sub>	0.59	0.72	NC	0.59	0.59	NC
F	0.09	NA	NC	0.09	NA	NC
Fe <sub>2</sub> O <sub>3</sub>	0.28	0.41	NC	0.28	0.40	NC
I	§	0.11	NC	§	0.12	NC
K <sub>2</sub> O	5.61	6.37	13.45	5.74	5.70	-0.64
MgO	0.45	0.40	NC	0.44	0.63	NC
MnO	§	0.01	NC	§	0.01	NC
Na <sub>2</sub> O	20.52	20.82	1.50	21.00	20.42	-2.74
NiO	0.01	0.01	NC	0.01	0.01	NC
P <sub>2</sub> O <sub>5</sub>	0.14	0.21	NC	0.14	0.20	NC
PbO	0.01	0.01	NC	0.01	<0.01	NC
SiO <sub>2</sub>	42.74	40.76	-4.63	42.05	42.36	0.74
SnO <sub>2</sub>	3.24	3.63	12.23	3.18	3.49	9.58
SO <sub>3</sub>	§	<0.01	NC	0.50	0.30	NC
TiO <sub>2</sub>	§	0.14	NC	§	0.13	NC
V <sub>2</sub> O <sub>5</sub>	§	<0.01	NC	§	<0.01	NC
ZnO	2.73	2.92	7.06	2.68	2.54	-5.51
ZrO <sub>2</sub>	6.54	6.53	-0.15	6.43	6.29	-2.14
Sum	100.00	100.00	NC	100.00	100.00	NC

\* - Target values

§ - Not a target constituent

NA – Not analyzed

NC – Not calculated

**Table 2.22. Target and XRF Analyzed Composition of Vitrified Melter Feed Samples (wt%)  
(continued).**

Test	B					
Sample Info	ORPLA38-1-S0			Combined B1, B2, B3 and B4		
Constituent	Target	I10-F-130A	%Dev.	Target	J10-F-24A	%Dev.
Al <sub>2</sub> O <sub>3</sub>	7.05	6.46	-8.44	6.95	6.30	-9.27
B <sub>2</sub> O <sub>3</sub> *	8.34	8.34	NC	8.22	8.22	NC
CaO	3.18	3.45	8.73	3.13	3.36	7.21
Cl	0.67	0.61	NC	0.66	0.52	NC
Cr <sub>2</sub> O <sub>3</sub>	0.50	0.74	NC	0.49	0.67	NC
Fe <sub>2</sub> O <sub>3</sub>	0.26	0.38	NC	0.26	0.39	NC
I	§	0.10	NC	§	0.11	NC
K <sub>2</sub> O	0.53	0.60	NC	0.52	0.56	NC
MgO	0.99	0.99	-0.84	0.98	1.21	NC
MnO	§	0.02	NC	§	0.01	NC
Na <sub>2</sub> O	23.72	22.94	-3.29	24.00	22.70	-5.41
NiO	§	0.01	NC	§	<0.01	NC
P <sub>2</sub> O <sub>5</sub>	§	0.01	NC	§	<0.01	NC
PbO	§	<0.01	NC	§	<0.01	NC
SiO <sub>2</sub>	42.15	41.21	-2.24	41.55	41.61	0.15
SnO <sub>2</sub>	2.71	3.27	20.83	2.67	3.34	24.88
SO <sub>3</sub>	§	<0.01	NC	0.80	0.52	NC
TiO <sub>2</sub>	§	0.05	NC	§	0.05	NC
V <sub>2</sub> O <sub>5</sub>	0.93	1.15	NC	0.92	1.05	NC
ZnO	2.86	3.20	12.16	2.82	2.99	6.08
ZrO <sub>2</sub>	6.12	6.48	5.86	6.03	6.39	6.05
Sum	100.00	100.00	NC	100.00	100.00	NC

\* - Target values

§ - Not a target constituent

NA - Not analyzed

NC - Not calculated

**Table 2.23. DCP and XRF Analyzed Compositions of Vitrified Melter Feed Samples (wt%).**

Test	A					
Sample Info	ORPLG27-S0			Combined A1, A2, A3 and A4		
	I10-F-82A			I10-F-131A		
Constituent	Target	XRF	DCP	Target	XRF	DCP
Al <sub>2</sub> O <sub>3</sub>	6.06	5.71	6.11	6.02	5.91	6.18
B <sub>2</sub> O <sub>3</sub>	8.04	8.04*	7.78	7.91	7.91*	7.33
CaO	2.73	3.04	2.71	2.68	2.83	2.63
Cl	0.23	0.16	NA	0.23	0.15	NA
Cr <sub>2</sub> O <sub>3</sub>	0.59	0.72	0.56	0.59	0.59	0.49
F	0.09	NA	NA	0.09	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	0.28	0.41	0.41	0.28	0.40	0.43
I	§	0.11	NA	§	0.12	NA
K <sub>2</sub> O	5.61	6.37	5.76	5.74	5.70	5.36
Li <sub>2</sub> O	§	NA	0.03	§	NA	0.03
MgO	0.45	0.40	0.49	0.44	0.63	0.73
MnO	§	0.01	0.01	§	0.01	0.02
Na <sub>2</sub> O	20.52	20.82	18.44	21.00	20.42	19.11
NiO	0.01	0.01	0.03	0.01	0.01	0.03
P <sub>2</sub> O <sub>5</sub>	0.14	0.21	0.25	0.14	0.20	0.12
PbO	0.01	0.01	0.03	0.01	<0.01	0.03
SiO <sub>2</sub>	42.74	40.76	41.48	42.05	42.36	42.78
SnO <sub>2</sub>	3.24	3.63	3.64	3.18	3.49	3.30
SO <sub>3</sub>	§	<0.01	NA	0.50	0.30	NA
TiO <sub>2</sub>	§	0.14	0.14	§	0.13	0.15
V <sub>2</sub> O <sub>5</sub>	§	<0.01	0.02	§	<0.01	0.02
ZnO	2.73	2.92	2.70	2.68	2.54	2.48
ZrO <sub>2</sub>	6.54	6.53	6.52	6.43	6.29	6.64
Sum	100.00	100.00	97.11	100.00	100.00	97.86

\* - Target values

§ - Not a target constituent

NA – Not analyzed



**Table 2.23. DCP and XRF Analyzed Compositions of Vitrified Melter Feed Samples (wt%)  
(continued).**

Test	B					
Sample Info	ORPLA38-1-S0			Combined B1, B2, B3 and B4		
	I10-F-130A			J10-F-24A		
Constituent	Target	XRF	DCP	Target	XRF	DCP
Al <sub>2</sub> O <sub>3</sub>	7.05	6.46	6.84	6.95	6.30	6.77
B <sub>2</sub> O <sub>3</sub>	8.34	8.34*	8.12	8.22	8.22*	7.94
CaO	3.18	3.45	2.98	3.13	3.36	3.04
Cl	0.67	0.61	NA	0.66	0.52	NA
Cr <sub>2</sub> O <sub>3</sub>	0.50	0.74	0.55	0.49	0.67	0.52
Fe <sub>2</sub> O <sub>3</sub>	0.26	0.38	0.37	0.26	0.39	0.38
I	§	0.10	NA	§	0.11	NA
K <sub>2</sub> O	0.53	0.60	0.67	0.52	0.56	0.62
Li <sub>2</sub> O	§	NA	0.03	§	NA	0.03
MgO	0.99	0.99	1.25	0.98	1.21	1.37
MnO	§	0.02	0.02	§	0.01	0.02
Na <sub>2</sub> O	23.72	22.94	20.92	24.00	22.70	21.20
NiO	§	0.01	0.03	§	<0.01	0.03
P <sub>2</sub> O <sub>5</sub>	§	0.01	<0.01	§	<0.01	<0.01
PbO	§	<0.01	0.02	§	<0.01	0.02
SiO <sub>2</sub>	42.15	41.21	41.66	41.55	41.61	42.49
SnO <sub>2</sub>	2.71	3.27	3.10	2.67	3.34	2.90
SO <sub>3</sub>	§	<0.01	NA	0.80	0.52	NA
TiO <sub>2</sub>	§	0.05	0.06	§	0.05	0.06
V <sub>2</sub> O <sub>5</sub>	0.93	1.15	1.03	0.92	1.05	1.00
ZnO	2.86	3.20	2.83	2.82	2.99	2.73
ZrO <sub>2</sub>	6.12	6.48	6.05	6.03	6.39	6.11
Sum	100.00	100.00	96.53	100.00	100.00	97.23

\* - Target values

§ - Not a target constituent

NA – Not analyzed

**Table 3.1. Summary of Test A (Simulant: LAW Sub-Envelope A2 (AP-101), Glass Formulation: ORPLG27) Conditions and Results.**

Test Segment		A1	A2	A3	A4
Time	Feed Start	5/6/10 17:00	5/7/10 9:40	5/10/10 10:15	5/11/10 2:00
	Feed End	5/7/10 8:15	5/7/10 23:12	5/10/10 23:10	5/11/10 15:30
	Net Slurry Feeding (hr)	15.25	13.5	12.9	13.5
Feed	wt% SO <sub>3</sub> as glass	0.2	0.4	0.6	0.5
	Feed Used (kg)	55.2	55.5	54.2	54.0
Average Production Rate (kg/m <sup>2</sup> /day)*		2068	2349	2401	2286
Average Bubbling Rate (lpm)		2.4	3.6	3.0	3.6
Average Temperatures (°C)	Glass, 2" from floor	1152	1152	1152	1152
	Glass, 4" from floor	1150	1151	1148	1148
	Electrode	1085	1078	1081	1085
	Plenum, thermowell	489	476	550	491
	Plenum, exposed	469	415	536	472
Product	Secondary Phases on Melt Surface at Test End	No	No	Yes	No
	Measured wt% SO <sub>3</sub>	0.20	0.33	0.51	0.47
	% Feed Sulfur in Glass Product	100	83	85	94
Average Concentrations monitored in stack exhaust by FTIR (ppmv)	N <sub>2</sub> O	79.1	75.0	74.0	75.7
	NO	546	532	523	519
	NO <sub>2</sub>	88.7	84.2	94.1	92.8
	CO	13.8	12.0	12.9	12.3
	NH <sub>3</sub>	30.0	32.2	26.1	25.8

\* – Glass production rates calculated from feed data

**Table 3.2. Summary of Test B (Simulant: LAW Sub-Envelope A1 (AN-105), Glass Formulation: ORPLA38-1) Conditions and Results.**

Test Segment		B1	B2	B3	B4
Time	Feed Start	5/11/10 17:40	5/12/10 8:15	5/12/10 23:15	5/13/10 15:25
	Feed End	5/12/10 6:15	5/12/10 21:30	5/13/10 12:30	5/14/10 4:45
	Net Slurry Feeding (hr)	12.6	13.25	13.25	13.3
Feed	wt% SO <sub>3</sub> as glass	0.5	0.7	0.9	0.8
	Feed Used (kg)	53.9	58.1	56.2	57.2
Average Production Rate (kg/m <sup>2</sup> /day)*		2301	2355	2278	2310
Average Bubbling Rate (lpm)		4.1	3.5	3.9	4.0
Average Temperatures (°C)	Glass, 2" from floor	1152	1147	1144	1147
	Glass, 4" from floor	1149	1143	1142	1145
	Electrode	1057	1029	1088	1088
	Plenum, thermowell	468	481	470	494
	Plenum, exposed	458	460	450	491
Product	Secondary Phases on Melt Surface at Test End	No	No	Yes	No
	Measured wt% SO <sub>3</sub>	0.42	0.60	0.77	0.76
	% Feed Sulfur in Glass Product	84	86	86	95
Average Concentrations monitored in stack exhaust by FTIR (ppmv)	N <sub>2</sub> O	70.0	71.7	70.1	70.1
	NO	561	590	577	578
	NO <sub>2</sub>	98.3	99.8	87.4	83.0
	CO	17.6	20.3	18.5	19.3
	NH <sub>3</sub>	31.4	28.0	28.8	31.8

\* – Glass production rates calculated from feed data

NM – Not Measured.

**Table 4.1. Listing of DM10 Glasses Discharged, Masses, Target Sulfur Contents and Analysis Performed.**

Test	Formulations	Target SO <sub>3</sub> (wt%)	Date	Name	Analyses	Mass (kg)	Cumulative (kg)	Observations of Sulfate	
A1	ORPLG27	0.2	5/6/10	I10-G-94A	-	-	-	-	
				I10-G-94B	XRF	3.40	3.40	No	
				I10-G-94C	-	-	-	-	
				I10-G-94D	XRF	2.64	6.04	No	
				I10-G-94E	-	-	-	-	
				I10-G-95A	XRF	3.96	10.00	No	
				I10-G-95B	-	-	-	-	
			5/7/10	I10-G-95C	XRF	2.80	12.80	No	
				I10-G-96A	-	-	-	-	
				I10-G-96B	XRF	4.94	17.74	No	
				I10-G-96C	-	-	-	-	
				I10-G-96D	XRF	3.44	21.18	No	
				I10-G-96E	-	-	-	-	
				I10-G-99A	XRF	3.46	24.64	No	
A2		0.4	5/7/10	I10-G-01A	-	-	-	-	
				I10-G-101B	XRF	3.02	27.66	No	
				I10-G-03A	-	-	-	-	
				I10-G-103B	XRF	4.00	31.66	Minor sulfate on exterior of drips	
				I10-G-103C	-	-	-	-	
				I10-G-04A	XRF	3.56	35.22	Minor sulfate on exterior of drips	
				I10-G-104B	-	-	-	-	
				I10-G-104C	XRF	3.64	38.86	Minor sulfate on exterior of drips	
				I10-G-04D	-	-	-	-	
				I10-G-05A	XRF	3.94	42.80	Minor sulfate on exterior of drips	
				I10-G-105B	-	-	-	-	
				I10-G-105C	XRF	3.58	46.38	Minor sulfate on exterior of drips	
				I10-G-05D	-	-	-	-	
A3		0.6	5/10/10	I10-G-108A	XRF	4.24	50.62	No	
				I10-G-108B	XRF	2.30	52.92	No	
				5/8/10	I10-G-109A	XRF	3.06	55.98	No
				5/10/10	I10-G-119A	-	-	-	-
					I10-G-119B	XRF	3.16	59.14	No
					I10-G-119C	-	-	-	-
					I10-G-119D	XRF	3.30	62.44	No
					I10-G-121A	-	-	-	-
					I10-G-121B	XRF	2.98	65.42	No
					I10-G-121C	-	-	-	-
I10-G-121D		XRF	3.56	68.98	No				
I10-G-122A	-	-	-	-					

**Table 4.1. Listing of DM10 Glasses Discharged, Masses, Target Sulfur Contents and Analysis Performed (continued).**

Test	Formulation	Target SO <sub>3</sub> (wt%)	Date	Name	Analysis	Mass (kg)	Cumulative (kg)	Observations of Sulfate			
A3	ORPLG27	0.6	5/10/10	I10-G-122B	XRF	3.88	72.86	No			
				I10-G-122C	-	-	-	-			
				I10-G-122D	XRF	4.10	76.96	No			
				I10-G-123A	-	-	-	-			
				I10-G-123B	XRF	3.32	80.28	No			
A4		0.5	5/11/10	I10-G-126A	-	-	-	-			
				I10-G-129A	XRF	3.38	83.66	No			
				I10-G-129B	-	-	-	-			
				I10-G-129C	XRF	4.48	88.14	No			
				I10-G-129D	-	-	-	-			
				I10-G-129E	XRF	4.28	92.42	No			
				I10-G-130A	-	-	-	-			
				I10-G-130B	XRF	3.76	96.18	No			
				I10-G-130C	-	-	-	-			
				I10-G-130D	XR	5.72	101.90	No			
				I10-G-131A	-	-	-	-			
				I10-G-135A	XRF	3.26	105.16	No			
				B1	0.5	5/11/10	I10-G-135B	-	-	-	-
							I10-G-140A	XRF	3.52	108.68	No
							I10-G-140B	-	-	-	-
I10-G-140C		XRF	3.90				112.58	No			
I10-G-140D		-	-				-	-			
I10-G-143A		XRF	3.94				116.52	No			
B2		0.7	5/12/10				I10-G-143B	-	-	-	-
							I10-G-143C	XRF	4.18	120.70	No
	I10-G-143D						-	-	-	-	
	I10-G-146A						XRF	3.82	124.52	No	
	I10-G-146B			-	-	-	-				
I10-G-146C	XRF			4.52	129.04	No					
I10-G-147A	-			-	-	-					
I10-G-147B	XRF			4.30	133.34	No					
I10-G-149A	-			-	-	-					
I10-G-149B	XRF			3.80	137.14	No					
I10-G-149C	-			-	-	-					
I10-G-149D	XRF			3.42	140.56	No					
I10-G-151A	-			-	-	-					
I10-G-151B	XRF			4.00	144.56	No					
I10-G-151C	-			-	-	-					
				J10-G-7A	XRF	3.92	148.48	No			
				J10-G-7B	-	-	-	-			
				J10-G-7C	XRF	3.74	152.22	No			
				J10-G-9A	XRF	1.18	153.40	No			

**Table 4.1. Listing of DM10 Glasses Discharged, Masses, Target Sulfur Contents and Analysis Performed (continued).**

Test	Formulations	Target SO <sub>3</sub> (wt%)	Date	Name	Analyses	Mass (kg)	Cumulative (kg)	Observations of Sulfate
B3	ORPLA38-1	0.9	5/12/10	J10-G-11A	-	-	-	-
				J10-G-11B	XRF	4.20	157.60	No
				J10-G-11C	-	-	-	-
				J10-G-11D	XRF	6.16	163.76	No
				J10-G-14A	-	-	-	-
				J10-G-14B	XRF	4.44	168.20	No
				J10-G-14C	-	-	-	-
				J10-G-14D	XRF	4.38	172.58	No
				J10-G-14E	-	-	-	-
				J10-G-14F	XRF	4.00	176.58	No
J10-G-15A		XRF	1.82	178.40	No			
B4		0.8	5/13/10	J10-G-17A	-	-	-	-
				J10-G-19A	XRF	3.54	181.94	No
				J10-G-19B	-	-	-	-
				J10-G-19C	XRF	3.62	185.56	No
				J10-G-19D	-	-	-	-
				J10-G-19E	XRF	3.56	189.12	No
				J10-G-22A	-	-	-	-
				J10-G-22B	XRF	3.82	192.94	No
			J10-G-22C	-	-	-	-	
	5/14/10		J10-G-23A	XRF	4.36	197.30	No	
J10-G-23B		-	-	-	-			
J10-G-23C		XRF	3.80	201.10	No			
J10-G-24A		-	-	-	-			
J10-G-24B	XRF	2.68	203.78	No				

**Table 4.2. XRF Analyzed Compositions for DM10 Discharged Glass Samples (wt%).**

Formulation	ORPLG27									
Test	A1									
Target SO <sub>3</sub>	0.2%									
Glass (kg)		3.40	6.00	10.00	12.80	17.70	21.20	24.60	27.70	
Constituent	Target	I10-G-94B	I10-G-94D	I10-G-95A	I10-G-95C	I10-G-96B	I10-G-96D	I10-G-99A	I10-G-101B	%Dev
Al <sub>2</sub> O <sub>3</sub>	6.05	5.63	5.62	5.52	5.67	5.64	5.66	5.69	5.69	-5.91
B <sub>2</sub> O <sub>3</sub> *	7.94	9.11	8.84	8.54	8.40	8.22	8.14	8.08	8.04	NC
CaO	2.69	3.66	3.53	3.38	3.23	3.26	3.13	3.11	3.09	14.69
Cl	0.23	0.12	0.14	0.15	0.16	0.16	0.17	0.16	0.17	NC
Cr <sub>2</sub> O <sub>3</sub>	0.59	0.43	0.56	0.62	0.68	0.75	0.76	0.85	0.82	NC
Cs <sub>2</sub> O	§	0.03	0.01	0.02	<0.01	<0.01	<0.01	<0.01	<0.01	NC
F	0.09	NA	NA	NA	NA	NA	NA	NA	NA	NC
Fe <sub>2</sub> O <sub>3</sub>	0.28	4.56	3.51	2.96	2.21	2.00	1.52	1.04	1.19	NC
I	§	0.03	0.06	0.07	0.08	0.09	0.09	0.11	0.11	NC
K <sub>2</sub> O	5.76	1.87	3.15	3.69	4.33	4.64	5.12	5.74	5.57	-3.34
Li <sub>2</sub> O*	§	0.70	0.54	0.36	0.28	0.17	0.12	0.08	0.06	NC
MgO	0.45	1.73	1.19	1.14	0.94	0.83	0.70	0.66	0.64	NC
MnO	§	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.01	NC
Na <sub>2</sub> O	21.00	19.46	19.61	20.61	20.86	20.76	20.96	20.65	20.76	-1.16
NiO	0.01	0.06	0.05	0.04	0.04	0.04	0.04	0.04	0.04	NC
P <sub>2</sub> O <sub>5</sub>	0.14	0.18	0.17	0.17	0.19	0.20	0.19	0.19	0.19	NC
PbO	0.01	0.01	<0.01	0.01	0.01	0.01	0.01	0.01	0.01	NC
SiO <sub>2</sub>	42.21	42.49	41.77	41.22	41.33	40.93	41.20	40.73	40.74	-3.48
SnO <sub>2</sub>	3.19	0.94	1.79	2.12	2.44	2.69	2.96	3.20	3.28	2.64
SO <sub>3</sub>	0.20	0.33	0.28	0.27	0.25	0.22	0.22	0.20	0.21	NC
TiO <sub>2</sub>	§	0.80	0.62	0.53	0.41	0.38	0.30	0.23	0.23	NC
V <sub>2</sub> O <sub>5</sub>	§	0.09	0.07	0.06	0.04	0.03	0.03	0.02	0.02	NC
ZnO	2.69	2.83	2.91	2.85	2.76	2.86	2.77	2.86	2.83	5.04
ZrO <sub>2</sub>	6.46	4.92	5.55	5.65	5.67	6.08	5.90	6.35	6.30	-2.43
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	NC

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed boron and lithium concentrations in the first discharged glass sample

§ - Not a target constituent

NA – not analyzed by XRF

"-" Empty data field

**Table 4.2. XRF Analyzed Compositions for DM10 Discharged Glass Samples (wt%) (continued).**

Formulation	ORPLG27									
Test	A2									
Target SO <sub>3</sub>	0.4%									
Glass (kg)		31.70	35.20	38.90	42.80	46.40	50.60	52.90	56.00	
Constituent	Target	I10-G-103B	I10-G-104A	I10-G-104C	I10-G-105A	I10-G-105C	I10-G-108A	I10-G-108B	I10-G-109A	%Dev
Al <sub>2</sub> O <sub>3</sub>	6.03	5.68	5.70	5.64	5.79	5.70	5.67	5.58	5.79	-3.97
B <sub>2</sub> O <sub>3</sub> *	7.92	8.00	7.98	7.96	7.95	7.94	7.93	7.93	7.93	NC
CaO	2.69	2.95	3.00	3.01	2.87	2.96	3.05	3.02	3.10	15.24
Cl	0.23	0.23	0.16	0.15	0.15	0.15	0.16	0.15	0.09	NC
Cr <sub>2</sub> O <sub>3</sub>	0.59	0.81	0.83	0.85	0.82	0.94	0.90	0.89	0.79	NC
F	0.09	NA	NA	NA	NA	NA	NA	NA	NA	NC
Fe <sub>2</sub> O <sub>3</sub>	0.28	0.88	0.85	0.72	0.61	0.65	0.60	0.58	1.02	NC
I	§	0.10	0.10	0.12	0.10	0.11	0.12	0.13	0.10	NC
K <sub>2</sub> O	5.75	5.64	5.77	5.85	5.77	5.99	6.27	6.19	5.82	1.30
Li <sub>2</sub> O*	§	0.04	0.03	0.02	0.01	0.01	0.01	<0.01	<0.01	NC
MgO	0.44	0.54	0.59	0.51	0.45	0.49	0.43	0.52	0.63	NC
MnO	§	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.02	NC
Na <sub>2</sub> O	21.00	21.70	21.56	21.41	22.42	21.53	19.91	20.57	20.18	-3.91
NiO	0.01	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.07	NC
P <sub>2</sub> O <sub>5</sub>	0.14	0.19	0.19	0.20	0.18	0.19	0.20	0.19	0.19	NC
PbO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	NC
SiO <sub>2</sub>	42.10	40.65	40.37	40.21	40.27	40.13	40.55	40.07	40.73	-3.26
SnO <sub>2</sub>	3.19	3.23	3.32	3.63	3.33	3.53	3.88	3.81	3.40	6.59
SO <sub>3</sub>	0.40	0.25	0.25	0.29	0.31	0.31	0.35	0.34	0.33	NC
TiO <sub>2</sub>	§	0.20	0.19	0.17	0.15	0.16	0.16	0.16	0.22	NC
V <sub>2</sub> O <sub>5</sub>	§	0.02	0.02	0.01	0.01	0.00	0.01	0.01	0.02	NC
ZnO	2.69	2.74	2.82	2.82	2.72	2.82	2.93	2.93	2.89	7.41
ZrO <sub>2</sub>	6.44	6.07	6.21	6.36	6.01	6.34	6.82	6.88	6.67	3.63
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	NC

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed boron and lithium concentrations in the first discharged glass sample

§ - Not a target constituent

NA – not analyzed by XRF

NC – not calculated.



**Table 4.2. XRF Analyzed Compositions for DM10 Discharged Glass Samples (wt%) (continued).**

Formulation	ORPLG27										
Test	A3									A4	
Target SO <sub>3</sub>	0.6%									0.5%	
Glass (kg)	Target	59.10	62.40	65.40	69.00	72.90	77.00	80.30		Target	83.70
Constituent		I10-G-119B	I10-G-119D	I10-G-121B	I10-G-121D	I10-G-122B	I10-G-122D	I10-G-123B	%Dev		I10-G-129A
Al <sub>2</sub> O <sub>3</sub>	6.02	5.83	5.76	5.80	5.76	5.73	5.78	5.85	-2.83	6.02	5.83
B <sub>2</sub> O <sub>3</sub> *	7.90	7.90	7.90	7.90	7.90	7.90	7.90	7.90	NC	7.91	7.90
CaO	2.68	2.99	2.99	2.96	2.88	2.97	2.90	2.80	4.39	2.68	2.88
Cl	0.23	0.11	0.11	0.13	0.14	0.15	0.15	0.15	NC	0.23	0.14
Cr <sub>2</sub> O <sub>3</sub>	0.59	0.80	0.84	0.85	0.84	0.88	0.84	0.84	NC	0.59	0.84
F	0.09	NA	NA	NA	NA	NA	NA	NA	NC	0.09	NA
Fe <sub>2</sub> O <sub>3</sub>	0.28	0.86	0.81	0.70	0.59	0.57	0.51	0.48	NC	0.28	0.47
I	§	0.12	0.12	0.10	0.09	0.10	0.10	0.11	NC	§	0.10
K <sub>2</sub> O	5.73	5.81	5.80	5.91	5.84	6.09	5.96	5.96	3.91	5.74	5.99
MgO	0.44	0.61	0.62	0.54	0.50	0.44	0.43	0.52	NC	0.44	0.44
MnO	§	0.01	0.01	0.01	0.01	0.01	0.01	0.01	NC	§	0.01
Na <sub>2</sub> O	21.00	20.74	20.96	21.18	22.12	21.32	21.58	21.51	2.42	21.00	21.52
NiO	0.01	0.07	0.07	0.06	0.05	0.05	0.04	0.04	NC	0.01	0.05
P <sub>2</sub> O <sub>5</sub>	0.14	0.20	0.19	0.20	0.19	0.19	0.19	0.20	NC	0.14	0.19
PbO	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.00	NC	0.01	0.01
SiO <sub>2</sub>	42.00	41.02	40.34	40.76	40.44	40.36	40.82	40.98	-2.42	42.05	41.05
SnO <sub>2</sub>	3.18	3.30	3.59	3.39	3.27	3.51	3.42	3.42	7.57	3.18	3.33
SO <sub>3</sub>	0.60	0.40	0.41	0.43	0.47	0.49	0.51	0.51	NC	0.50	0.50
TiO <sub>2</sub>	§	0.20	0.18	0.17	0.15	0.15	0.14	0.13	NC	0.00	0.14
V <sub>2</sub> O <sub>5</sub>	§	0.02	0.02	0.01	0.01	0.01	<0.01	0.01	NC	0.00	<0.01
ZnO	2.68	2.78	2.86	2.80	2.73	2.85	2.71	2.62	-2.35	2.68	2.68
ZrO <sub>2</sub>	6.42	6.23	6.41	6.09	5.99	6.22	5.99	5.97	-7.11	6.43	5.95
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	NC	100.00	100.00

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed boron and lithium concentrations in the first discharged glass sample

§ - Not a target constituent

NA – not analyzed by XRF

NC – not calculated.

**Table 4.2. XRF Analyzed Compositions for DM10 Discharged Glass Samples (wt%) (continued).**

Formulation	ORPLG27							ORPLA38-1			
Test	A4							B1			
Target SO <sub>3</sub>	0.5%							0.5%			
Glass (kg)	Target	88.10	92.40	96.20	101.90	105.20		Target	108.70	112.60	116.50
Constituent		I10-G-129C	I10-G-129E	I10-G-130B	I10-G-130D	I10-G-135A	%Dev		I10-G-140A	I10-G-140C	I10-G-143A
Al <sub>2</sub> O <sub>3</sub>	6.02	5.74	5.82	5.77	5.82	5.89	-2.29	6.97	5.93	6.22	6.31
B <sub>2</sub> O <sub>3</sub> *	7.91	7.91	7.91	7.91	7.91	7.91	NC	8.26	8.01	8.09	8.14
CaO	2.68	2.92	2.87	2.92	2.82	2.81	4.62	3.14	3.05	3.03	3.08
Cl	0.23	0.14	0.14	0.15	0.16	0.15	NC	0.66	0.25	0.32	0.38
Cr <sub>2</sub> O <sub>3</sub>	0.59	0.88	0.87	0.88	0.84	0.85	NC	0.50	0.89	0.83	0.81
F	0.09	NA	NA	NA	NA	NA	NC	0.00	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	0.28	0.47	0.46	0.46	0.41	0.42	NC	0.26	0.42	0.39	0.38
I	§	0.11	0.12	0.10	0.12	0.10	NC	§	0.09	0.09	0.10
K <sub>2</sub> O	5.74	6.09	6.02	6.04	5.95	6.08	5.86	0.53	4.99	3.78	2.93
MgO	0.44	0.45	0.45	0.43	0.42	0.46	NC	0.98	0.47	0.60	0.72
MnO	§	0.01	0.01	0.01	0.01	0.01	NC	§	0.01	0.01	0.01
Na <sub>2</sub> O	21.00	21.29	21.23	21.20	21.68	21.79	3.74	24.00	21.38	22.23	23.09
NiO	0.01	0.04	0.04	0.04	0.04	0.04	NC	§	0.04	0.04	0.04
P <sub>2</sub> O <sub>5</sub>	0.14	0.20	0.19	0.20	0.20	0.20	NC	§	0.16	0.13	0.10
PbO	0.01	0.01	0.01	0.01	0.01	<0.01	NC	§	0.01	0.01	<0.01
SiO <sub>2</sub>	42.05	40.41	40.66	40.77	41.04	40.83	-2.91	41.71	40.83	41.61	40.85
SnO <sub>2</sub>	3.18	3.56	3.65	3.56	3.49	3.32	4.27	2.68	3.44	3.19	3.33
SO <sub>3</sub>	0.50	0.49	0.47	0.44	0.46	0.47	NC	0.50	0.47	0.45	0.45
TiO <sub>2</sub>	§	0.13	0.13	0.13	0.12	0.13	NC	0.00	0.11	0.09	0.07
V <sub>2</sub> O <sub>5</sub>	§	0.01	<0.01	0.01	<0.01	<0.01	NC	0.92	0.26	0.44	0.61
ZnO	2.68	2.79	2.76	2.76	2.62	2.65	-1.36	2.83	2.91	2.76	2.82
ZrO <sub>2</sub>	6.43	6.35	6.20	6.23	5.88	5.92	-8.02	6.05	6.27	5.71	5.79
Sum	100.00	100.00	100.00	100.00	100.00	100.00	NC	100.00	100.00	100.00	100.00

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed boron and lithium concentrations in the first discharged glass sample

§ - Not a target constituent

NA – not analyzed by XRF

NC – not calculated.

**Table 4.2. XRF Analyzed Compositions for DM10 Discharged Glass Samples (wt%) (continued).**

Formulation	ORPLA38-1										
Test	B1					B2					
Target SO <sub>3</sub>	0.5%					0.7%					
Glass (kg)	-	120.70	124.50	129.00		-	133.30	137.10	140.60	144.60	148.50
Constituent	Target	I10-G-143C	I10-G-146A	I10-G-146C	%Dev	Target	I10-G-147B	I10-G-149B	I10-G-149D	I10-G-151B	J10-G-7A
Al <sub>2</sub> O <sub>3</sub>	6.97	6.51	6.39	6.53	-6.38	6.96	6.55	6.58	6.58	6.58	6.52
B <sub>2</sub> O <sub>3</sub> *	8.26	8.18	8.21	8.22	NC	8.23	8.23	8.23	8.23	8.23	8.23
CaO	3.14	3.07	3.22	3.14	0.07	3.13	3.31	3.27	3.18	3.12	3.21
Cl	0.66	0.43	0.44	0.46	NC	0.66	0.42	0.43	0.47	0.45	0.47
Cr <sub>2</sub> O <sub>3</sub>	0.50	0.78	0.82	0.77	NC	0.49	0.84	0.81	0.75	0.76	0.76
F	0.00	NA	NA	NA	NC	0.00	NA	NA	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	0.26	0.36	0.38	0.37	NC	0.26	0.42	0.38	0.36	0.35	0.36
I	§	0.09	0.10	0.10	NC	§	0.10	0.10	0.07	0.10	0.09
K <sub>2</sub> O	0.53	2.19	1.79	1.36	NC	0.52	1.30	1.09	0.85	0.77	0.74
MgO	0.98	0.73	0.80	1.04	5.58	0.98	0.91	0.93	1.03	1.11	1.08
MnO	§	0.01	0.01	0.01	NC	§	0.01	0.01	0.01	0.01	0.01
Na <sub>2</sub> O	24.00	23.61	23.35	24.02	0.09	24.00	23.20	23.66	24.35	25.06	24.56
NiO	§	0.03	0.03	0.03	NC	§	0.04	0.04	0.03	0.03	0.03
P <sub>2</sub> O <sub>5</sub>	§	0.06	0.06	0.05	NC	§	0.05	<0.01	0.02	<0.01	0.02
PbO	§	<0.01	<0.01	<0.01	NC	§	<0.01	<0.01	<0.01	<0.01	<0.01
SiO <sub>2</sub>	41.71	41.63	40.78	40.89	-1.97	41.60	40.89	41.25	41.26	40.58	40.45
SnO <sub>2</sub>	2.68	2.94	3.29	3.08	14.65	2.68	3.15	3.02	2.90	3.03	3.16
SO <sub>3</sub>	0.50	0.47	0.45	0.42	NC	0.70	0.46	0.51	0.54	0.55	0.58
TiO <sub>2</sub>	§	0.06	0.07	0.06	NC	§	0.06	0.05	0.06	0.04	0.05
V <sub>2</sub> O <sub>5</sub>	0.92	0.72	0.84	0.87	NC	0.92	0.97	0.97	0.96	0.95	1.01
ZnO	2.83	2.73	2.96	2.81	-0.74	2.82	3.02	2.93	2.82	2.76	2.89
ZrO <sub>2</sub>	6.05	5.39	6.01	5.76	-4.81	6.04	6.08	5.73	5.54	5.51	5.79
Sum	100.00	100.00	100.00	100.00	NC	100.00	100.00	100.00	100.00	100.00	100.00

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed boron and lithium concentrations in the first discharged glass sample

§ - Not a target constituent

NA – not analyzed by XRF

NC – not calculated.

**Table 4.2. XRF Analyzed Compositions for DM10 Discharged Glass Samples (wt%) (continued).**

Formulation	ORPLA38-1									
Test	B2				B3					
Target SO <sub>3</sub>	0.7%				0.9%					
Glass (kg)	Target	152.20	153.40		Target	157.60	163.80	168.20	172.60	176.60
Constituent		J10-G-7C	J10-G-9A	%Dev		J10-G-11B	J10-G-11D	J10-G-14B	J10-G-14D	J10-G-14F
Al <sub>2</sub> O <sub>3</sub>	6.96	6.49	6.51	-6.41	6.94	6.59	6.50	6.56	6.65	6.57
B <sub>2</sub> O <sub>3</sub> *	8.23	8.23	8.23	NC	8.21	8.23	8.22	8.22	8.22	8.21
CaO	3.13	3.30	3.27	4.48	3.13	3.17	3.27	3.29	3.20	3.18
Cl	0.66	0.50	0.48	NC	0.66	0.46	0.48	0.48	0.50	0.51
Cr <sub>2</sub> O <sub>3</sub>	0.49	0.79	0.75	NC	0.49	0.74	0.79	0.75	0.72	0.74
F	0.00	NA	NA	NC	0.00	NA	NA	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	0.26	0.38	0.40	NC	0.26	0.36	0.39	0.36	0.35	0.34
I	§	0.10	0.09	NC	§	0.09	0.10	0.09	0.10	0.10
K <sub>2</sub> O	0.52	0.72	0.67	NC	0.52	0.66	0.66	0.65	0.61	0.62
MgO	0.98	0.95	1.03	4.85	0.98	1.13	1.02	1.09	1.01	1.15
MnO	§	0.01	0.01	NC	§	0.01	0.01	0.02	0.01	0.02
Na <sub>2</sub> O	24.00	22.97	23.37	-2.61	24.00	24.04	23.95	23.88	24.21	24.48
NiO	§	0.03	0.03	NC	§	0.03	0.03	0.03	0.03	0.03
P <sub>2</sub> O <sub>5</sub>	§	0.02	0.02	NC	§	0.03	0.02	0.02	0.01	0.01
PbO	§	<0.01	<0.01	NC	§	<0.01	<0.01	<0.01	<0.01	<0.01
SiO <sub>2</sub>	41.60	41.35	41.35	-0.62	41.49	41.44	40.58	41.06	41.37	40.90
SnO <sub>2</sub>	2.68	3.25	3.05	13.84	2.67	2.96	3.22	3.02	2.92	2.90
SO <sub>3</sub>	0.70	0.59	0.60	NC	0.90	0.67	0.72	0.72	0.76	0.78
TiO <sub>2</sub>	§	0.05	0.05	NC	§	0.04	0.05	0.05	0.04	0.05
V <sub>2</sub> O <sub>5</sub>	0.92	1.06	1.03	NC	0.92	0.99	1.05	1.04	1.02	1.01
ZnO	2.82	3.01	2.94	4.13	2.81	2.76	2.95	2.95	2.79	2.80
ZrO <sub>2</sub>	6.04	6.19	6.12	1.40	6.02	5.61	5.97	5.75	5.50	5.60
Sum	100.00	100.00	100.00	NC	100.00	100.00	100.00	100.00	100.00	100.00

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed boron and lithium concentrations in the first discharged glass sample

§ - Not a target constituent

NA – not analyzed by XRF

NC – not calculated.

**Table 4.2. XRF Analyzed Compositions for DM10 Discharged Glass Samples (wt%) (continued).**

Formulation	ORPLA38-1									
Test	B3			B4						
Target SO <sub>3</sub>	0.9%			0.8%						
Glass (kg)	178.40									
Constituent	Target	J10-G-15A	%Dev	Target	J10-G-19A	J10-G-19C	J10-G-19E	J10-G-22B	J10-G-23A	J10-G-23C
Al <sub>2</sub> O <sub>3</sub>	6.94	6.53	-5.86	6.95	6.57	6.66	6.59	6.62	6.57	6.58
B <sub>2</sub> O <sub>3</sub> *	8.21	8.21	NC	8.22	8.22	8.22	8.22	8.22	8.22	8.22
CaO	3.13	3.23	3.18	3.13	3.28	3.27	3.23	3.22	3.18	3.17
Cl	0.66	0.50	NC	0.66	0.42	0.48	0.48	0.47	0.46	0.47
Cr <sub>2</sub> O <sub>3</sub>	0.49	0.75	NC	0.49	0.76	0.76	0.77	0.76	0.73	0.74
F	0.00	NA	NC	0.00	NA	NA	NA	NA	NA	NA
Fe <sub>2</sub> O <sub>3</sub>	0.26	0.36	NC	0.26	0.38	0.36	0.35	0.35	0.36	0.36
I	§	0.10	NC	§	0.10	0.09	0.09	0.09	0.09	0.10
K <sub>2</sub> O	0.52	0.61	NC	0.52	0.68	0.68	0.64	0.63	0.61	0.60
MgO	0.98	1.04	6.17	0.98	0.92	0.97	1.06	1.11	1.10	1.07
MnO	§	0.01	NC	§	0.01	0.01	0.01	0.01	0.01	0.01
Na <sub>2</sub> O	24.00	24.15	0.61	24.00	23.82	23.70	23.88	23.94	24.32	24.25
NiO	§	0.03	NC	§	0.03	0.03	0.03	0.03	0.02	0.02
P <sub>2</sub> O <sub>5</sub>	§	0.02	NC	§	0.01	<0.01	<0.01	0.02	0.02	0.02
PbO	§	<0.01	NC	§	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
SiO <sub>2</sub>	41.49	40.73	-1.83	41.55	40.98	41.54	41.28	41.09	40.93	41.13
SnO <sub>2</sub>	2.67	3.13	17.16	2.67	3.11	2.88	3.05	3.03	3.06	3.01
SO <sub>3</sub>	0.90	0.77	NC	0.80	0.72	0.75	0.76	0.72	0.72	0.72
TiO <sub>2</sub>	§	0.04	NC	§	0.05	0.05	0.05	0.04	0.05	0.04
V <sub>2</sub> O <sub>5</sub>	0.92	1.04	NC	0.92	1.03	1.03	1.03	1.04	1.00	0.99
ZnO	2.81	2.89	2.65	2.82	2.94	2.91	2.87	2.91	2.83	2.80
ZrO <sub>2</sub>	6.02	5.87	-2.46	6.03	5.98	5.64	5.62	5.70	5.71	5.69
Sum	100.00	100.00	NC	100.00	100.00	100.00	100.00	100.00	100.00	100.00

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed boron and lithium concentrations in the first discharged glass sample

§ - Not a target constituent

NA – not analyzed by XRF

NC – not calculated.

**Table 4.2. XRF Analyzed Compositions for DM10 Discharged Glass Samples (wt%) (continued).**

Formulation	ORPLA38-1		
Test	B4		
Target SO <sub>3</sub>	0.8%		
Glass (kg)	203.80		
Constituent	Target	J10-G-24B	%Dev
Al <sub>2</sub> O <sub>3</sub>	6.95	6.55	-5.72
B <sub>2</sub> O <sub>3</sub> *	8.22	8.22	NC
CaO	3.13	3.17	1.42
Cl	0.66	0.46	NC
Cr <sub>2</sub> O <sub>3</sub>	0.49	0.75	NC
F	0.00	NA	NC
Fe <sub>2</sub> O <sub>3</sub>	0.26	0.36	NC
I	§	0.08	NC
K <sub>2</sub> O	0.52	0.59	NC
MgO	0.98	1.19	NC
MnO	§	0.01	NC
Na <sub>2</sub> O	24.00	24.71	2.96
NiO	§	0.02	NC
P <sub>2</sub> O <sub>5</sub>	§	0.02	NC
PbO	§	<0.01	NC
SiO <sub>2</sub>	41.55	40.60	-2.27
SnO <sub>2</sub>	2.67	2.84	6.14
SO <sub>3</sub>	0.80	0.76	NC
TiO <sub>2</sub>	§	0.05	NC
V <sub>2</sub> O <sub>5</sub>	0.92	1.01	NC
ZnO	2.82	2.81	-0.35
ZrO <sub>2</sub>	6.03	5.78	-4.10
Sum	100.00	100.00	NC

\* - Target values calculated based on simple well-stirred tank model using DCP-AES analyzed boron and lithium concentrations in the first discharged glass sample

§ - Not a target constituent

NA – not analyzed by XRF

NC – not calculated.

**Table 4.3. Listing of Dip Samples and Presence of Sulfate Layer During DM10 Melter Tests.**

Test	Target SO <sub>3</sub> (wt%)	Date	Time	Sample Name	Sample Location	Secondary Phase Observed
Before A1	0.2	5/6/10	16:47	I10-D-93A	West	No
				I10-D-93B	East	No
				I10-D-93C	North	No
After A1	0.2	5/7/10	8:45	I10-D-101A	North	Foamy, no
				I10-D-101B	East	Foamy, no
				I10-D-101C	West	No
After A2	0.4	5/7/10	23:30	I10-D-108A	North	Foamy, no
				I10-D-108B	East	Foamy, yes
				I10-D-108C	West	Foamy, no
		5/8/10	0:05	I10-D-109A	East	Less foamy, no
Before A3		5/10/10	8:35	I10-D-109B	North	Yes
				I10-D-109C	East	No
				I10-D-109D	West	No
	0.6	5/10/10	9:32	I10-D-109E	North	No
				I10-D-123A	North	Yes
				I10-D-123B	East	Yes
After A3	0.6	5/11/10	23:40	I10-D-123C	West	Yes
				I10-D-126A	North	Yes
				I10-D-126B	East	Yes
	0.5	5/11/10	0:10	I10-D-126C	West	No
				I10-D-126D	North	No
				I10-D-126E	East	No
After A4	0.5	5/11/10	16:03	I10-D-131A	North	No
				I10-D-131B	East	All foam, no
				I10-D-131C	West	No
			16:24	I10-D-135A	East	Less foamy, no
After B1	0.5	5/12/10	6:45	I10-D-147A	North	No
				I10-D-147B	East	No
				I10-D-147C	West	No

**Table 4.3. Listing of Dip Samples and Presence of Sulfate Layer During DM10 Melter Tests  
(continued).**

Test	Target SO <sub>3</sub> (wt%)	Date	Time	Sample Name	Sample Location	Secondary Phase Observed
After B2	0.7	5/12/10	22:00	J10-D-9A	North	Foamy yellow, no
			22:17	J10-D-9B	East	Foamy yellow, no
				J10-D-9C	West	Foamy yellow, no
				J10-D-9D	North	No
				J10-D-9E	East	No
				J10-D-9F	West	No
After B3	0.9	5/13/10	12:50	J10-D-15A	West	Yes
				J10-D-15B	North	Yes
				J10-D-15C	East	Yes
				J10-D-15D	North	Small amount
			13:35	J10-D-15E	East	Small amount
				J10-D-15F	West	No
				J10-D-17A	North	Small amount
			13:58	J10-D-17B	East	No
				J10-D-17C	North	No
After B4	0.8	5/14/10	5:15	J10-D-24A	North	No
				J10-D-24B	East	No
				J10-D-24C	West	No



**Table 4.4. Results of PCT Leaching Procedure (ASTM C1285, 7-days at 90°C, Stainless Steel Vessel; S/V=2000 m<sup>-1</sup>) for Crucible Glass and Corresponding Melter Glass Samples from DM10 ORP LAW Tests.**

Region		A		G		ANL-LRM-2	WTP Contract Limit
Tank Waste/Sub-Envelope Identification		AN-105/Sub-Envelope A1		AP-101/Sub-Envelope A2			
Sample Type		Crucible Glass	Melter Glass	Crucible Glass	Melter Glass		
Sample I.D.		ORPLA38-1	J10-G-24B	ORPLG27	I10-G-135A		
7-Day PCT Concentration in mg/L	B	37.11	44.78	32.65	45.57	35.96	
	Na	240.74	302.30	240.00	329.10	176.30	
	Si	88.53	100.90	84.69	101.20	89.07	
7-Day PCT Normalized Concentrations, g/L	B	1.46	1.75	1.33	1.86	1.45	
	Na	1.35	1.65	1.54	2.04	1.19	
	Si	0.46	0.53	0.43	0.53	0.35	
	pH	11.78	11.77	11.95	11.93	11.10	
7-Day PCT Normalized Mass Loss (g/m <sup>2</sup> )	B	0.73	0.88	0.66	0.93	0.72	< 2.0
	Na	0.68	0.82	0.77	1.02	0.59	< 2.0
	Si	0.23	0.27	0.22	0.27	0.18	< 2.0
7-Day PCT Normalized Loss Rate, g/d/m <sup>2</sup>	B	0.10	0.13	0.09	0.13	0.10	
	Na	0.10	0.12	0.11	0.12	0.09	
	Si	0.03	0.04	0.03	0.04	0.03	

**Table 4.5. VHT Results (24 Day) for Crucible Glass and Corresponding Melter Glass Samples that Contain the Maximum Sulfur Concentration Without Formation of Secondary Phases During DM10 ORP LAW Tests.**

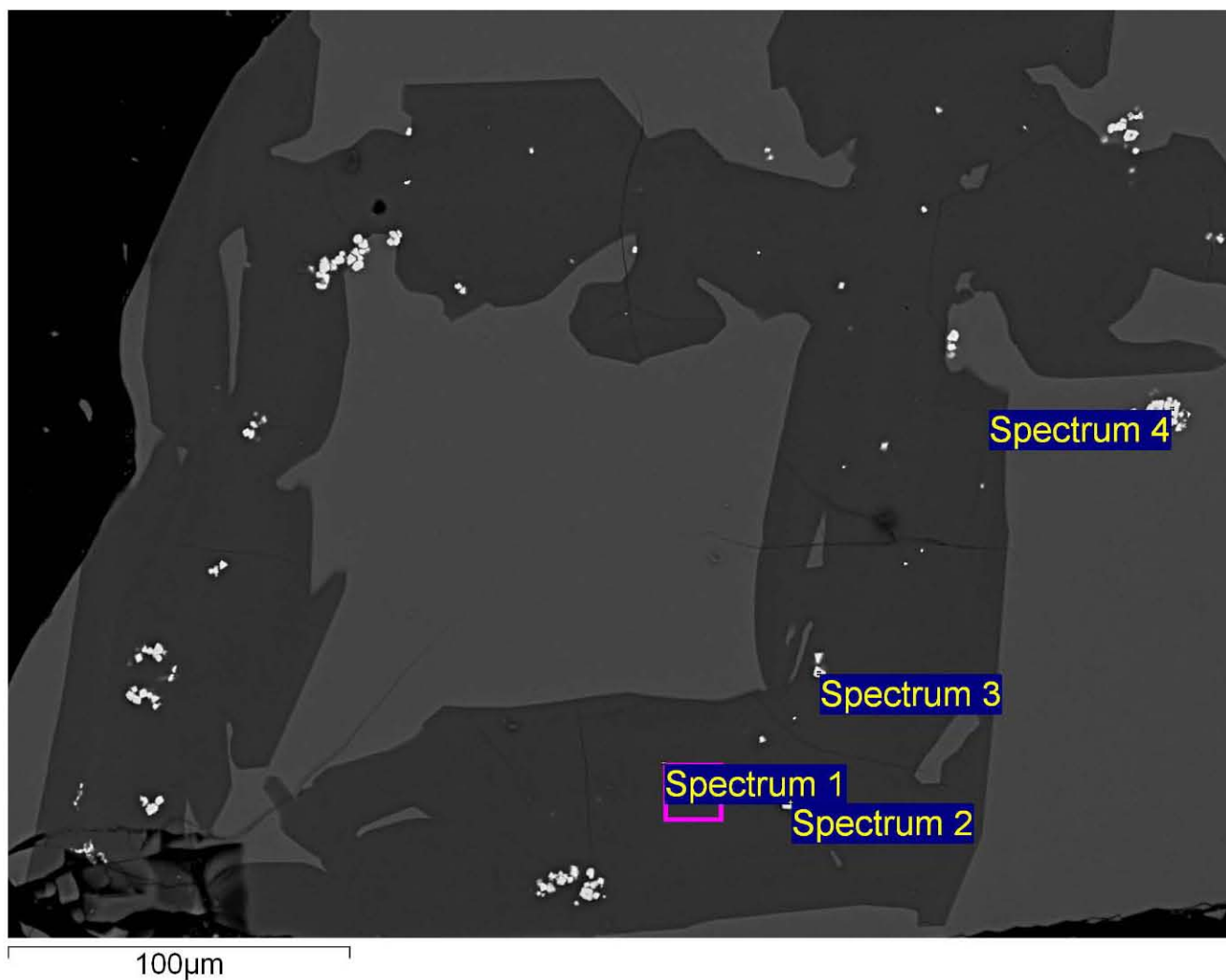
Region		A		G	
Tank Waste/Sub-Envelope Identification		AN-105/ Sub-Envelope A1		AP-101/ Sub-Envelope A2	
Sample Type		Crucible Glass	Melter Glass	Crucible Glass	Melter Glass
Sample I.D.		ORPLA38-1	J10-G-24BC	ORPLG27	I10-G-135A
Based on Layer Thickness	Alteration depth ( $\mu\text{m}$ )	71	102	120	700
	Rate ( $\text{g}/\text{m}^2/\text{d}$ )	8	11	13	77
	Compared to limit of $50 \text{ g}/\text{m}^2/\text{d}$	16%	23%	27%	155%
Based on Remaining Glass	Alteration depth ( $\mu\text{m}$ )	Not measured because alteration depth was below $100 \mu\text{m}$	Not measurable due to cracks	34	658
	Rate ( $\text{g}/\text{m}^2/\text{d}$ )			4	73
	Compared to limit of $50 \text{ g}/\text{m}^2/\text{d}$			8%	145%

Rates calculated with an average density of  $2.65 \text{ g}/\text{cm}^3$

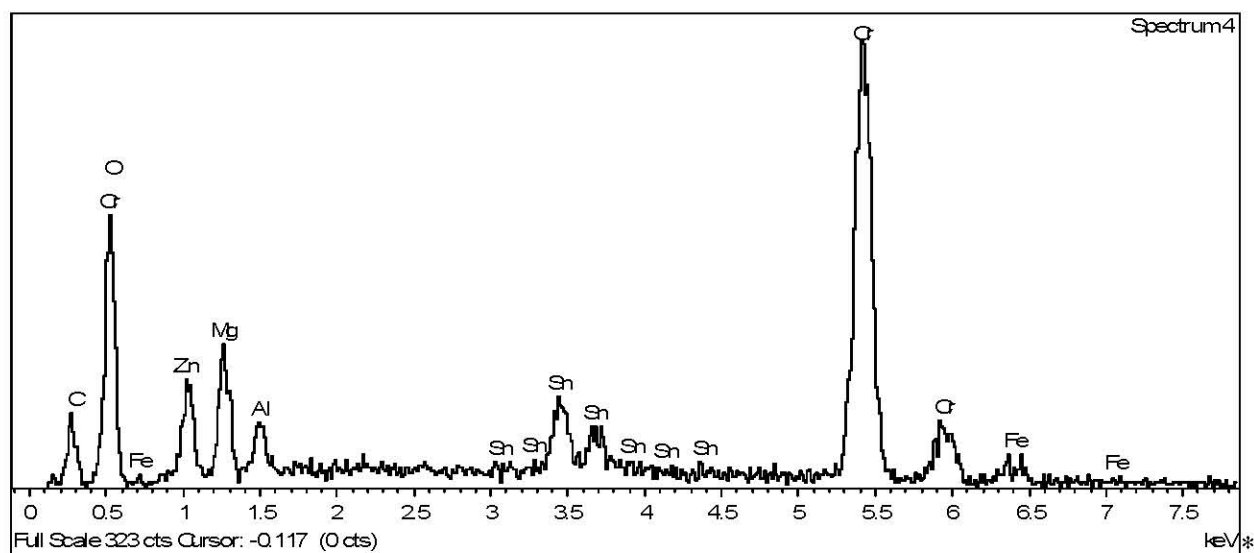
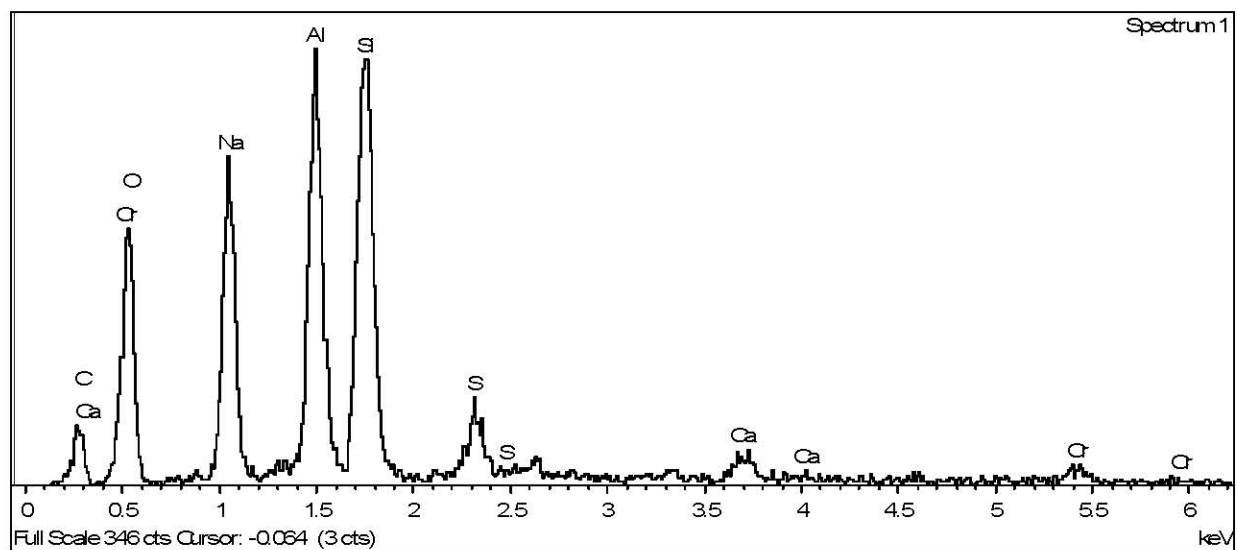
**Table 5.1. Waste Loading Improvements in LAW Glass Formulations Developed for WTP and ORP.**

Waste ID	Loading	Bechtel Baseline		Bechtel Correlation (2007)		ORP (2006)		ORP (2007)		ORP (2008)		ORP (2010)		ORP Summary	
AN-105	Waste	Glass LAWA44	26.0%	Glass LAWA44H	27.2%	Glass LAWA187	30.5%	Glass ORPLA15	31.6%	Glass ORPLA20	31.6%	Glass ORPLA38-1	31.5%	Glass ORPLA38-1	31.5%
	Na <sub>2</sub> O		20.0%		21.3%		22.9%		24.0%		24.0%		24.0%		24.0%
	K <sub>2</sub> O		0.5%		0.5%		0.5%		0.5%		0.5%		0.5%		0.5%
	SO <sub>3</sub>		0.2%		0.4%		1.0%		0.6%		0.7%		0.8%		0.8%
AP-101	Waste	Glass LAWA126	24.5%	Glass LAWA126H	25.4%	-	-	-	-	Glass ORPLG9	29.1%	Glass ORPLG27	29.1%	Glass ORPLG27	29.1%
	Na <sub>2</sub> O		18.5%		18.2%						21.0%		21.0%		21.0%
	K <sub>2</sub> O		3.8%		5.0%						5.8%		5.8%		5.8%
	SO <sub>3</sub>		0.4%		0.4%						0.2%		0.5%		0.5%
AN-107	Waste	Glass LAWC22	16.2%	Glass LAWC22H	20.6%	-	-	Glass ORPLB4	26.2%	-	-	-	-	Glass ORPLB4	26.2%
	Na <sub>2</sub> O		14.4%		19.0%				24.0%						24.0%
	K <sub>2</sub> O		0.1%		0.5%				0.1%						0.1%
	SO <sub>3</sub>		0.4%		0.5%				0.9%						0.9%
AN-104	Waste	Glass LAWA137	20.0%	Glass LAWA137H	20.9%	-	-	Glass ORPLC5	31.0%	-	-	-	-	Glass ORPLC5	31.0%
	Na <sub>2</sub> O		14.6%		15.8%				23.6%						23.6%
	K <sub>2</sub> O		0.3%		0.5%				0.5%						0.5%
	SO <sub>3</sub>		0.4%		0.5%				0.7%						0.7%
AN-102	Waste	Glass LAWC35	14.0%	Glass LAWC35H	16.2%	Glass LAWC100	24.1%	Glass ORPLD1	24.9%	Glass ORPLD6	25.9%	-	-	Glass ORPLD6	25.9%
	Na <sub>2</sub> O		12.0%		13.5%		20.0%		21.0%		22.0%				22.0%
	K <sub>2</sub> O		0.1%		0.5%		0.2%		0.2%		0.2%				0.2%
	SO <sub>3</sub>		0.6%		0.6%		1.1%		1.1%		1.2%				1.2%
AZ-101	Waste	Glass LAWB83	7.3%	Glass LAWB83H	11.3%	-	-	Glass ORPLE12	19.8%	-	-	-	-	Glass ORPLE12	19.8%
	Na <sub>2</sub> O		5.5%		8.9%				16.0%						16.0%
	K <sub>2</sub> O		0.2%		0.5%				0.6%						0.6%
	SO <sub>3</sub>		0.7%		0.7%				1.5%						1.5%
AZ-102	Waste	Glass LAWB96	3.7%	Glass LAWB96H	6.2%	Glass LAWB99	11.9%	-	-	Glass ORPLF7	14.3%	-	-	Glass ORPLF7	14.3%
	Na <sub>2</sub> O		5.5%		5.7%		10.0%				12.0%				12.0%
	K <sub>2</sub> O		0.1%		0.5%		0.4%				0.5%				0.5%
	SO <sub>3</sub>		0.7%		0.8%		1.5%				1.5%				1.5%

- Empty data field



**Figure 2.1. SEM image of ORPLA28 heat treated for 20 hours at 950°C.**



**Figure 2.2. EDS spectrum from site 1 (top) and site 4 (bottom) of Figure 2.1.**  
The large crystals are sulfate-rich aluminosilicate and the small crystals are chromium rich spinels with Mg, Zn and Sn (sites 2 & 3 also are spinel crystals).



**Figure 2.3. Optical image of as-melted ORPLA28 glass showing sulfate phase.**

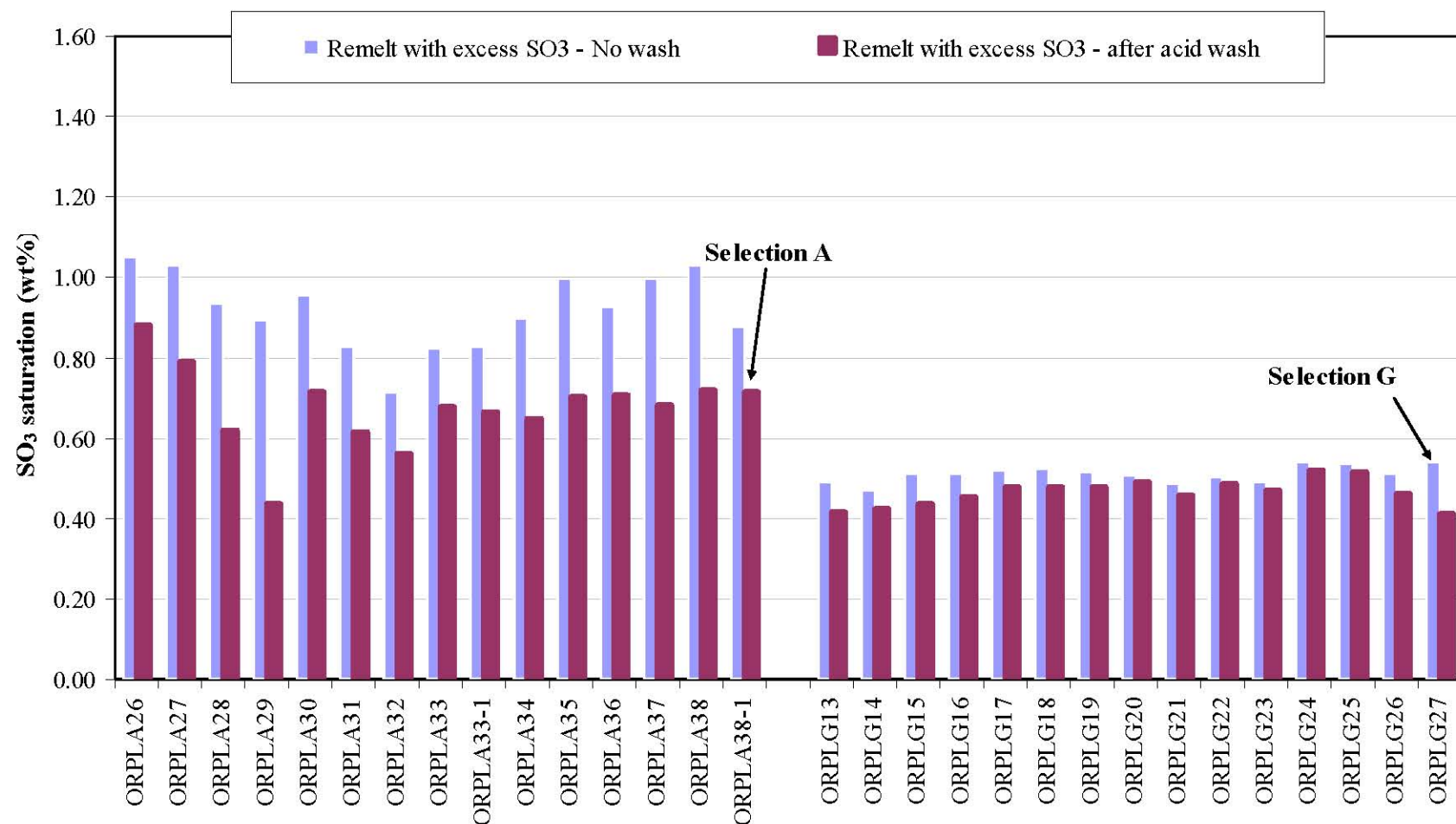
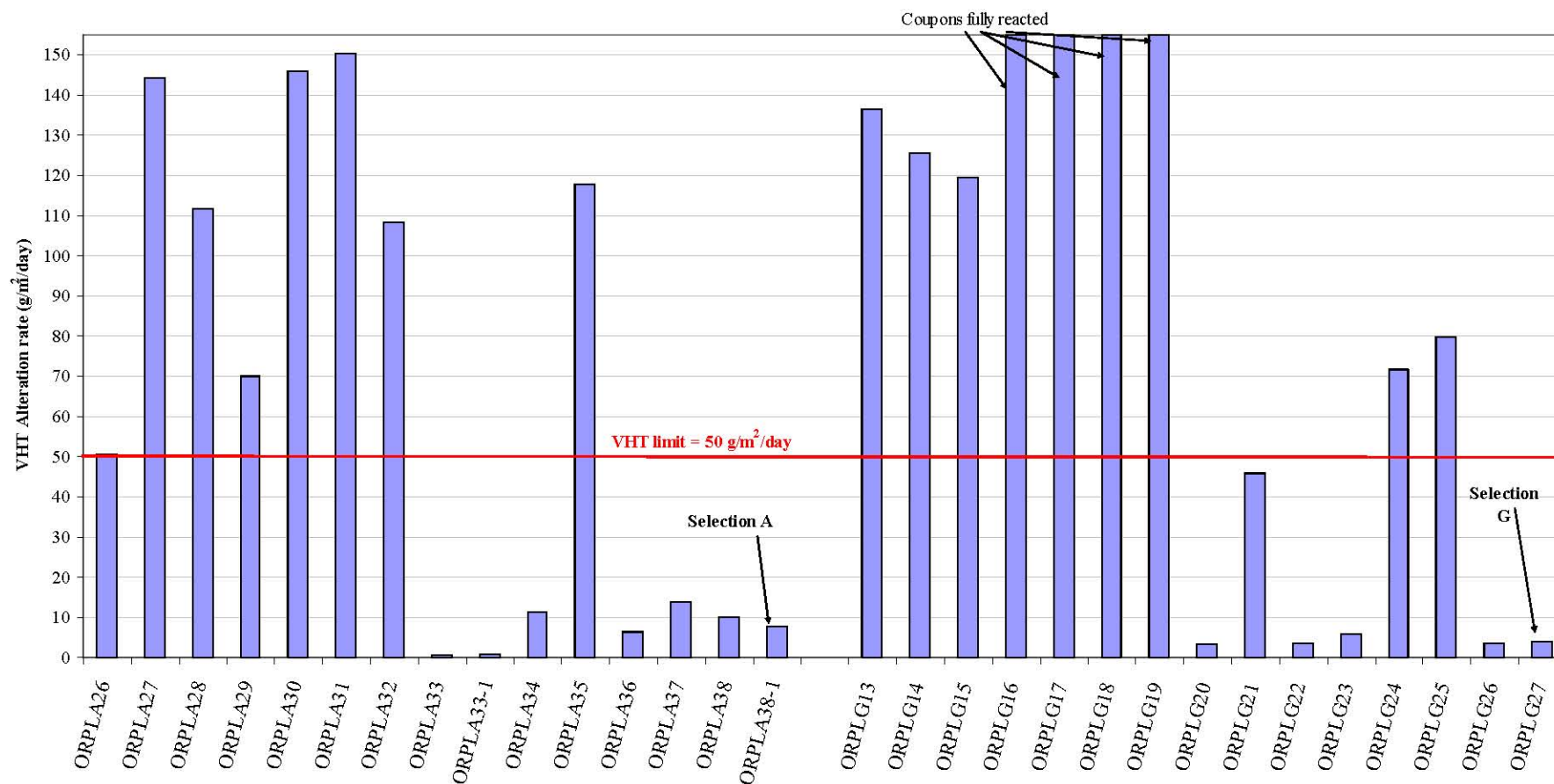
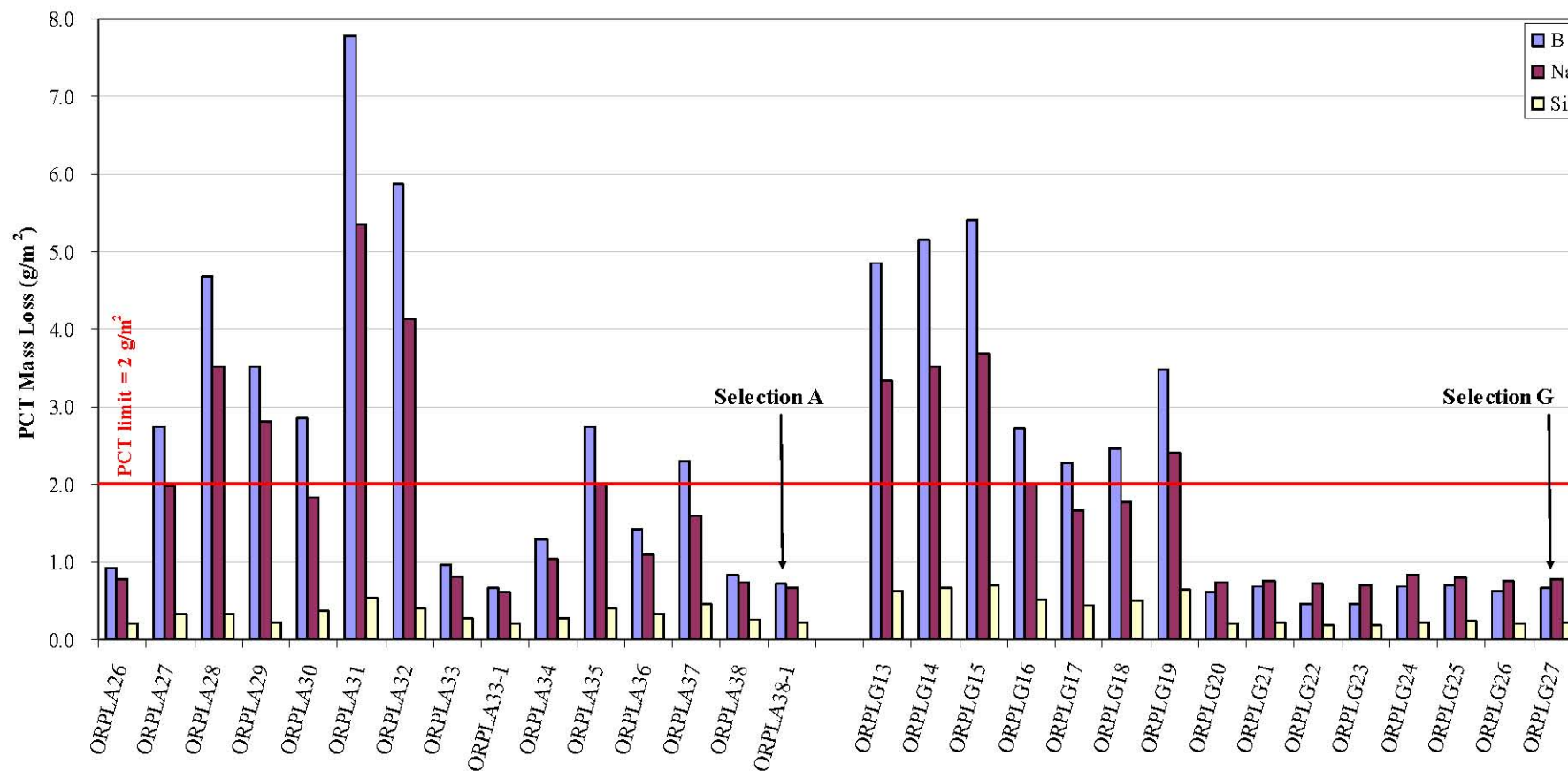


Figure 2.4. Sulfate solubility determined by remelting with excess SO<sub>3</sub> for thirty new ORP LAW crucible glasses.



**Figure 2.5. VHT results for thirty new ORP LAW crucible glasses.**  
(Relative standard deviation (RSD) of VHT measurement is estimated to be 31% [20])





**Figure 2.6. Normalized PCT releases for thirty new ORP LAW crucible glasses.**  
(Relative standard deviations (RSDs) of PCT measurements estimated from round robin testing are  
PCT-B 27%, PCT-Na 21%, and PCT-Si 15% [59])

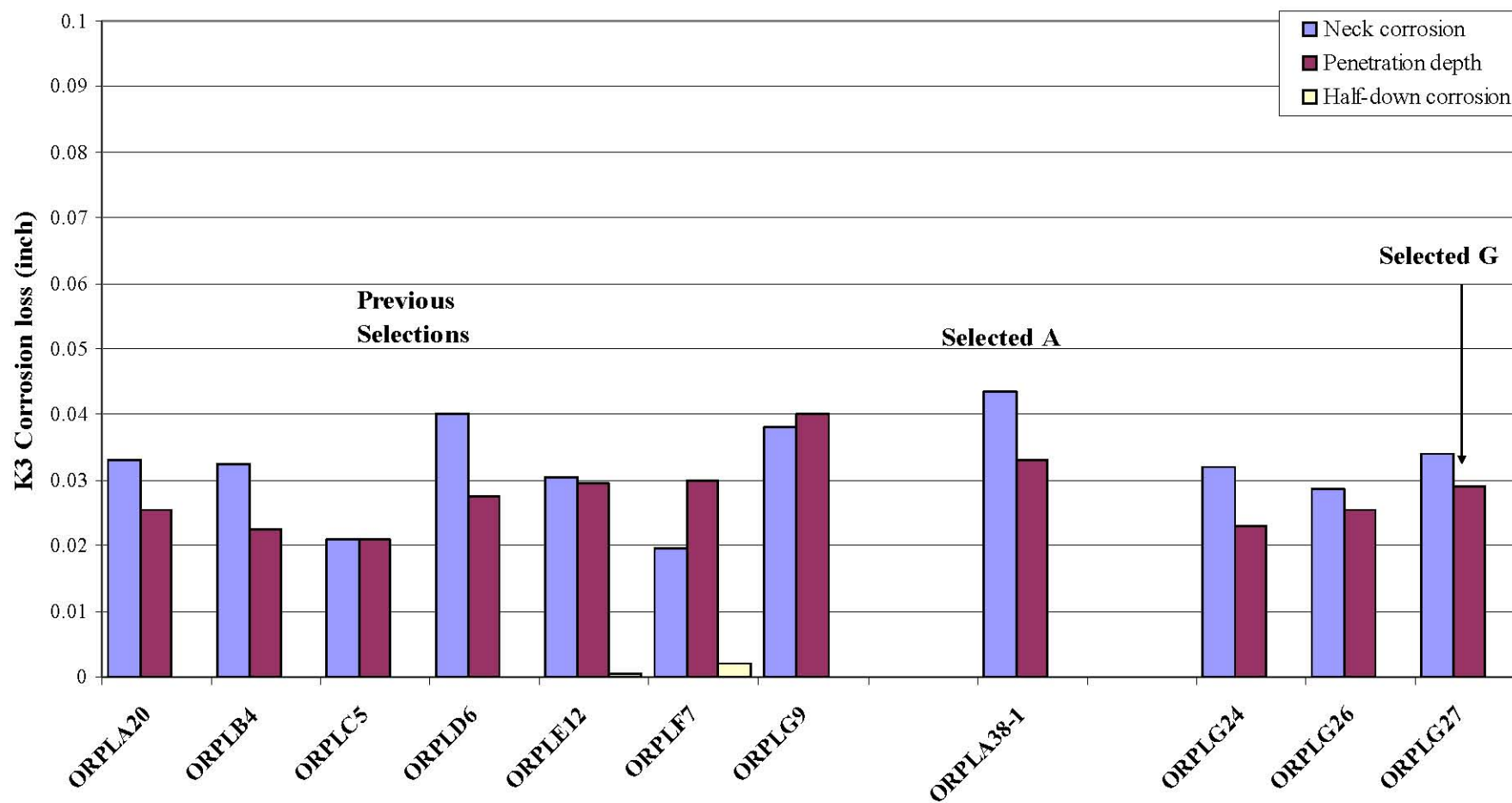
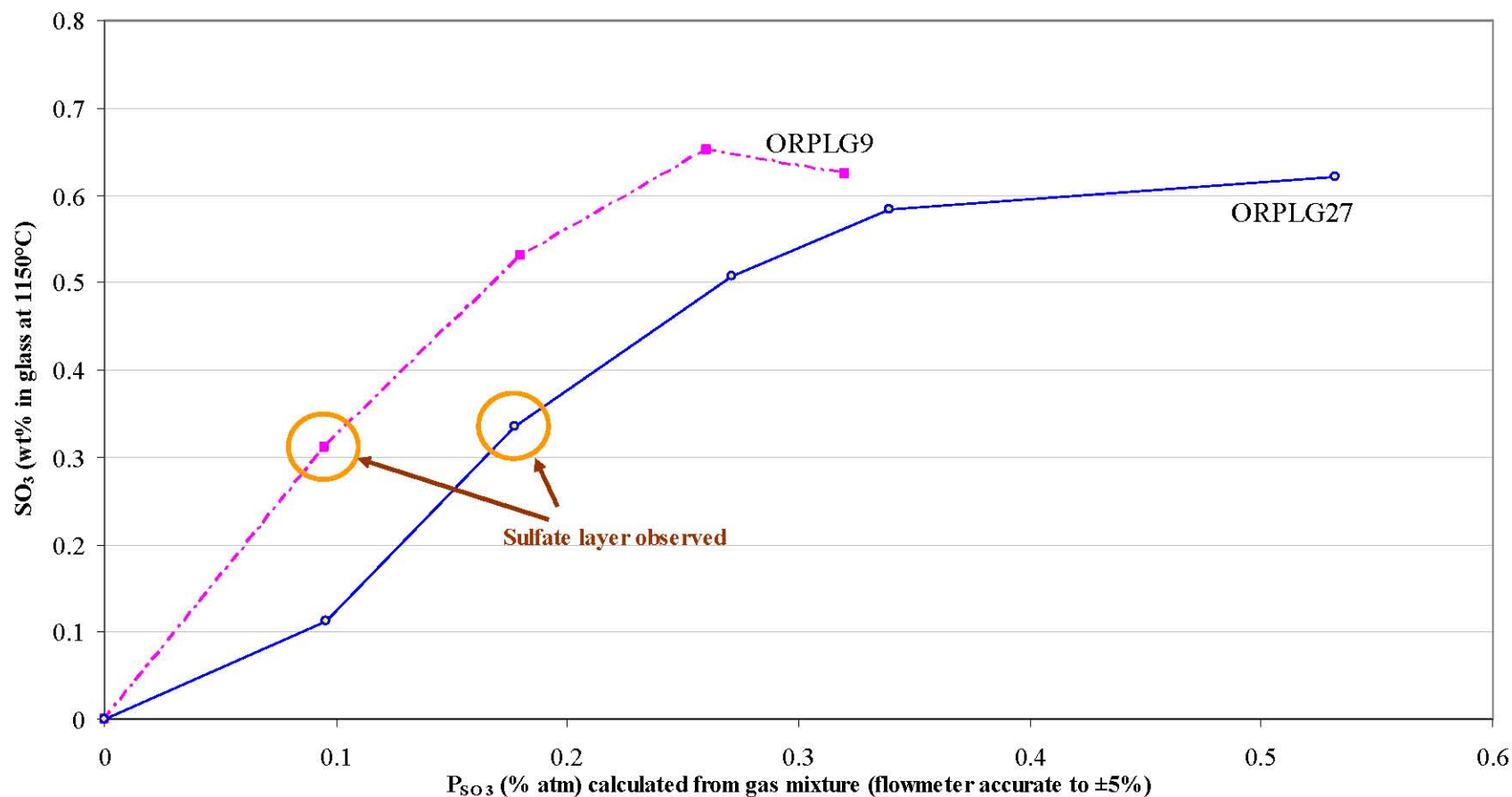
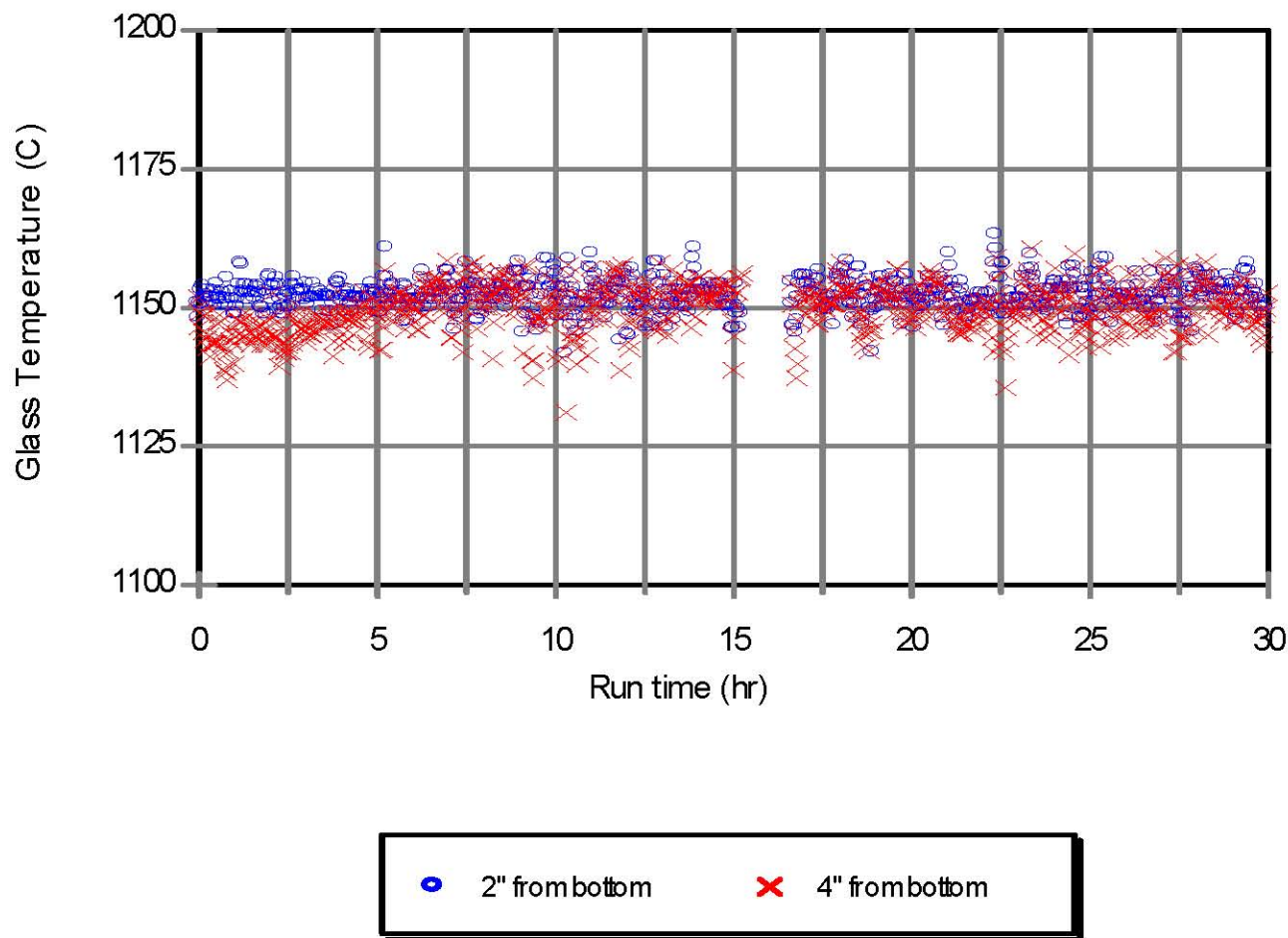


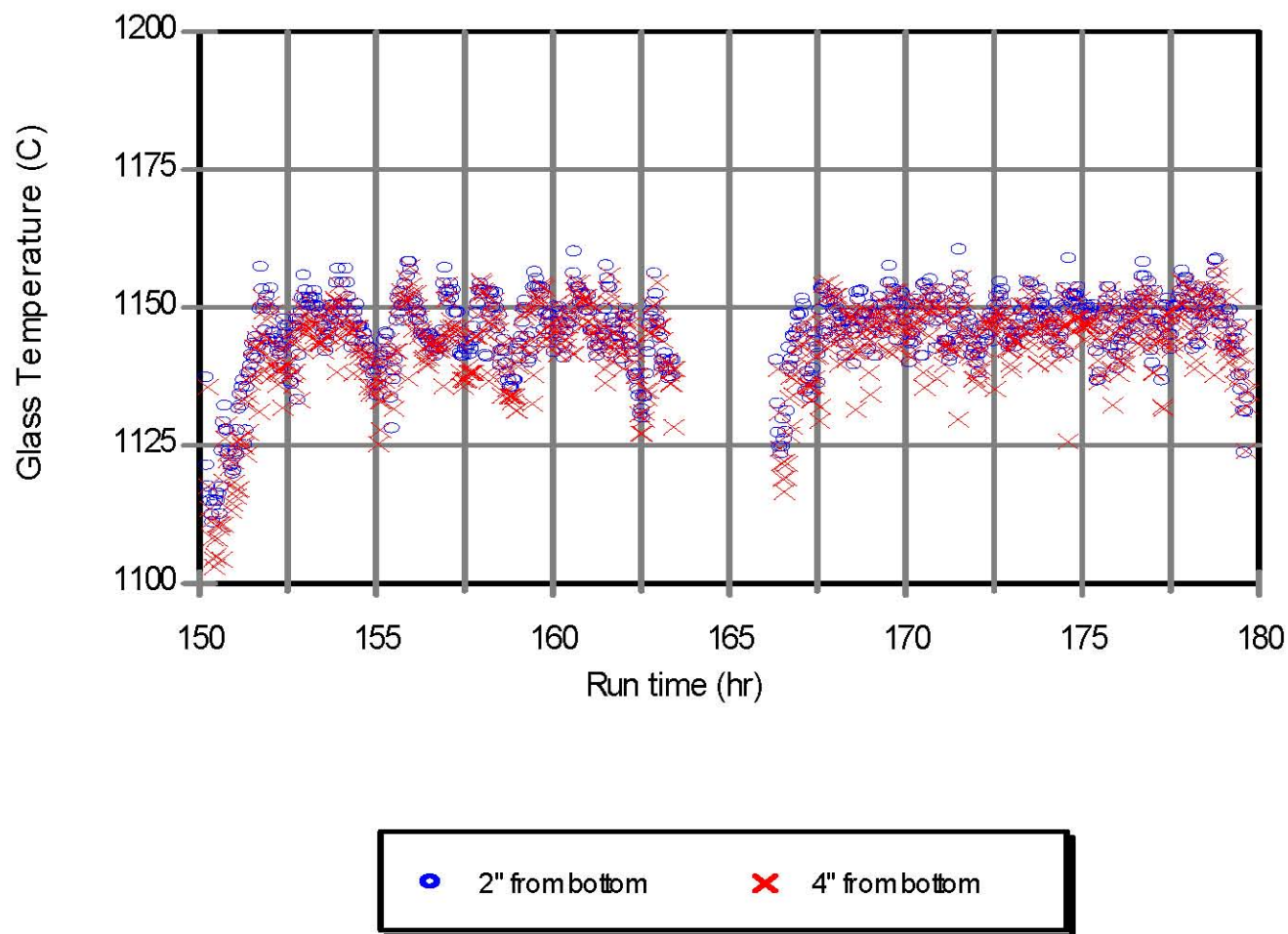
Figure 2.7. K-3 corrosion results for four new ORP LAW crucible glasses and seven previous formulations.



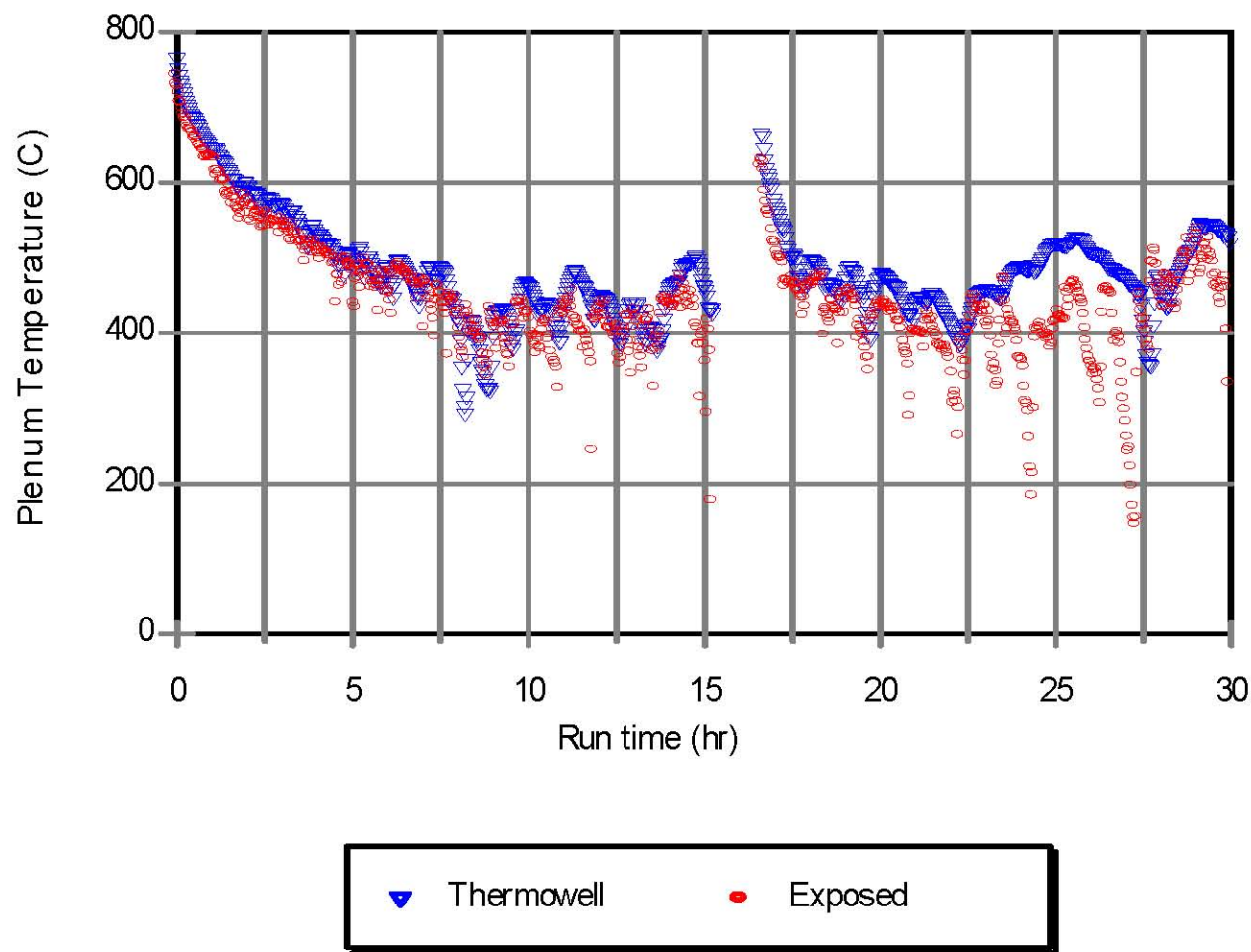
**Figure 2.8. Results of  $\text{SO}_2/\text{O}_2$  gas bubbling tests on the new ORP LAW glass ORPLG27 and the previous glass ORPLG9 at  $1150^\circ\text{C}$ . Partial pressure of  $\text{SO}_3$  vs. the  $\text{SO}_3$  concentration in the glass melt. The near-horizontal portions indicate the solubility limits while the slopes at lower concentrations provide measures of the activity coefficient of  $\text{SO}_3$  in the melt and the onset of a sulfate layer.**



**Figure 3.1.a. Representative plot of glass pool temperatures during DM10 tests. This plot is from the first two test segments performed (A1 and A2). The temperatures at 2" above the floor, which are most representative of the bulk glass temperature, closely approximate the target of 1150°C.  
(Temperature measurements are accurate to  $\pm 5^{\circ}\text{C}$ )**

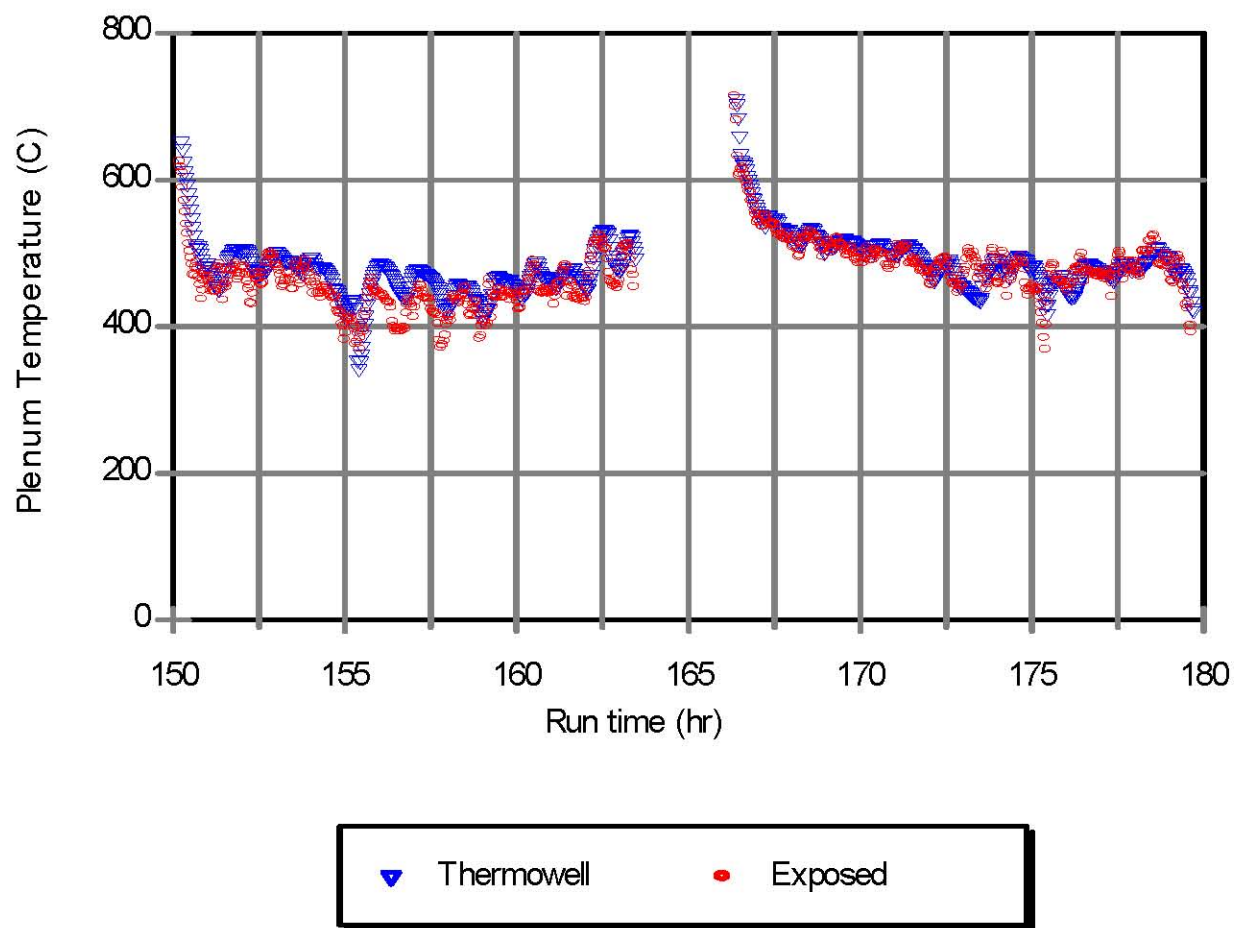


**Figure 3.1.b. Representative plot of glass pool temperatures during DM10 tests. This plot is from the last two test segments performed (B3 and B4). The temperatures at 2" above the floor, which are most representative of the bulk glass temperature, closely approximate the target of 1150°C. (Temperature measurements are accurate to  $\pm 5^{\circ}\text{C}$ )**

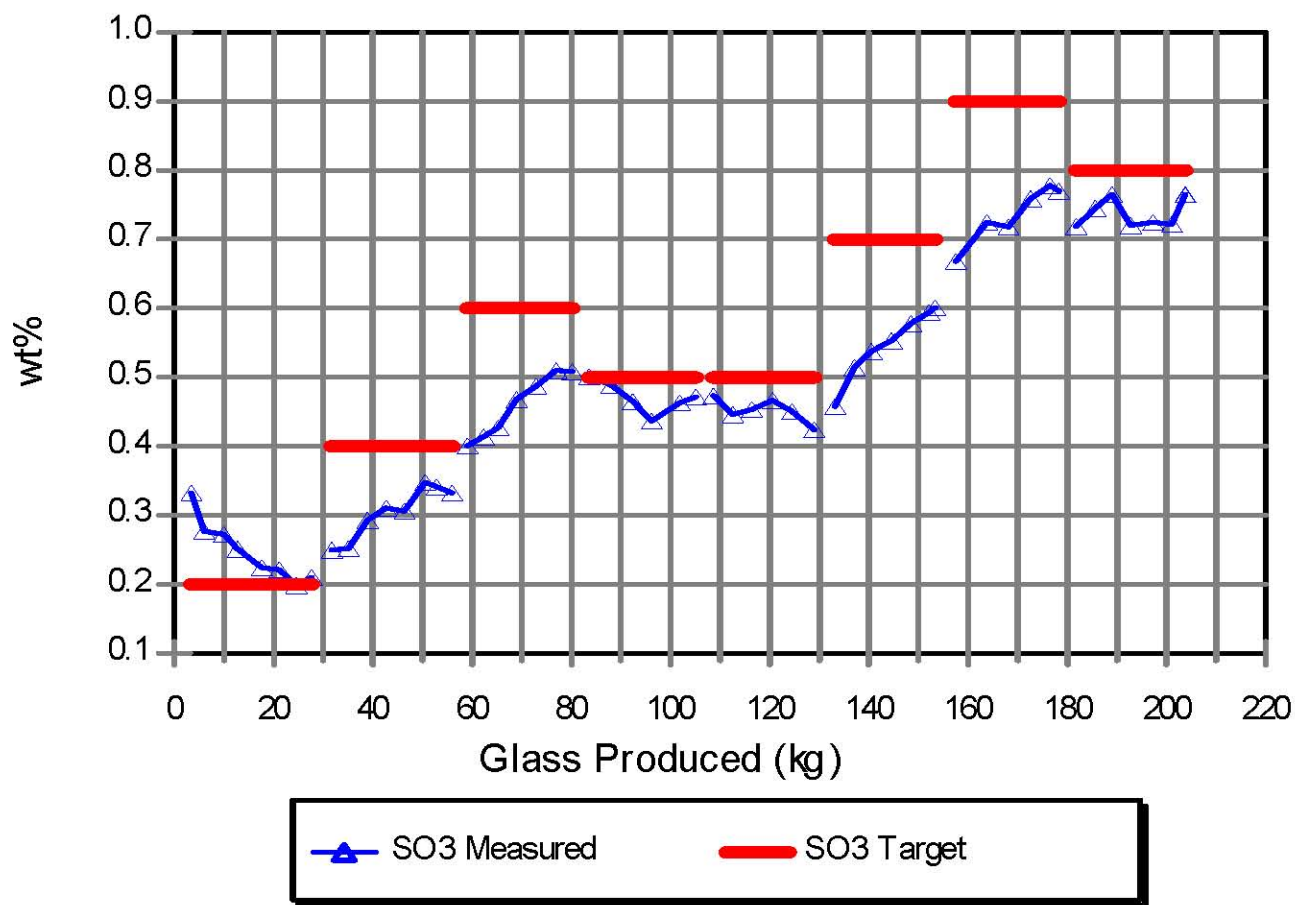


**Figure 3.2.a. Representative plot of plenum temperatures during DM10 tests. This plot is from the first two test segments performed (A1 and A2). The temperatures fall into the 550 to 350°C range during steady state processing (higher temperatures occurred at the beginning of testing and during sampling periods in between test segments). (Temperature measurements are accurate to  $\pm 5^{\circ}\text{C}$ )**





**Figure 3.2.b. Representative plot of plenum temperatures during DM10 tests. This plot is from the last two test segments performed (B3 and B4). The temperatures fall into the 500 to 350°C range during steady state processing (higher temperatures occurred at the beginning of testing and during sampling periods in between test segments). (Temperature measurements are accurate to  $\pm 5^{\circ}\text{C}$ )**

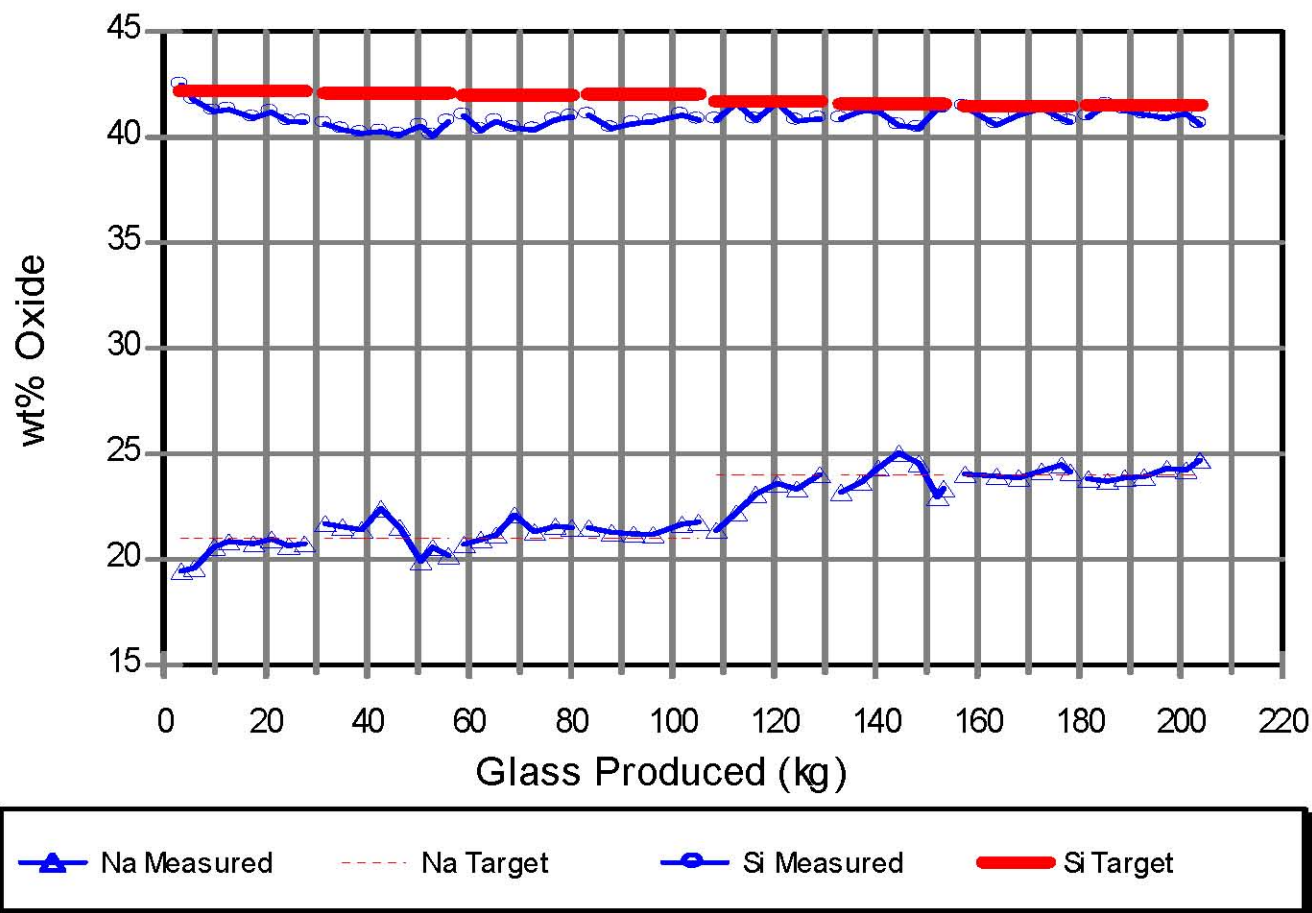


**Figure 3.3. XRF analysis of sulfur in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)

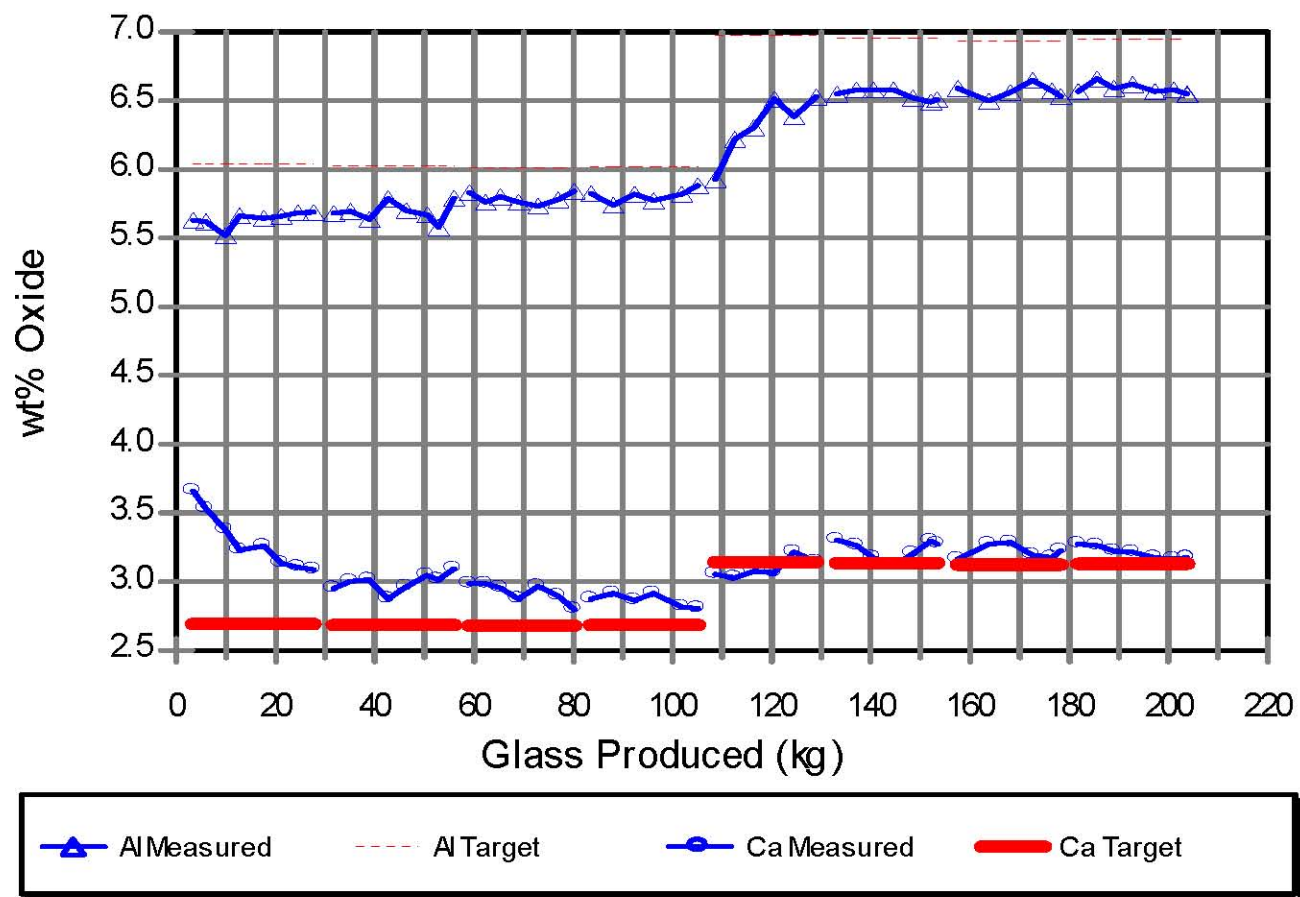




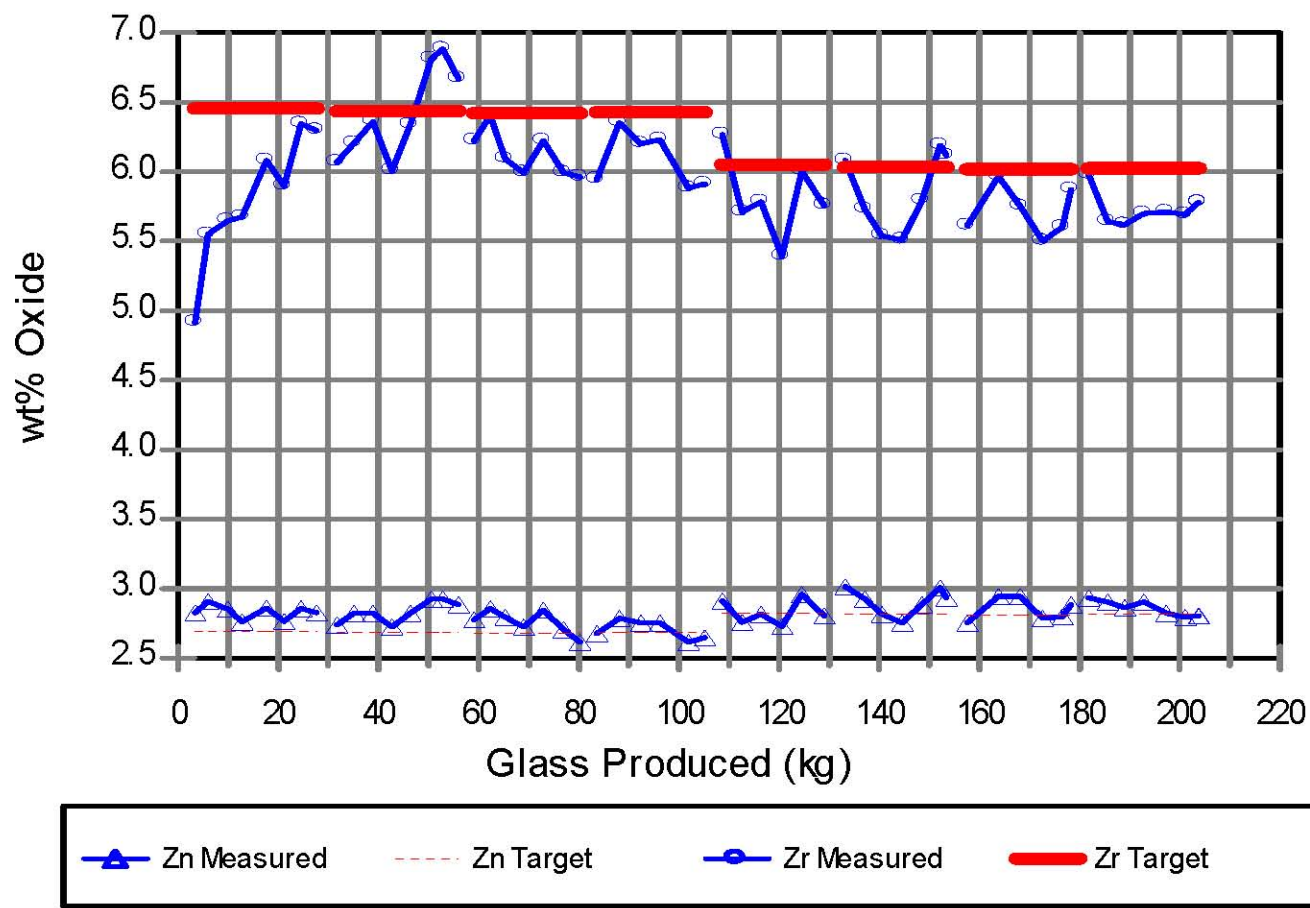
**Figure 4.1. Sulfate phase observed on the exterior surface of glass from the end of discharge during Test A2.**



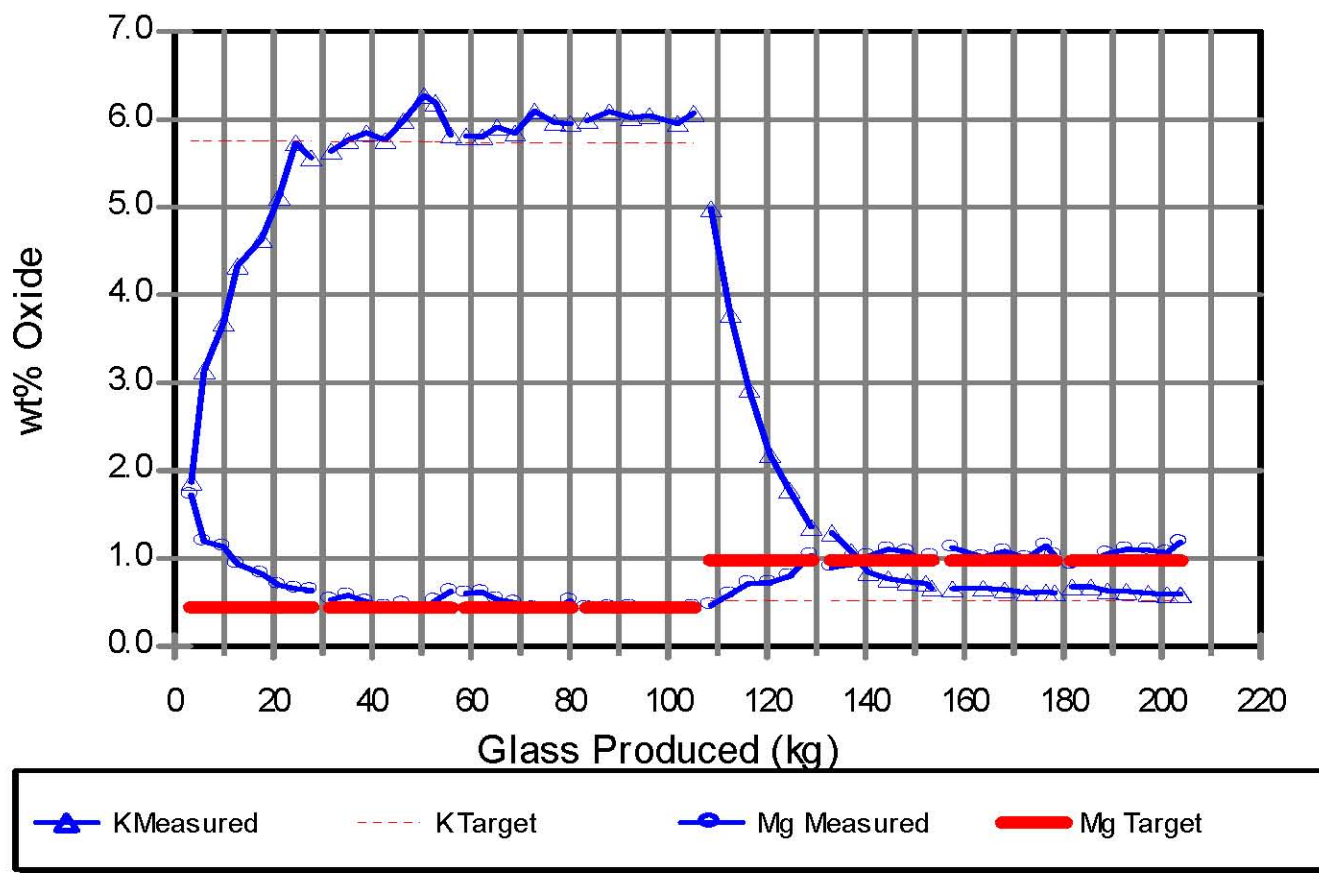
**Figure 4.2. XRF analysis of sodium and silicon in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)



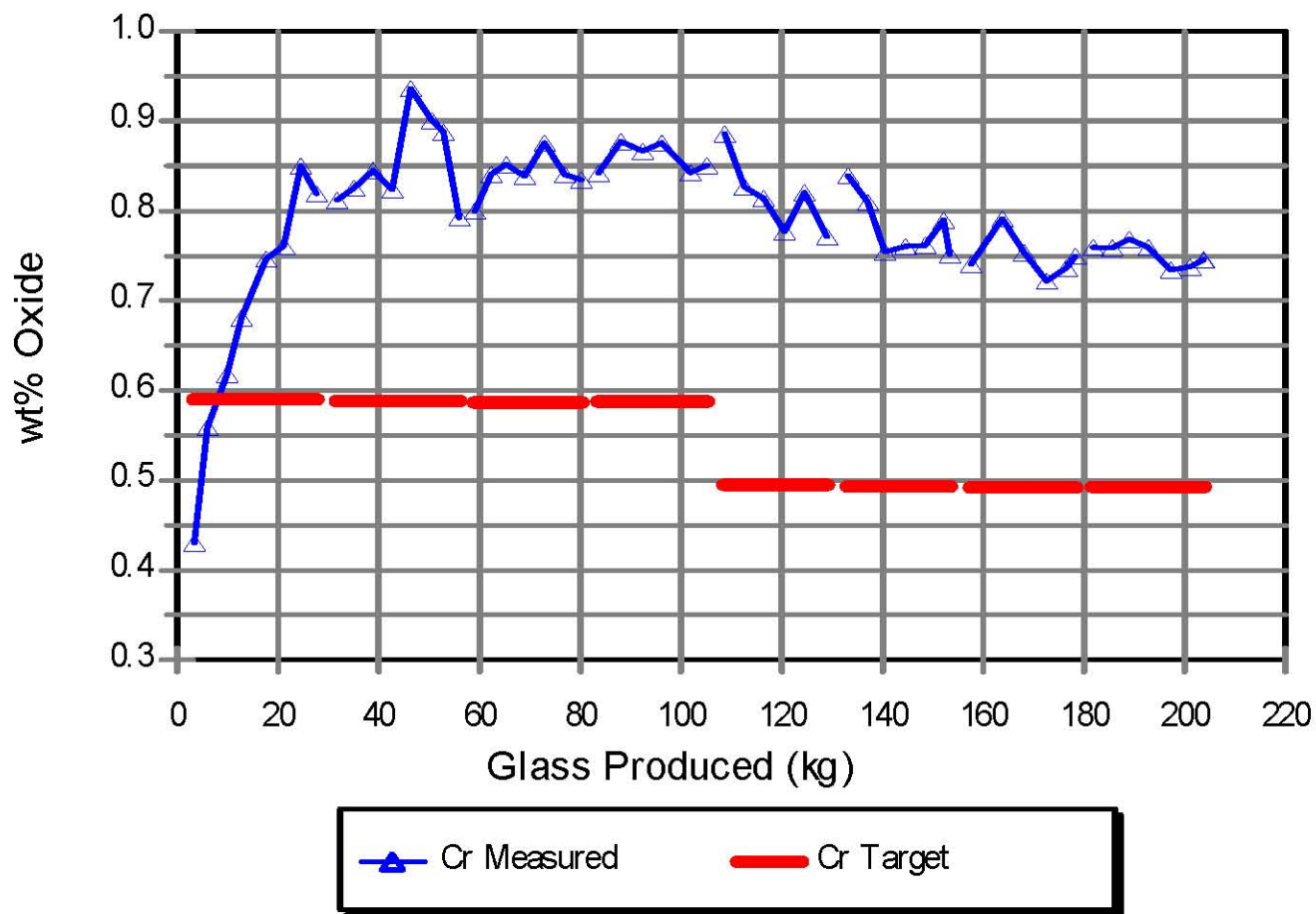
**Figure 4.3. XRF analysis of aluminum and calcium in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)



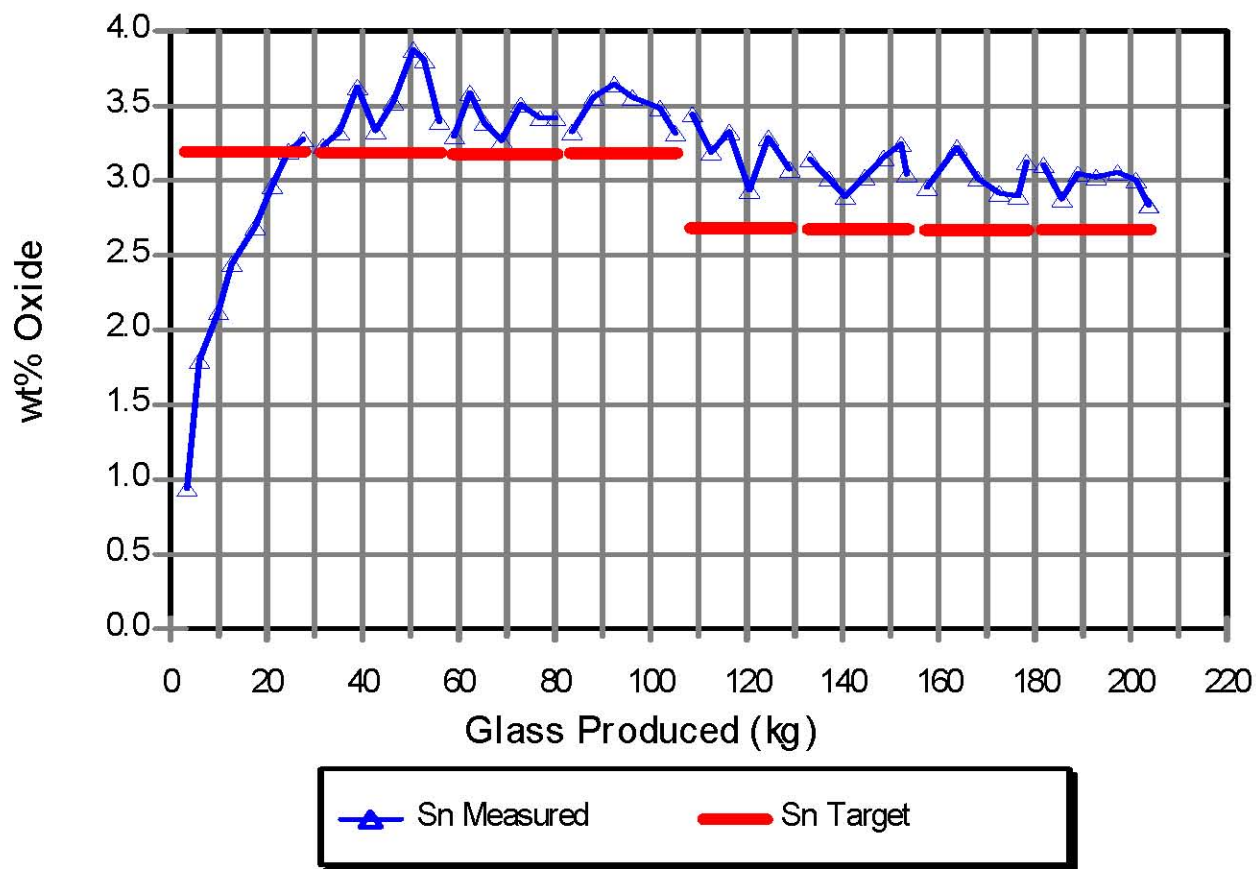
**Figure 4.4. XRF analysis of zinc and zirconium in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)



**Figure 4.5. XRF analysis of potassium and magnesium in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)

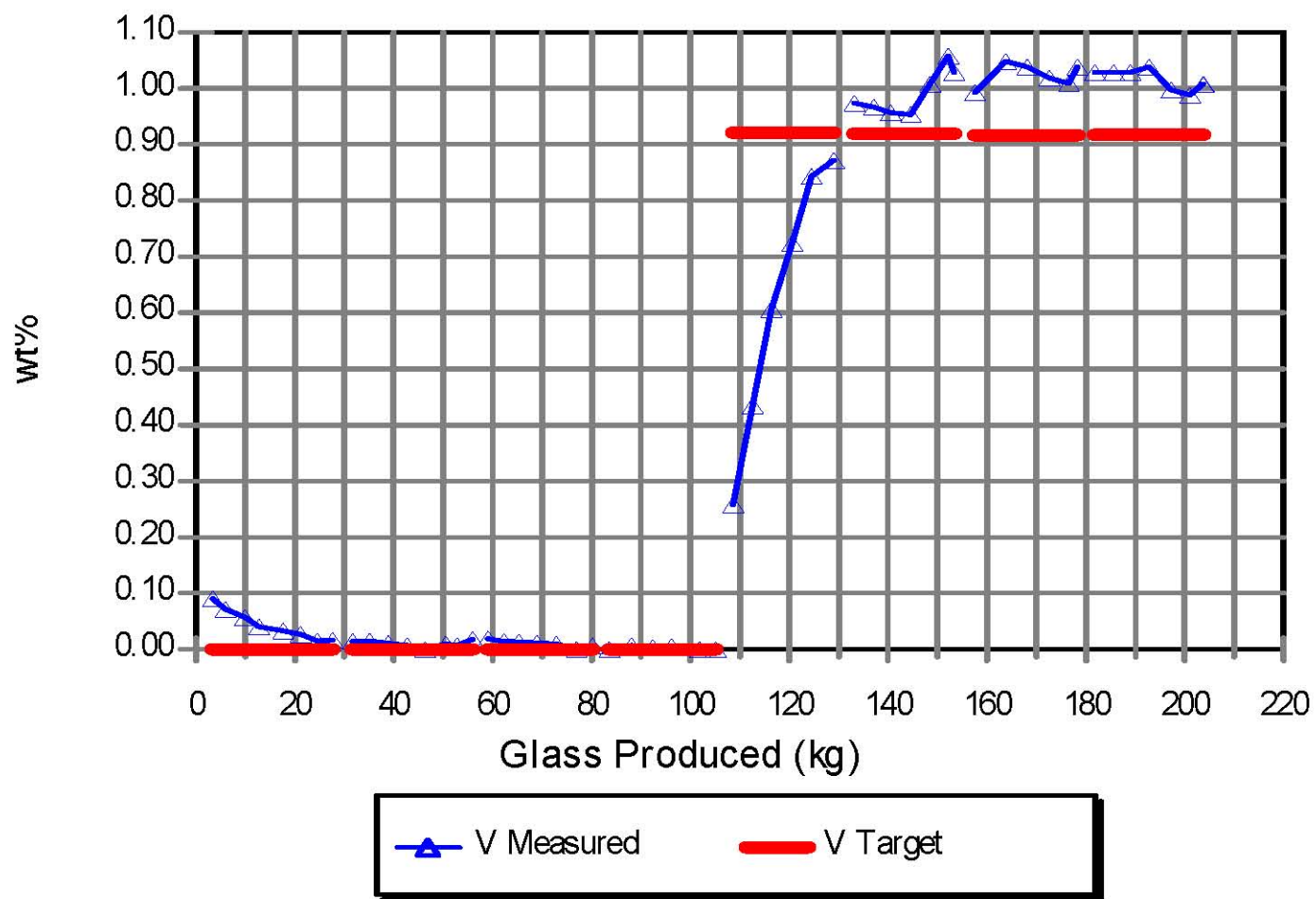


**Figure 4.6. XRF analysis of chromium in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)



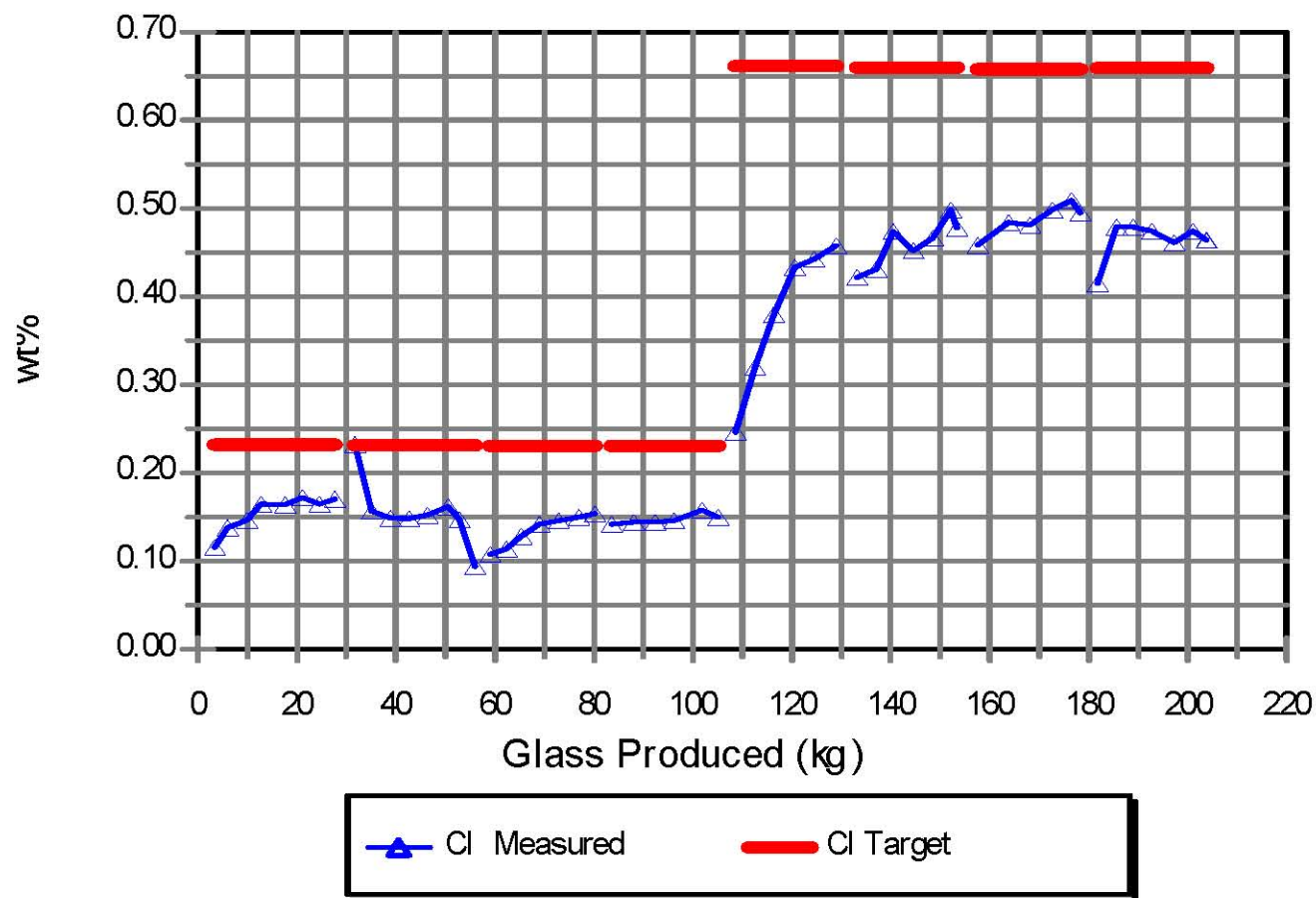
**Figure 4.7. XRF analysis of tin in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)





**Figure 4.8. XRF analysis of vanadium in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)





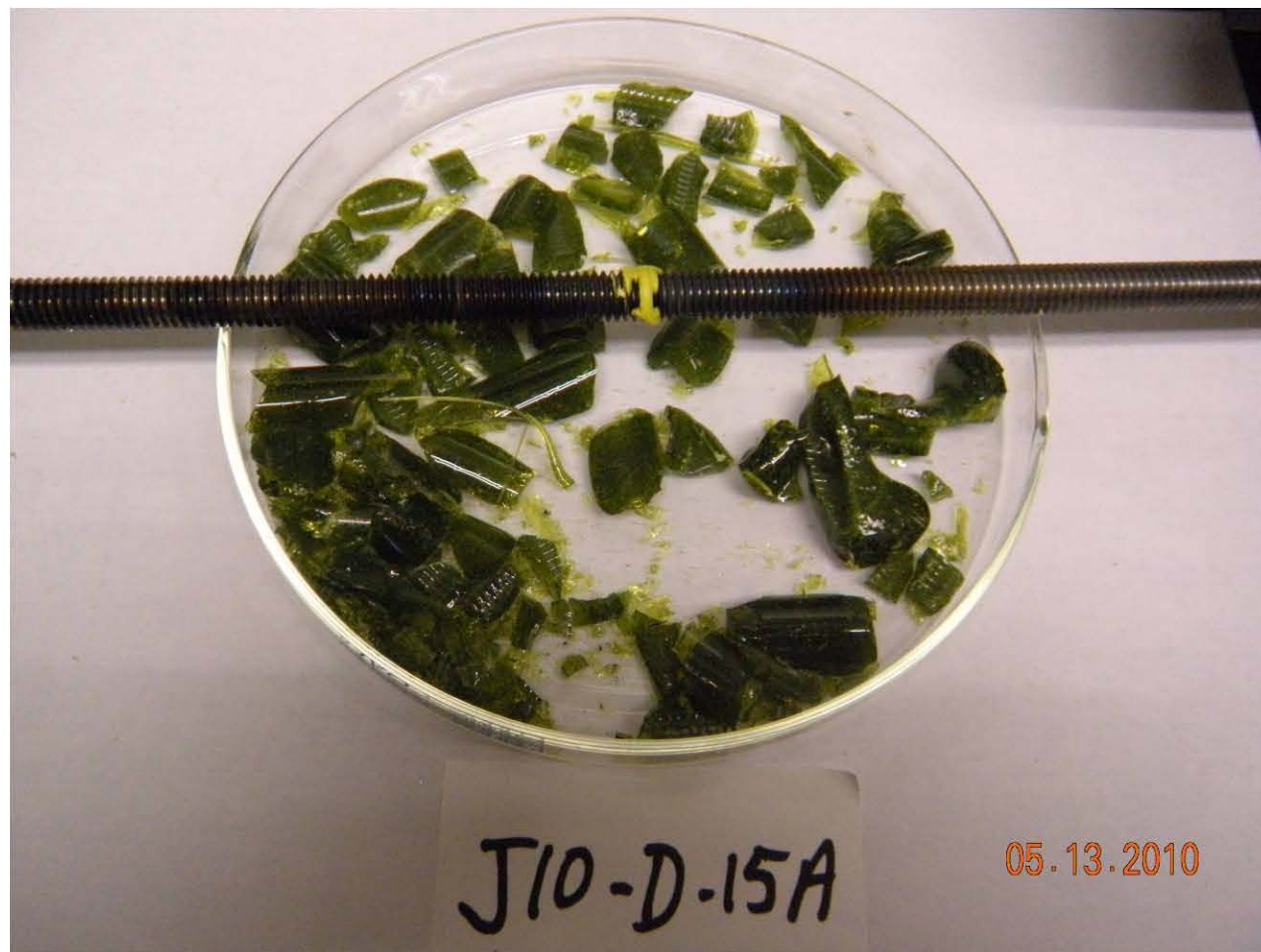
**Figure 4.9. XRF analysis of chlorine in DM10 product glasses.**  
(See Table 4.2 and Section 4.1 for details of composition analysis)



**Figure 4.10. Secondary sulfur phases on dip sample I10-D-123C from the end of Test A3.**



**Figure 4.11. Secondary sulfur phases on dip sample J10-D-15C from the end of Test B3.**



**Figure 4.12. Secondary sulfur phases on dip sample J10-D-15A from the end of Test B3.**



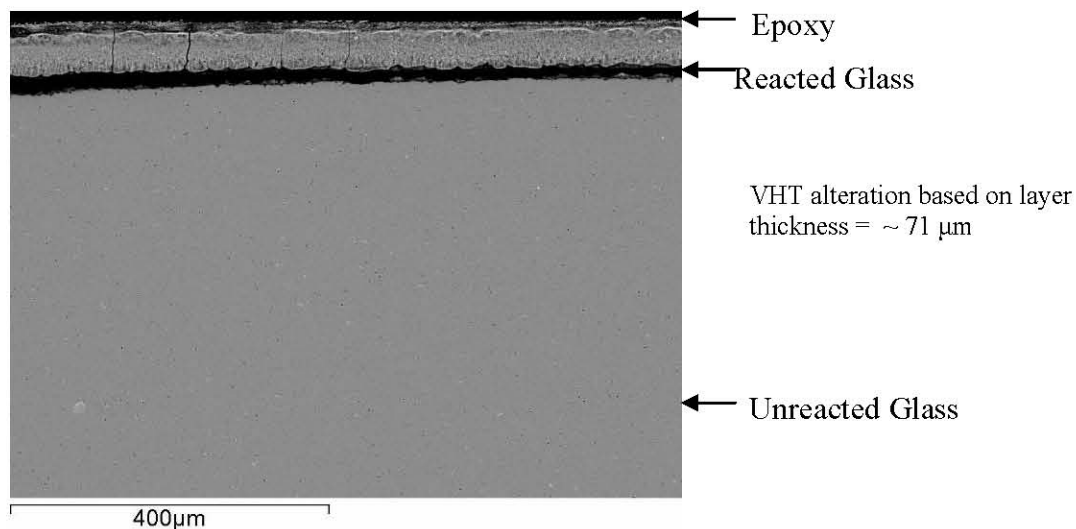


Figure 4.13 a. SEM image of cross section of crucible glass ORPLA38-1 after VHT.

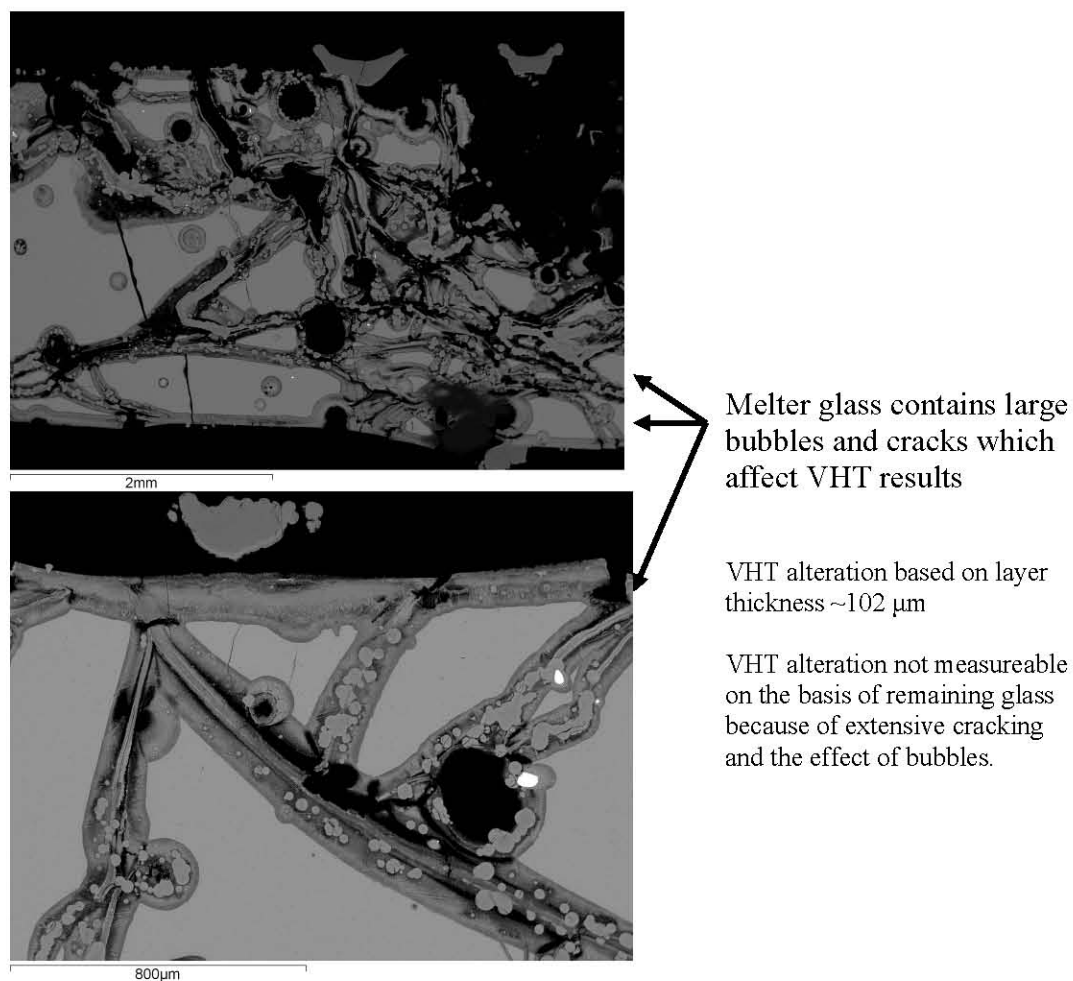


Figure 4.13 b. SEM images of cross section of DM10 melter glass J10-G-24B after VHT.

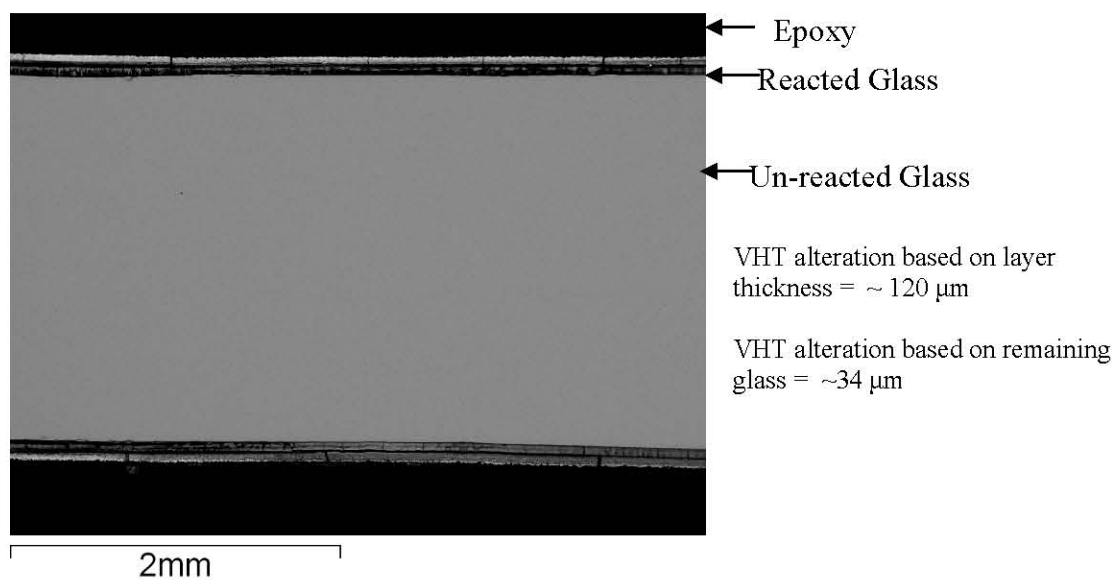


Figure 4.14 a. SEM image of cross section of crucible glass ORPLG27 after VHT.

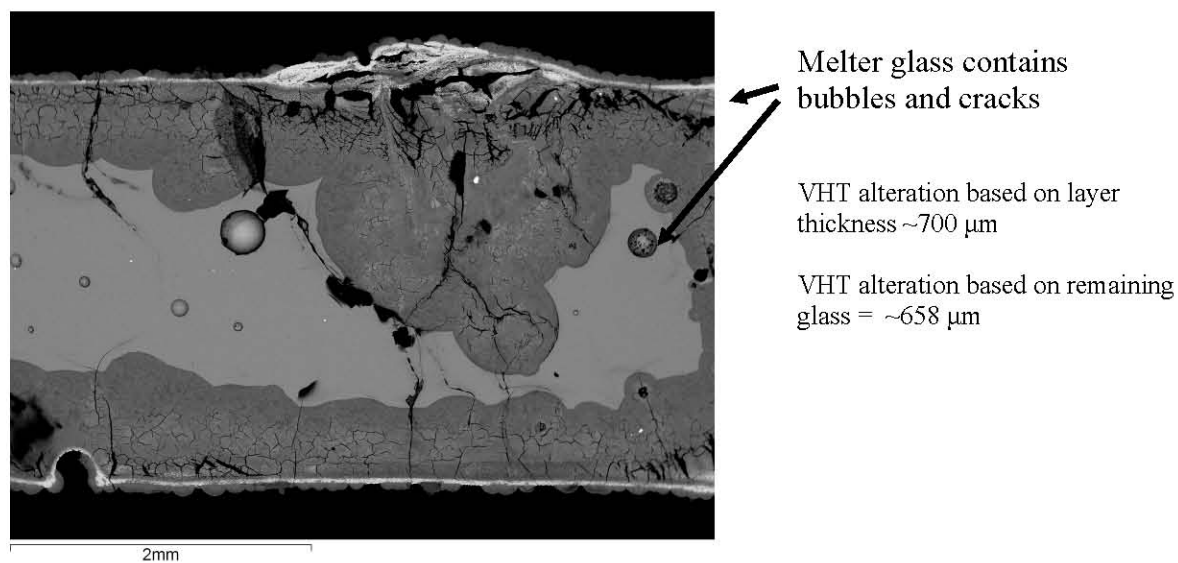


Figure 4.14 b. SEM image of cross section of DM10 melter glass J10-G-24B after VHT.

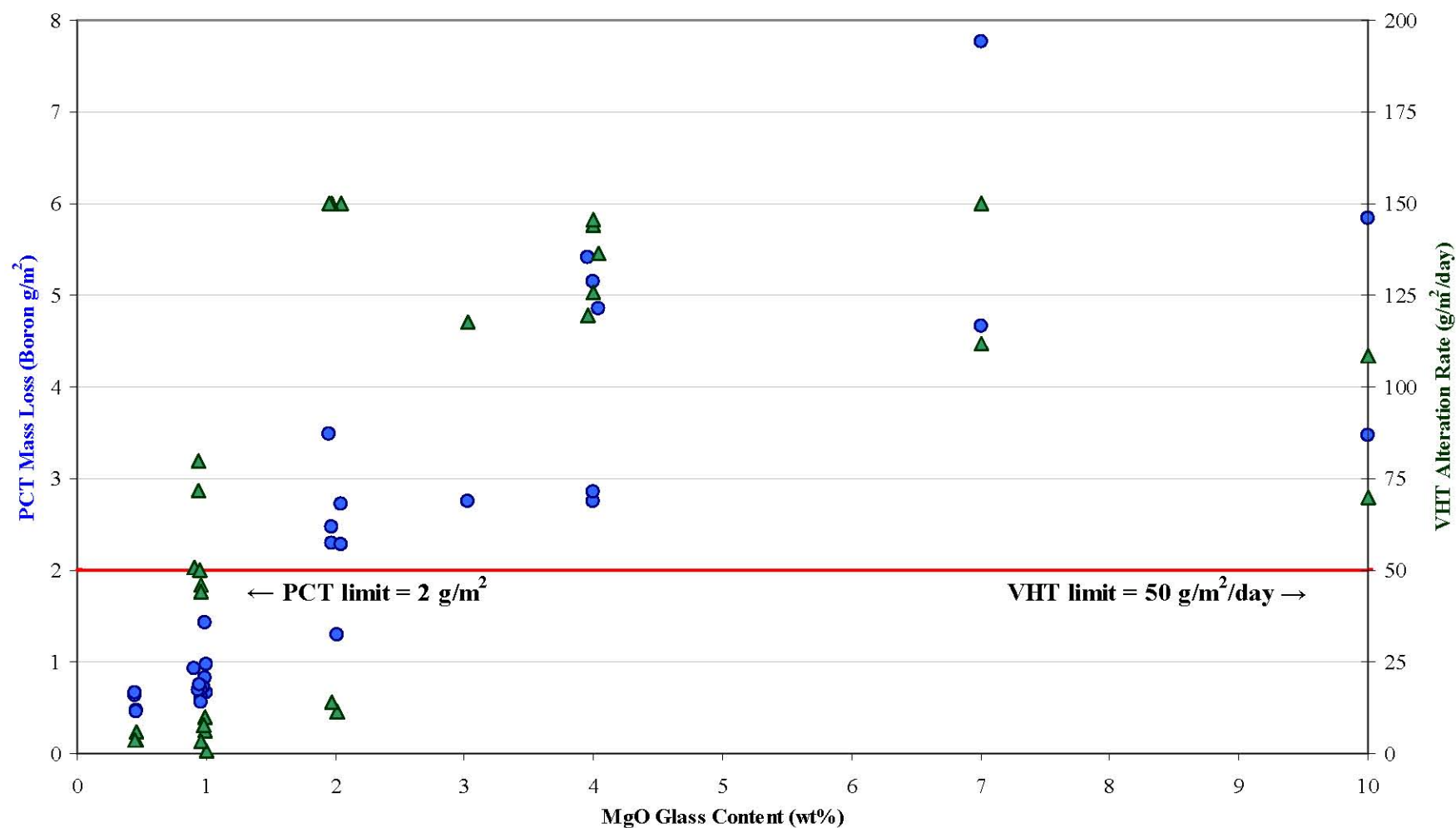


Figure 5.1. Effect of MgO on PCT (blue circles) and VHT (green triangles) for 30 ORP LAW glasses.

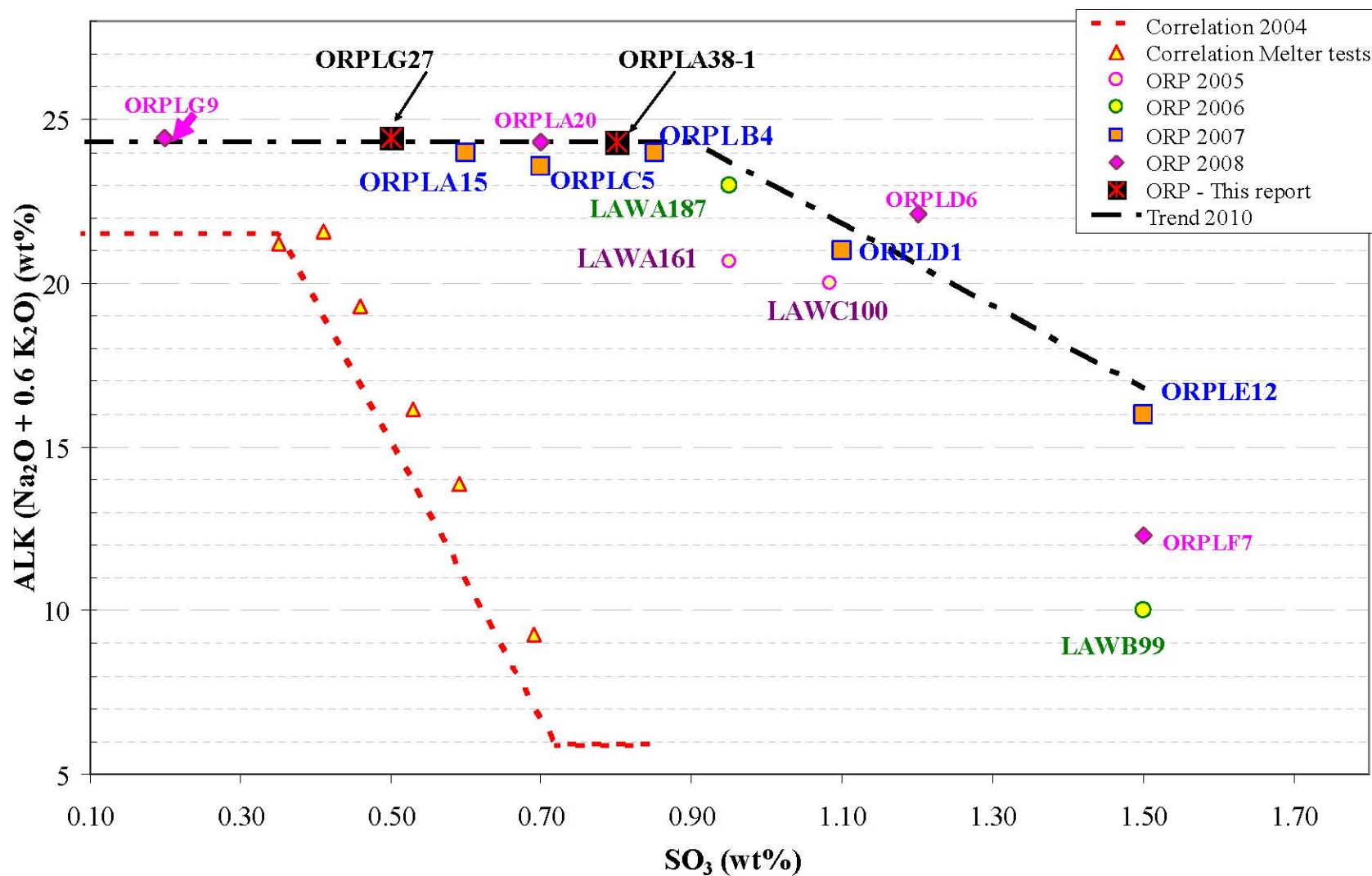


Figure 5.2. Overview of alkali oxides (Na<sub>2</sub>O and K<sub>2</sub>O) and SO<sub>3</sub> loadings for WTP and ORP LAW glasses.