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August 2002

Prepared for the U.S. Department of Energy
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Abstract

Studies were performed to develop and test a glass formulation for immobilization of sodium-bearing waste (SBW), which is a high soda, acidic, high-activity waste stored at the Idaho National Engineering and Environmental Laboratory (INEEL) in 10 underground tanks. It was determined in previous studies that SBW's sulfur content dictates its loading in borosilicate glasses to be melted by currently assumed processes. If the sulfur content (which is ~4.5 mass% SO_3 on a non-volatile oxide basis in SBW) of the melter feed is too high, then a molten, alkali-sulfate-containing salt phase accumulates on the melt surface. The avoidance of salt accumulation during the melter process and the maximization of sulfur incorporation into the glass melt were the main focus of this development work. A glass was developed for 20 mass% SBW (on a non-volatile oxide basis), which contained 0.91 mass% SO_3, that met all the processing and product-quality constraints determined for SBW vitrification at a planned INEEL treatment plant—SBW-22-20. This report summarizes the formulation efforts and presents the data developed on a series of glasses with simulated SBW.

Summary

The U.S. Department of Energy Office of Science and Technology (through the Tanks Focus Area) and the Office of Waste Management (through the Idaho National Engineering and Environmental Laboratory (INEEL) High-Level Waste Program) are sponsoring a joint effort by INEEL, Pacific Northwest National Laboratory, and the Savannah River Technology Center to investigate processes and formulations for vitrifying sodium-bearing waste (SBW). This report evaluates the option of directly vitrifying the SBW stored at INEEL, which would involve either no feed conditioning or at the most minimal conditioning before vitrification. Various other projects have developed technologies for vitrifying waste, but the composition of SBW differs significantly from those wastes, and it is unlikely that additive compositions developed for them could be applied to SBW in a cost-effective manner. SBW is similar to Hanford low-activity waste (LAW), however, making it possible to use the results of development activities for Hanford LAW vitrification to some extent. This report updates the status of the glass-formulation efforts in support of melter demonstrations.

The studies described in this report focused on efforts to develop a flowsheet for vitrifying waste stored in INEEL Tank WM-180. An acceptable glass must meet product properties required for waste-form acceptance and also allow for adequate operation of the melter. Acceptable glass must conform to limitations on the response of glass to the product consistency test, and chemical and phase-stability information must be reported. The glass formulation was developed assuming properties that would be pertinent to processing either in a liquid-fed, Joule-heated, ceramic-lined melter (JHCM) or in a cold-crucible induction melter (CCIM), both of which are considered viable options for treating SBW. The processing constraints are not identical for the two technologies, but the goal was to develop a glass that would work with either technology. Glass compositions for immobilizing SBW waste were formulated based on models that relate glass properties to composition.

A liquid simulant for WM-180 waste was prepared after a series of crucible tests using reagent-grade oxides, carbonates, and boric acid. The objective of these tests was to assess various additive components and their effects on sulfate solubility and glass properties of interest. Based on the batch chemical testing results, only the following 6 of the 17 additive compositions produced glasses that met all of the constraints: SBW-12, -14, -16, -22, -26, and -27 (although it should be noted that PCT was not performed on -26 and -27 based glasses).

To gain a better understanding of the effects of glass composition on sulfur partitioning from a slurry-fed melter operation, tests were performed to better simulate that process than was allowed by the crucible melts. WM-180 simulant was fabricated, and test experiments were performed. Ramp-heated crucible melt tests were performed to determine the appropriate waste loading (WL), the need for any acid additions or adjustments, and appropriate additive compositions. The centimeter scale melter (CSM) was developed to better simulate those processes that are important to determining the behavior of sulfur in a slurry-fed melter system with batch heating from the glass melt below. No significant difference between the sulfur partitioning was seen in tests with CaO or V_2O_5 or both being removed from the frit or between tests with frit and the hydroxide based feeds.

Before research-scale melter (RSM) or pilot scale tests, it was desirable to evaluate various additive formulations (or melter feeds) for their potential to form salt layers using the slurry-fed melt-rate furnace (SMRF). The results of SMRF were similar to crucible melts and CSM melts.

The results from the tests presented above suggest that the SBW-22 frit (or additive mix) is appropriate for scale-up tests for the direct vitrification of 2001 WM-180 simulant at 20 mass% loading. The main focus of formulation work and testing was minimizing the likelihood of salt accumulation and maximizing sulfur retention in the melt. Since these are both strong functions of the chemical and physical processes involved in the melter, it is difficult to extrapolate the results from smaller scale tests performed during this study to the melter. Therefore, it is recommended that the glass be fabricated in scaled melter tests to assure that processability is adequate at a larger scale.

Glossary

ARM	Approved Reference Material (ARM-1)
ASTM	American Society for Testing and Materials
CCC	centerline canister cooling
CCIM	cold-crucible induction melter
CETL	Clemson Environmental Technology Laboratory
CSM	centimeter scale melter
CVS	composition variation study
DIW	deionized water
DOE	U.S. Department of Energy
DWPF	Defense Waste Processing Facility
EA	environmental assessment
GC-MS	gas chromatography-mass spectrometry
GFC	glass-forming chemical
HLW	high-level waste
IC	ion chromatography
ICP-AES	inductively coupled plasma-atomic emission spectroscopy
ICP-OES	inductively coupled plasma-optical emission spectrometry
ICPP	Idaho Chemical Processing Plant
INEEL	Idaho National Environmental and Engineering Laboratory
INTEC	Idaho Nuclear Technology and Engineering Center
JHCM	Joule-heated, ceramic-lined melter
LAW	low-activity waste
PCT	Product Consistency Test
PNNL	Pacific Northwest National Laboratory
RH-TRU	remote-handled transuranic (waste)
RSM	research-scale melter
SBW	sodium-bearing waste

SMRF	slurry fed melt rate furnace
SRS	Savannah River Site
SRTC	Savannah River Technology Center
T	temperature
TFA	Tanks Focus Area
T_L	liquidus temperature
η	viscosity
WAPS	Waste Acceptance Product Specification
WIPP	Waste Isolation Pilot Plant
WL	waste loading
WSRC	Westinghouse Savannah River Company
WVDP	West Valley Demonstration Project
XRD	X-ray diffraction
XRF	X-ray fluorescence spectroscopy

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1.0 Introduction

For about four decades (beginning in the early 1960s), radioactive wastes have been collected and calcined from nuclear-fuels reprocessing at the Idaho Nuclear Technology and Engineering Center (INTEC), formerly Idaho Chemical Processing Plant (ICPP).(a) Over this time span, secondary radioactive wastes have also been collected and stored as liquid from reprocessing, decontamination, laboratory, and fuel-storage activities. These liquid wastes are collectively called sodium-bearing wastes (SBW). About 3.8 M L (1 M gal) of these wastes are temporarily stored in stainless steel tanks at the Idaho National Engineering and Environmental Laboratory (INEEL). These liquid SBWs may be vitrified and sent to the federal geologic repository or the Waste Isolation Pilot Plant (WIPP) for final disposal.

The Batt Settlement Agreement was established in August 1995 between the U.S. Navy, the State of Idaho, and the U.S. Department of Energy (DOE 1995). One of the requirements per this agreement is to remove liquid wastes (i.e., SBW) from and cease the use of the INEEL liquid waste storage tanks by the end of 2012. More immediately, technology must be developed to provide information required to initiate the design of the SBW treatment facility in the year 2002. Direct vitrification (no or minimal feed conditioning before vitrification) is being considered as a possible immobilization process for SBW, which is the focus of this report.(b)

Vitrification is considered the “Best Demonstrated Available Technology” for immobilizing high-level waste (HLW). Precedents for vitrifying INTEC HLW into borosilicate glass have been established by the production-scale operation of the Defense Waste Processing Facility (DWPF) at the Savannah River Site (SRS), the West Valley Demonstration Project (WVDP) at West Valley, New York, and certain European facilities. Production-scale technologies exist for the waste chemistries at these plants. However, it is unlikely that the additive (or frit) formulations developed for immobilizing waste compositions at these sites could be applied to the SBW in a cost-effective manner. The composition of SBW differs significantly from those wastes being vitrified at the DWPF and the WVDP. However, there are similarities between the compositions of SBW and Hanford’s low-activity waste (LAW). Therefore, efforts are underway at INEEL to develop integrated flowsheets and identify glass-composition regions for vitrifying SBW while utilizing the results of development activities for Hanford LAW vitrification to the extent possible.

The U.S. Department of Energy (DOE) Office of Science and Technology (through the Tanks Focus Area [TFA]) and the Office of Waste Management (through the INEEL High-Level Waste Program) are sponsoring a joint effort by INEEL, Pacific Northwest National Laboratory (PNNL), and the Savannah River Technology Center (SRTC) to investigate processes and formulations for vitrifying SBW. The overall strategy of this integrated task includes developing glass-property data and glass-composition/property models that cover the composition region expected from individual and/or blended SBWs. The strategy, in terms of glass-formulation activities, has focused on two primary tasks:

(a) The Idaho National Engineering and Environmental Laboratory, located in Eastern Idaho, consists of an 890-square-mile reservation located 32 miles west of Idaho Falls, Idaho.

(b) Subsequent to the completion of this work, INEEL’s Environmental Impact Statement indicated that vitrification of SBW would not be pursued. Alternatives or options being considered include: calcination; steam reforming; cesium ion exchange; and direct evaporation.

1) collaborative (phased) glass composition variation studies (CVS) that “bound” the composition regions of interest to proposed direct-vitrification flowsheets and 2) preliminary glass-formulation development aimed at specific waste streams to perform melter tests to demonstrate feasibility and collect data required for plant design. Both tasks are focused on providing data required to establish a baseline flowsheet for SBW vitrification.

Edwards et al. (2001) and Scholes et al. (2002) described the SBW CVS design and results. Previous glass-formulation efforts for SBW were reported by Vienna et al. (1999) and Peeler et al. (2001). The results of melter tests using SBW were reported by Goles et al. (2001) and Perry et al. (2001). Preliminary results to identify the parameters influencing sulfur partitioning between the glass melt, off-gas, and a molten salt phase were summarized by Darab et al. (2001), with more in-depth analyses reported by Vienna et al. (2002). The results from radioactive tests with WM-180 tank samples were reported by Olson et al. (2001). This report gives an updated status of the glass-formulation efforts in support of melter demonstrations.

2.0 Waste-Stream Basis for Current Testing

The INEEL SBW is stored in 10 tanks with one spare. These wastes will be consolidated into four tanks (WM-180, -188, -189, and -187) before treatment. In early 1999, INTEC supplied an estimate of the blended SBW composition.[(a)] Table 2.1shows this 1998 SBW composition, which was used in the formulation work of Vienna et al. (1999). The focus of further studies was changed to the waste found in Tank WM-180. This tank was scheduled for processing first, and the content was not to be changed by the consolidation of wastes into WM-188 and -189. The initial composition of WM-180, supplied by INTEC[(b)] and listed in Table 2.1as 2000 WM-180, was the focus of the formulation work of Peeler et al. (2001). The composition of WM-180 was updated with new tank-characterization data. Christian (2001) reports the 2001 WM-180 composition (shown in Table 2.1), which like the previous WM-180 composition contains a small fraction of undissolved solids. Comparing these waste compositions, the most substantial difference (with reference to waste vitrification) is in the concentration of SO_3. The concentration of SO_3 increases from 3.57 to 4.55 mass% when changing from 2000 WM-180 to 2001 WM-180. Since previous formulation studies with WM-180 have shown that the loading of SBW in alkali-borosilicate waste glasses was limited by the allowable sulfur content, this ~20% increase in sulfur content will likely result in a decrease in waste loading (WL).

(a) C. A. Musick e-mail to J.D. Vienna (PNNL), D. F. Bickford (SRTC), C. M. Jantzen (SRTC), and D. K. Peeler (SRTC); dated January 19, 1999; Subject, “Direct Vitrification for SBW Composition,” Idaho National Engineering and Environmental Laboratory, Idaho Falls, Idaho.

(b) J. D. Christian e-mail to T.A. Batcheller, T.G. Garn, D.L. Griffith, R.R. Kimmitt, J.A. McCray, A.L. Olson, D.K. Peeler, D.D. Siemer, N.R. Soelberg, and J.D. Vienna, dated November 6, 2000, Subject: WM-180 Simulant.

Table 2.1. Comparison of SBW Simulant Compositions Used in Formulation and Testing

Component	1998 SBW	2000 WM-180	2001 WM-180
Oxides (mass% non-volatile oxides)			
Al_2O_3	27.34	27.96	27.52
As_2O_3	0	0	0.04
B_2O_3	0.65	0.35	0.35
BaO	0	0.01	0.01
CaO	2.23	2.22	2.15
CdO	0	0.08	0.08
Ce_2O_3	0	0.01	0.01
CoO	0	0.21	0
Cr_2O_3	0.25	0	0.21
CuO	0	0.05	0.05
Fe_2O_3	1.55	1.43	1.41
Gd_2O_3	0	0.03	0.03
K_2O	7.92	7.62	7.53
MgO	0.05	0.4	0.39
MnO	0.78	0.82	0.81
MoO_3	0.13	0.02	0.02
Na_2O	50.05	52.54	51.91
NiO	0.55	0.09	0.09
P_2O_5	1.19	0.8	0.79
PbO	0.31	0.24	0.24
RuO_2	0.04	0.01	0.01
SO_3	**3.73**	**3.57**	**4.55**
Sb_2O_5	0	0	0.01
SeO_2	0	0	0.01
SiO_2	0.18	0	0
SnO	0.02	0	0
SrO	0	0	0.01
V_2O_5	0	0	0.07
ZnO	0	0.07	0.07
ZrO_2	1	0.01	0.01
Halogens (mass% of non-volatile oxides)			
Cl	1.04	0.88	0.87
F	0.98	0.57	0.73
I	0.02	0.01	0.01
Volatiles (moles/L)			
H^+	1.94	1.08	1.01
NO_3^-	6.96	5.11	5.27
Oxide and Halogen Loading (g/L)			
Solids	145.26	114.55	122.62

3.0 Glass Property/Composition Constraints

With the goal of developing an acceptable glass to demonstrate the direct vitrification of INEEL SBW, we must first establish a definition of an acceptable glass. Two types of glass-property limitations must be considered: 1) those product properties required for waste-form acceptance and 2) those processing properties required to allow for adequate melter processability. The product-property requirements for acceptance in the federal repository are dictated by the Waste Acceptance Product Specification (WAPS) (DOE 1996). The WAPS imposes limitations on the response of glass to the product consistency test (PCT) (ASTM 1998) and requires that chemical and phase-stability information be reported. The specific limit set on the PCT response is that the releases of boron, sodium, and lithium, normalized to glass composition, must be significantly less than those of the DWPF Environmental Assessment (EA) glass. The normalized releases of boron (r_B), sodium (r_{Na}), and lithium (r_{Li}) for the DWPF-EA glass are 8.35 g/m^2, 6.67 g/m^2, and 4.78 g/m^2, respectively (Jantzen et al. 1993). For the purposes of this study, we took a conservative upper release limit of 1 g/m^2 for r_B, r_{Na}, and r_{Li} to account for uncertainty in the PCT measurement and/or glass composition in the proposed INEEL vitrification plant. This limit is consistent with that used by Vienna et al. (1999) and Peeler et al. (2001).

However, glasses with predicted r_B, r_{Na}, or r_{Li} higher than 1 g/m^2 were also tested in this study to obtain information on the compositional effects that may help to increase sulfate incorporation in glass (see Section 4.0 for property predictions basis). There currently are no set acceptance criteria for remote-handled transuranic waste (RH-TRU) at WIPP. We have assumed that glasses capable of meeting these conservative PCT response requirements will also meet the eventual acceptance criteria for RH-TRU. An additional product-property-related restriction placed on glass compositions in this study relates to the formation of secondary phases during cooling, which may detract from the durability of glass. Specifically, glasses formed from wastes high in Na and Al are susceptible to nepheline crystallization during cooling, which has been shown to increase the normalized releases of some glasses subjected to the PCT (Li et al. 1997; Li et al. 1998). Li et al. (1997) showed that glasses with $Na_2O \cdot Al_2O_3 \cdot SiO_2$ sub-mixtures within the nepheline primary phase field in that ternary mixture are susceptible to nepheline formation. For practical purposes, glasses with $[SiO_2]/([Na_2O] + [Al_2O_3] + [SiO_2]) \geq 0.62$ are less susceptible to nepheline formation.

The processing-related properties used to develop the glass formulation include those assumed to be pertinent to processing in a liquid-fed, Joule-heated, ceramic-lined melter (JHCM) and in a cold-crucible induction melter (CCIM). Both of these technologies are considered viable options for treating SBW. Generally, the processing criteria for either technology option depend on the plant design, which in turn depends on the glass properties. In addition, the processing criteria for the two technology options may not overlap. Since the melter technology and hence processing-related properties have not been selected and/or are not clearly defined, we will estimate them from operating plants with similar technologies and compare them.

We let the HLW vitrification experience at the DWPF and WVDP be our guide for processing constraints for the JHCM. For these HLW vitrification plants, the nominal melter operating temperature is maintained at or close to 1150°C. The viscosity (η) at the operating temperature is maintained between 2 and 10 Pa·s. Finally, the liquidus temperature (T_L) of glass in the melter is maintained at least 100°C below the operating temperature (i.e., T_L nominally $\leq$1050°C). The electrical conductivity of the melt was restricted to between 10 and 100 S/m at a nominal 100 Hz and at melt temperature. Two additional processing concerns related to corrosion and sulfur partitioning were used to guide glass-formulation efforts. The accumulation of a salt layer on the melter surface was deemed unacceptable for this study. However, it should be mentioned that relying solely on the sole use of laboratory-scale tests to predict the accumulation of a salt layer during actual melter processing adds risk. In addition, it is assumed, for the purposes of glass formulation, that any sulfur partitioned to the off-gas system would be recycled back to the melter. No specific constraint was used to avoid melts that are excessively corrosive to melter construction material other than the lower η constraint.

We let the waste-vitrification experiences in France and Russia be our guide for processing constraints for the CCIM. For this melter technology, there is not a nominal melter operating temperature limit as the glass contact material is not susceptible to corrosion. However, at high temperatures, the volatility from the glass melt can be excessive for high-alkali waste glasses, and at excessively low temperatures, the feed-to-glass process rates are low. Based on the process temperatures used in waste immobilization by CCIM, we will consider the acceptable processing range to be between 1100 and 1400°C and determine if this criterion is restrictive (if necessary, changes will be made). The process viscosity can be easily adjusted through temperature by altering the melter power input. The nominal operating viscosity was assumed to be between 5 and 10 Pa·s. The electrical conductivity of glass was restricted to between 10 and 100 S/m at the nominal melter operating temperature and a frequency >1 kHz.(a) With a bottom drain configuration, operated in semi-batch mode, the CCIM is expected to tolerate a larger fraction of solid phase than in the standard DWPF- or WVDP-designed JHCMs. Therefore, in place of a T_L restriction, a volume fraction of crystalline phase in equilibrium with the melt will be restricted to ≤3 vol%. As with the JHCM constraints, the accumulation of a salt layer on the melter surface was deemed unacceptable for this study. As previously mentioned, using only laboratory-scale tests to predict the accumulation of a salt layer during actual melter processing adds risk. In addition, it is assumed for the purposes of glass formulation that any sulfur lost to the off-gas system would be recycled back to the melter. No specific constraints on the power absorption or the related properties of skull stability were used.

Table 3.1 summarizes the glass-property and composition constraints used to develop a glass to demonstrate the SBW (WM-180) vitrification flowsheet. Although these constraints define the criteria for glass acceptability, glasses outside these constraints were fabricated and tested to better understand the process of salt segregation from high-sulfur waste-glass melts and the impacts of compositional variation on sulfur partitioning and other glass properties of interest.

As discussed earlier, the processing constraints for the JHCM and CCIM are not identical. Our objective is to develop a glass that will be useful for demonstrating the processing of SBW vitrification by either technology. The formulation work will be performed toward a single set of constraints that will likely yield a non-ideal, but acceptable, glass for both technologies. There is an overlap in the T_M constraint between the two technologies; the T_M constraint used in formulation is $T_M \leq 1150°C$. This value represents what is thought to be nearly ideal for the JHCM with Inconel® electrodes. However, for high-temperature melters such as CCIM, the ideal T_M is significantly higher. There is also an overlap in η_M constraints between 5 and 10 Pa·s. This overlapping range is used as the constraint. An η_M between 3 and 5 Pa·s is thought to be ideal for the WVDP- and DWPF-type JHCMs. Initial formulation work found

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(a) The electrical conductivity is assumed to be independent of frequency at frequencies ≥1 kHz.

that the T_L constraint and quenched glass homogeneity did not limit the loading of SBW or greatly affect the composition of the resulting glass. Therefore, the more restrictive of these constraints (both for JHCM) were retained for the purposes of formulation. All other constraints were the same.

Table 3.1. Constraints Used in SBW Glass Formulation

Property	**JHCM**	**CCIM**
Nominal operating temperature (T_M)	$T_M \leq 1150°C$	$1100 \leq T_M \leq 1400$
Viscosity at T_M (η_M)	$2 < \eta_M < 10$ Pa·s	$5 < \eta_M < 10$ Pa·s
Electrical conductivity at T_M (ε_M)	$10 < \varepsilon_M < 100$ S/m	$10 < \varepsilon_M < 100$ S/m
Liquidus temperature (T_L)	$T_L \leq 1050°C$	--
$[SiO_2]/([SiO_2]+[Na_2O]+[Al_2O_3])$	≥ 0.62	≥ 0.62
Normalized PCT boron release (r_B) (both quenched and CCC samples)	$r_B < 1$ g/m^2	$r_B < 1$ g/m^2
Normalized PCT sodium release (r_{Na}) (both quenched and CCC samples)	$r_{Na} < 1$ g/m^2	$r_{Na} < 1$ g/m^2
Normalized PCT lithium release (r_{Li}) (both quenched and CCC samples)	$r_{Li} < 1$ g/m^2	$r_{Li} < 1$ g/m^2
Salt Layer Accumulation	Unacceptable	Unacceptable
Quenched Glass Homogeneity (H_Q)	Homogeneous$^{(a)}$	≤3 vol.%
CCC Glass Homogeneity (H_C)	<5 vol.% secondary phase	<5 vol.% secondary phase

(a) Homogeneity was judged by the lack of second-phase identification by X-ray diffraction (XRD) and by optical microscopy at magnifications of ≥100×.

4.0 Functions to Relate Glass Properties to Composition

Glass compositions for immobilizing SBW waste can be formulated based on models that relate glass properties to composition. This approach was successfully used by Vienna et al. (1999) in the development of SBW-1 and by Peeler et al. (2001) in the development of SBW-9; thus, it provides the starting point for glass-formulation activities using the latest WM-180 composition and constraints. To calculate a glass composition for SBW waste that meets the criteria listed in Table 3.1, we first must describe the glass properties as functions of composition or waste and additive concentrations. For the purposes of these scoping tests, selected glass properties were empirically modeled as linear functions of composition:

$$P = \sum_{i=1}^{n} a_i x_i \tag{4.1}$$

where

P = transformed property
a_i = i^{th} component coefficient
x_i = i^{th} component mole fraction in glass
n = number of components in glass (thus Σx_i=1).[a]

Here, the normalized mole fraction of the n components should be used for x_i. This corresponds to the assumption that the glass properties depend only on the relative proportions (mole fractions) of the n components that have a_i values. Table 4.1 lists the a_i values used to model glass properties in this study, which are from Hrma et al. (2001). The viscosity model used the Arrhenius form

$$\ln(\eta) = A + \frac{B}{T} \tag{4.2}$$

where A and B are constants, and T is the absolute temperature. The constants A and B are modeled using Equation (4.1), and viscosity is calculated as a function of temperature from Equation (4.2).

The first-order approximations for the properties listed in Table 4.1 provide a good foundation for initial glass-formulation efforts. However, two significant shortcomings must be pointed out: 1) the use of these approximations is recommended only within the composition region covered by the experimental data used to fit component coefficients (i.e., the a_i values) and 2) no data set sufficient to model T_L, H_Q, H_C, melter corrosion, or salt formation related properties was found for this specific composition range. Experimental data on salt formation, T_L, H_Q, and H_C were generated on glasses with systematically varied composition that meet or exceed the requirements for those properties for which models exist.

(a) See Hrma et al. (1994) for details on this type of property model.

Table 4.1. Property-Composition Coefficients (*a_i* values)

Oxide	Viscosity, η(Pa·s)		Normalized Release, r_i in g/m^2		
	A$^{(a)}$	B (K)$^{(a)}$	ln(r_B)	ln(r_{Li})	ln(r_{Na})
Al_2O_3	-2.860	27599	-32.132	-29.331	-32.858
B_2O_3	-13.594	8765	14.509	11.958	9.957
CaO	-25.804	27511	-12.976	-9.121	-4.018
Fe_2O_3	-3.490	-835	-6.74	-9.463	-9.11
K_2O	-16.589	14436	-10.312	-6.515	0.323
Li_2O	-7.100	-10377	9.558	9.507	7.328
MgO	-19.102	25120	0.399	-0.484	2.575
MnO_x			-18.462	-14.389	-16.326
Na_2O	-9.974	632	16.821	13.193	18.873
P_2O_5			-36.1	-28.686	-36.605
SiO_2	-10.136	26427	-4.41	-3.605	-4.278
ZrO_2	-55.621	95153	-14.976	-13.177	-18.384
$Ln_2O_3$$^{(e)}$	43.460	-78677			
F	-83.850	108852			

(a) A and B are η parameters

5.0 Additive Compositions Formulation

This section describes previous efforts at formulating additive compositions as well as current studies. Seventeen additive compositions were fabricated for the current effort.

5.1 Previous Efforts

Ten additive compositions were previously developed for SBW vitrification (Vienna et al. 1999; Peeler et al. 2001). The compositions for these additive compositions, SBW-1 to -10, reported earlier, are listed in Table 5.1. SBW-1 was developed to demonstrate 1998 SBW simulant vitrification in the Envitco EV-16 melter at Clemson Environmental Technologies Laboratory (CETL) (Vienna et al. 1999). The objectives for this formulation were to develop a glass with acceptable process and product-quality–related properties without any salt-formation restrictions. The composition of 65 mass% of the SBW-1 additives and 35% of the 1998 SBW simulant was successfully processed in the EV-16 without any detectable salt formation. However, due to a misbatch in the melter feed, the nominal target SO_3 in the feed was only 1.07 mass% rather than the designed value of 1.35 mass%. These results are summarized by Darab et al. (2001) and again by Vienna et al. (2002).

Table 5.1. Compositions and Results from Previously Developed SBW Additive Mixes (in mass% Oxides)

	SBW-1	SBW-2	SBW-3	SBW-4	SBW-5	SBW-6	SBW-7	SBW-8	SBW-9	SBW-10
B_2O_3	14.26	12.00	15.00	12.00	15.00	15.00	10.00	15.00	15.00	12.00
BaO	---	---	---	---	---	---	---	---	---	2.00
CaO	---	---	---	2.00	4.00	2.00	4.00	2.00	5.00	---
Fe_2O_3	11.31	11.31	11.31	12.00	12.00	12.00	15.00	15.00	10.00	12.00
Li_2O	2.67	4.00	4.50	4.00	4.00	4.00	5.00	5.00	5.00	4.00
SiO_2	68.69	69.61	66.11	70.00	65.00	67.00	66.00	63.00	65.00	70.00
TiO_2	3.08	3.08	3.08	---	---	---	---	---	---	---

The formulation for SBW-2 was an adjustment of SBW-1 to allow for the development of an acceptable glass with 2000 WM-180 concentrations between 22 and 35 mass% (equivalent to 0.8 to 1.25 mass% SO_3 in the glass). The concentrations of B_2O_3, BaO, CaO, Fe_2O_3, Li_2O, SiO_2, and TiO_2 were altered from those of SBW-2 to determine their effects on salt formation and crystallization of the glass—resulting in SBW-3 through -10. Of the glasses formed from different ratios of SBW-2 through -10 and 2000 WM-180, it was found that SBW-9 could tolerate the highest SBW loading without the formation of a molten salt in ramp-heated crucible tests. These results are described in Peeler et al. (2001). SBW-9 was processed with between 30 and 35 mass% of 2000 WM-180 simulant in the research-scale melter (RSM) at PNNL (Goles et al. 2001). Salt accumulation was seen for WLs greater than 32 mass% (1.14 mass% SO_3). SBW-9-30 composition (SBW-9 additives, 30 mass% of 2000 WM-180 simulant) was processed in the EV-16 at CETL (Perry et al. 2001). Salt was found to accumulate in the melter during this test. The cause of salt accumulation was determined to be a misbatch—the as-batched SO_3 concentration was the equivalent of 1.35 mass%, significantly higher than the target value of 1.07 mass%.

5.2 Current Studies

Using the constraints discussed in Section 3.0, the 2001 WM-180 waste-composition estimate in Table 2.1 and the property models listed in Section 4.0, additive and glass compositions were developed. Table 5.2 summarizes the additive compositions that were tested in this study.

SBW-11 was developed to be combined with 18.5 mass% of 2001 WM-180 simulant. At that loading, the glass would contain 0.84 mass% of SO_3 (assuming full retention). This WL was selected based on preliminary results[(a)] from previous melter tests using SBW-9 (Goles et al. 2001; Perry et al. 2001). The preliminary analyses of the sulfur mass balance of these tests showed a minimum fraction of sulfur to the off-gas of 29% and a minimum SO_3 loading in the glass of 0.59 mass%, which, when combined, suggested that the minimum allowable SO_3 loading would be 0.84 mass%. At the time SBW-11 was being developed, the acceptable range of viscosity was given by $2 \leq \eta_{1150} \leq 10$ Pa·s, since CCIM processing was not yet being considered. The composition of SBW-11-18.5[(b)] is summarized in Table 5.3. The additives were selected to optimize for sulfur retention in the glass melt while maintaining WL, η, ε, r_B, r_{Na}, and r_{Li} within acceptable ranges.

Previous studies suggested that high concentrations of alkali and alkali-earth elements would improve the solubility of SO_3 in glass (Li et al. 2001; Schreiber et al. 2000, for example). This is shown to increase sulfur retention as illustrated by the comparison of SBW-4 and -9 (Peeler et al. 2001). In the latter, higher concentrations of CaO and Li_2O led to higher sulfur retention and higher sulfur loadings before salt was formed in crucible tests. It was speculated at the time of this formulation that combinations of alkali and alkali-earth would be more effective at raising sulfur retention and avoiding salt formation than would any single component. This theme is described in more detail in Vienna et al. (2002). This led to consideration of CaO, MgO, Na_2O, and Li_2O as additive components. The addition of V_2O_5 in SBW-11 was to improve sulfur retention and help avoid salt accumulation, given that many researchers (Stefanovsky and Lifanov 1989, for example) have reported higher sulfur loadings in alkali-borosilicate glass melts by adding V_2O_5. Additions of between 10 and 20 mass% V_2O_5 were found to improve sulfur loading in glass to roughly 8% (as mass% SO_3). However, detailed analyses of the high-sulfur, high-vanadium glasses showed phase separation, the scale of which increased with increasing V_2O_5 concentration (Stefanovsky and Lifanov 1989). A smaller concentration of V_2O_5 was added to the SBW-11-18.5 glass (4 mass%) to avoid the immiscible phase separation while taking advantage of the capability of the melt to obtain higher SO_3 loadings without salt accumulation. Sufficient Fe_2O_3 was added to the SBW-11-18.5 glass to assure that iron redox of the resulting glass could be precisely determined. This was necessary to allow for the tailoring of the reductant concentration in the feed. The amounts of ZrO_2, B_2O_3, and SiO_2 were adjusted to obtain adequate η_{1150}, ε_{1150}, r_B, r_{Na}, and r_{Li}.

(a) The preliminary mass balances, reported by Darab et al. 2001, were used for the estimate. Final sulfur mass balances, reported by Perry et al. (2001) and Goles et al. (2001), were not available at the time of initial formulation work.

(b) Throughout the report, the nomenclature for glass compositions is as follows: frit composition, "-", loading of 2001 WM-180 simulant in mass%. For example, SBW-11-30 refers to Frit SBW-11 at 30% waste loading.

Table 5.2. Additive Compositions Tested in This Study

Oxide	SBW-11	SBW-12	SBW-13	SBW-14	SBW-15	SBW-16	SBW-17	SBW-18	SBW-19	SBW-20
B_2O_3	12.15	6.03	12.15	14.45	7.00	14.00	12.00	12.00	12.00	12.00
CaO	5.02	5.02	-	5.02	8.00	7.00	2.00	2.00	2.00	4.00
Fe_2O_3	1.52	1.52	1.52	1.52	8.00	---	12.00	12.00	12.00	12.00
K_2O	---	---	8.15	---	---	---	---	---	---	---
Li_2O	6.11	6.11	6.11	6.11	6.00	6.00	4.00	3.00	3.00	3.00
MgO	1.75	1.75	---	1.75	---	---	---	---	---	---
Na_2O	1.90	6.98	1.90	1.90	7.00	2.00	---	4.00	4.00	4.00
SiO_2	64.23	65.27	62.85	66.81	64.00	71.00	70.00	64.00	62.00	60.00
V_2O_5	4.88	4.88	4.88	---	---	---	---	2.00	4.00	4.00
ZrO_2	2.44	2.44	2.44	2.44	---	---	---	1.00	1.00	1.00
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00

Oxide	SBW-21	SBW-22	SBW-23	SBW-24	SBW-25	SBW-26	SBW-27
B_2O_3	10.00	6.03	12.15	11.10	12.15	6.03	6.03
CaO	4.00	5.02	---	5.02	5.02	14.19	11.75
Fe_2O_3	12.00	1.52	1.52	1.52	12.00	1.52	1.52
K_2O	---	---	4.32	---	---	---	---
Li_2O	3.00	6.11	6.11	6.11	3.44	3.52	4.08
MgO	---	1.75	---	1.75	1.75	1.75	1.75
Na_2O	4.00	4.29	1.90	1.90	---	1.90	1.90
SiO_2	62.00	67.95	66.68	70.16	58.32	63.77	63.21
V_2O_5	4.00	4.88	4.88	---	4.88	4.88	7.32
ZrO_2	1.00	2.44	2.44	2.44	2.44	2.44	2.44
Sum	100.00	100.00	100.00	100.00	100.00	100.00	100.00

Table 5.3. SBW-11-18.5 Glass Composition (mass% oxides)

Oxide	Glass	Oxide	Glass
Al_2O_3	5.09	Na_2O	11.15
B_2O_3	9.97	**SO_3**	**0.84**
CaO	4.49	SiO_2	52.35
Fe_2O_3	1.50	V_2O_5	3.99
K_2O	1.39	ZrO_2	1.99
Li_2O	4.98	Others(a)	0.76
MgO	1.50		

(a) Others composition can be obtained from the composition of 2001 WM-180 simulant in Table 2.1.

SBW-12 to SBW-14 were formulated to investigate the effects of increasing the glass basicity number (Krämer 1991), removing CaO and MgO, and removing V_2O_5 from SBW-11 on the sulfur retention in glass and salt formation. The calculated 1150ºC viscosity of glasses with 18.5 mass% WL was kept constant at the same value as in SBW-11-18.5 glass. In SBW-12, the B_2O_3 was decreased and the Na_2O was increased from SBW-11, which is the general direction for the increase of basicity. The concentration of B_2O_3 in a glass with 18.5 mass% SBW-12 was decreased to 5 mass%, which is considered a lower boundary in typical "borosilicate" glasses (and the lower boundary of glass-property–model validity). Then, the concentration of Na_2O was increased to have the calculated viscosity at the target value. In SBW-13, CaO and MgO were removed from SBW-11, and K_2O was increased until the predicted viscosity of a glass with 18.5 mass% SBW-13 reached the target value. Among alkali components that can be used to adjust the viscosity, Na_2O and Li_2O were not used because of their tendency to increase PCT releases. In SBW-14, V_2O_5 was removed from SBW-11, and B_2O_3 was increased until the predicted viscosity of a glass with 18.5 mass% SBW-14 reached the target value.

Regarding the basicity number used in formulating SBW-12, it is well known that the equilibrium solubility of sulfate increases as the glass basicity increases (Krämer 1991). The incorporation of sulfate in glass is not directly proportional to the equilibrium sulfate solubility because the kinetic factors are also involved in sulfate incorporation and may be more important than equilibrium. However, the effect of increasing basicity on sulfate incorporation was tested in this study to investigate the possible beneficial effect of increased sulfate solubility on sulfate incorporation.

As previously noted, SBW-12 to SBW-14 were formulated to evaluate the effect of basicity on sulfur retention while maintaining constant viscosity. Parallel with that effort was a series of scoping studies to evaluate the impacts of CaO, MgO, Na_2O, Li_2O, and V_2O_5 additions on sulfur solubility without constraining predicted properties to acceptable levels. The intent of this series of glasses was to vary the potential additive-composition region over a relatively broad range to gain some insight into the impacts of sulfur solubility as well as other glass properties of interest. SBW-15 to SBW-21 were also developed using an 18.5 mass% WL basis.

Additives up to SBW-21 were formulated assuming that the resulting glasses will be processed in the JHCM. While this work was still in progress, the possibility of using the CCIM emerged, and thus the objectives of the development work shifted to the possible processing of a single glass using both CCIM and JHCM. Therefore, the calculated η_{1150} was increased to the range appropriate for both melter technologies ($5 \leq \eta_{1150} \leq 10$ Pa·s, with a target near 7 Pa·s) in SBW-22 to SBW-27 based glasses.

SBW-22 to SBW-24 used the same guidelines as used in SBW-12 to SBW-14, respectively, except that the target viscosities changed to near 7 Pa·s. SBW-25 was formulated with increased Fe_2O_3 from SBW-11 to explore the effect of Fe_2O_3 on the sulfur behavior. To compensate for the decrease of viscosity caused due to the increase of Fe_2O_3 concentration, Na_2O was removed, and Li_2O was decreased until the glass with 18.5 mass% SBW-25 satisfied the viscosity target.

SBW-26 and SBW-27 were formulated to further investigate the effect of CaO and V_2O_5 on the sulfate incorporation. In SBW-26, CaO was increased from 4.5 to 12 mass% CaO in a glass with 18.5 mass% WL. The decrease in viscosity due to the increase of CaO was compensated by decreasing B_2O_3 and Li_2O at a similar proportion. In SBW-27, both CaO and V_2O_5 were increased, and the viscosity was compensated by also decreasing B_2O_3 and Li_2O at a similar proportion.

Table 5.4 summarizes the predicted viscosity and PCT normalized releases for the glasses with 18.5 mass% WL and SBW-11 to SBW-27 additive compositions. The effect of WL on sulfur behavior and glass properties was tested on glasses with selected additive compositions. These additive compositions were also used in glasses with varying WL (typically 15, 20, 25, and 30 mass% 2001 WM-180 simulant). Table 5.5 summarizes the calculated properties of these glasses. The testing results for all these glasses are given in Sections 6.0 and 7.0.

Table 5.4. Predicted Viscosity (η_{1150} in Pa·s) and r_i Values (in g/m^2) for Glasses Formulated with 18.5 mass% 2001 WM-180 Simulant Using a_i Values from Table 4.1

Glass	From Hrma et al. (2001)			
SBW-xx-18.5	η_{1150}	r_B	r_{Na}	r_{Li}
11	4.6	0.8	1.0	0.9
12	4.6	0.8	1.4	0.9
13	4.6	1.0	1.2	1.1
14	4.6	1.0	1.1	1.0
15	2.9	0.7	1.3	0.7
16	5.3	0.7	1.0	0.9
17	14.6	0.5	0.4	0.5
18	9.8	0.8	0.8	0.7
19	8.7	0.9	0.9	0.7
20	6.5	0.7	0.9	0.6
21	8.6	0.5	0.7	0.5
22	7.0	0.5	0.8	0.6
23	6.9	1.1	1.0	1.1
24	7.0	0.6	0.8	0.7
25	6.9	0.4	0.5	0.4
26	7.1	0.1	0.3	0.2
27	7.1	0.1	0.4	0.2

Table 5.5. Predicted Viscosity (η_{1150} in Pa·s) and r_i Values (in g/m^2) for Glasses Formulated with Various Loadings of 2001 WM-180 Simulant Using a_i Values from Table 4.1 and for Comparison, the a_i Values from Hrma et al. (1994)

	From Hrma et al. (2001)			
Glass	η_{1150}	r_B	r_{Na}	r_{Li}
SBW-11-15	5.6	0.7	0.8	0.9
SBW-11-18.5	4.6	0.8	1.0	0.9
SBW-11-20	4.3	0.9	1.1	0.9
SBW-11-25	3.3	1.0	1.4	1.0
SBW-11-30	2.5	1.2	1.8	1.1
SBW-22-15	8.4	0.5	0.7	0.6
SBW-22-18.5	7.0	0.5	0.8	0.6
SBW-22-20	6.4	0.5	0.9	0.6
SBW-22-25	4.8	0.7	1.1	0.7
SBW-22-30	3.6	0.8	1.5	0.8
SBW-23-15	8.5	1.0	0.9	1.1
SBW-23-18.5	6.9	1.1	1.0	1.1
SBW-23-20	6.4	1.1	1.1	1.1
SBW-23-25	4.8	1.3	1.5	1.2
SBW-23-30	3.6	1.5	1.9	1.3
SBW-24-15	8.4	0.5	0.6	0.7
SBW-24-18.5	7.0	0.6	0.8	0.7
SBW-24-20	6.4	0.6	0.8	0.7
SBW-24-25	4.9	0.7	1.1	0.8
SBW-24-30	3.7	0.9	1.4	0.9
SBW-25-15	8.6	0.3	0.4	0.4
SBW-25-18.5	6.9	0.4	0.5	0.4
SBW-25-20	6.3	0.4	0.5	0.4
SBW-25-25	4.7	0.5	0.7	0.5
SBW-25-30	3.4	0.6	0.9	0.5

6.0 Batch-Chemical Testing

A series of crucible tests was performed using reagent-grade oxides, carbonates, and boric acid before the WM-180 liquid simulant was prepared. Glasses produced in this series were coupled with the 2001 SBW simulant composition (see Table 2.1) with the candidate additive compositions (see Table 5.2). This section discusses the experimental procedures and results of these preliminary or scoping tests using batch chemicals as the SBW simulant. The objective of these tests was to assess various additives components and their effects on sulfate solubility and glass properties of interest.

Based on results of previous studies with SBW, using only laboratory-scale tests to predict the accumulation of a salt layer during actual melter processing adds risk. More specifically, differences in sulfur partitioning have been observed between laboratory-scale testing and melter demonstrations. Not only can there be an effect of testing scale on sulfur partitioning, but the use of reagent-grade chemicals and the liquid-feed tests can also produce differences as well. Although differences can exist, the use of laboratory-scale tests has been effective in the past to down-select potential additive composition candidates that will be carried forward in the testing methodology established as one transitions from the dry-fed, small-scale laboratory tests to slurry-fed melter demonstrations. The crucible-scale tests only lower the risk of salt formation and/or accumulation in the melter; they do not eliminate it.

6.1 Experimental

Each batch was prepared from the proper proportions of reagent-grade metal oxides, carbonates, H_3BO_3, and salts in 150-g batches using standard batching and melting procedures. Batch sheets were filled out as the materials were weighed. Once batched, the glasses were melted using a standard thermal heat treatment. In general, the raw materials were thoroughly mixed and placed into a 95%Pt/5%Au 250-mL crucible. The batch was subsequently placed into a high-temperature furnace, and the temperature was increased at ~8°C/min until the target melt temperature (1150°C) was reached. After an isothermal hold at 1150°C for 1.0 h, the crucible was removed, and the glass was poured onto a clean stainless steel plate and allowed to air cool. Visual observations on the resulting pour patty and residual crucible glass were documented.

The pour patty and residual crucible glass were ground, and the crushed glass was subsequently transferred to its original 95%Pt/5%Au 250-mL crucible for a second melt at 1150°C. After an isothermal hold at 1150°C for 1.0 h, the crucible was removed, and the glass was poured onto a clean stainless steel plate and allowed to air cool. Visual observations on the resulting pour patty and residual crucible glass were documented. Approximately 140 g of glass were removed (poured) from the crucible while ~10 g remained in the crucible along the walls. The pour patty was used as a sampling stock for the various heat treatments and property measurements (i.e., chemical composition, crystallinity, viscosity, and/or durability).

To bound the effects of thermal history on the product performance, approximately 25 g of each glass was heat treated to simulate cooling along the centerline of a DWPF-type canister (Marra and Jantzen 1993). The temperature schedule used to simulate canister centerline cooling (CCC) is listed in Table 6.1. This terminology will be used in this report to differentiate samples from different cooling schedules (quenched versus CCC).

Table 6.1. Centerline Canister Cooling Schedule Utilized

Step	Ramp (°C/min)	Target Temperature (°C)	Dwell (h)
1	10	1150	4
2	8	926	0.1
3	1	779	2.8
4	1	715	3.4
5	1	598	4.2
6	1	490	4.3
7	1	382	7.4
8	1	70	End

To confirm that the "as-fabricated" (or quenched) glasses corresponded to the defined target compositions, a representative sample from each glass pour patty was analyzed. Concentrations (as mass%) for the cations of interest were measured by inductively coupled plasma-atomic emission spectroscopy (ICP-AES). To assess the performance of the ICP over the course of these analyses, glass standards were intermittently analyzed.

Homogeneity was evaluated (visual and optical microscopy [minimum of 100× magnification]) and documented for each SBW glass (both quenched and CCC samples). Homogeneity in this context refers to the presence of crystallization and/or a salt layer, not the presence of glass-in-glass phase separation (amorphous phase separation).

Roughly 5-g samples of select as-fabricated (quenched) SBW glasses were heat treated at 1050°C for 24 h in a Pt/Rh crucible with a tight-fitting lid. Visual observations and optical microscopy analyses were made to assess the presence of crystallization or droplets of Na_2SO_4 (as observed by Vienna et al. 1999). Note that only a single isothermal heat treatment was performed to assess the 1050°C acceptance criterion. The T_L was not formally determined—only an assessment of T_L with respect to the acceptance criterion.

High-temperature η was measured as a function of temperature (T) using a spindle viscometer for selected SBW glasses. The measurements were obtained using standard procedures (SRTC 1999; Schumacher and Peeler 1998), which are compliant with ASTM C 965-81 (ASTM 1990). High-temperature η data were measured over the maximum temperature range allowable for each glass. The low-temperature limit was based on the effects of crystallization on the melt pool. The high-temperature limit was based on reducing the effects of volatilization. To validate the glass-η data, the η of the Batch 1 standard glass (Schumacher and Peeler 1998) was measured intermittently during the viscosity determinations. Viscosity at 1150°C (η1150°C) for each glass was calculated from a Vogel-Tamman-Fulcher fit of the measured data.

Electrical conductivity (ε) was measured as a function of both temperature and frequency using an apposing plate probe as described previously (Hrma et al. 1994).

The PCT was performed on each glass to assess chemical durability (ASTM 1998). The PCT was conducted in triplicate for each SBW glass (both quenched and CCC samples). Also included in this experimental test matrix were the EA glass (Jantzen et al. 1993), the Approved Reference Material

(ARM-1) glass, and blanks. Samples were ground, washed, and prepared according to procedure. Fifteen mL of Type I ASTM water was added to 1.5 g of glass in stainless steel vessels. The vessels were closed, sealed, and placed in an oven at 90 ± 2°C. Samples were left at 90°C ± 2°C for 7 days. The resulting solutions (once cooled) were sampled (filtered and acidified), labeled, and analyzed. Normalized release rates were calculated based on targeted compositions using the average of the logs of the leachate concentrations.

6.2 Results

6.2.1 Chemical Composition

Appendix A summarizes the target and measured compositions for all the SBW glasses. Overall, comparisons between the measured and targeted compositions suggest that there were no significant problems in the batching or fabrication of the study glasses.

6.2.2 Homogeneity

Table 6.2 summarizes the visual observations of homogeneity for each SBW glass as a function of WL. Observations after the initial and second melts at 1150°C and following the CCC heat treatment are shown. Appendix B provides a more detailed description of each glass. Again, it should be noted that these glasses were produced using reagent-grade oxides, carbonates, and boric acid (not using the liquid simulant). This evaluation provides an indication of the potential advantages of the additive-composition changes in terms of SO_3 retention in glass.

It is interesting to note that no sign of salt was observed after the second melt for those glasses in which a salt layer was observed on the surface of the melt (or above the melt line) after the initial melt at 1150°C. No effort was made to remove the salt after the initial melt. In fact, a special effort was made to transfer as much of the salt layer as possible to the second melt—where this became a real problem was the observation of the yellow salt plated on the crucible wall above the melt line. Before the second melt, the glasses with SO_3 layers (where applicable) were ground to a fine powder. The lack of salt layers in the second melts may be a function of the melting procedure, which may suggest that the SO_3 solubility limit had not been exceeded. To confirm this, a comparison of the target and measured SO_3 concentrations in glass should be reviewed (see Table 6.3).

As previously mentioned, the results of earlier studies with SBW indicate that the sole use of crucible tests to predict the accumulation of a salt layer during actual melter processing adds risk. More specifically, differences in sulfur behavior have been observed between crucible testing and melter demonstrations.[a] The crucible tests only lower the risk of salt formation and/or accumulation in the melter; they do not eliminate it.

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[a] Typically, salt formation occurs in crucible melts made from oxide and carbonate raw materials at lower sulfur concentrations than larger scale tests using simulants.

Table 6.2. Visual Observations of Homogeneity for Quenched and CCC SBW Glasses

Glass	WL	Initial Melt	Second Melt	CCC
SBW-11	18.5	Homogeneous	Homogeneous	Homogeneous
	30	Homogeneous	Homogeneous	Homogeneous
SBW-12	18.5	Homogeneous	Homogeneous	Homogeneous
SBW-13	18.5	Homogeneous	Homogeneous	Homogeneous
SBW-14	18.5	Homogeneous	Homogeneous	Homogeneous
SBW-15	18.5	Homogeneous	Homogeneous	Homogeneous
SBW-16	18.5	Homogeneous	Homogeneous	Homogeneous
SBW-17	18.5	Salt	Homogeneous	Homogeneous
SBW-18	18.5	Salt	Homogeneous	Homogeneous
SBW-19	18.5	Salt	Homogeneous	Homogeneous
SBW-20	18.5	Salt	Homogeneous	Homogeneous
SBW-21	18.5	Salt	Homogeneous	Homogeneous
SBW-22	15	Homogeneous	Homogeneous	Homogeneous
	18.5	Homogeneous	Homogeneous	Homogeneous
	20	Homogeneous	Homogeneous	Homogeneous
	25	Salt	Homogeneous	Homogeneous
SBW-23	15	Homogeneous	Homogeneous	Homogeneous
	18.5	Homogeneous	Homogeneous	Homogeneous
	20	Homogeneous	Homogeneous	Homogeneous
	25	Homogeneous	Homogeneous	Homogeneous
SBW-24	18.5	Salt	Homogeneous	Homogeneous
SBW-25	15	Homogeneous	Homogeneous	Homogeneous
	18.5	Homogeneous	Homogeneous	Homogeneous
	20	Homogeneous	Homogeneous	Homogeneous
	25	Homogeneous	Homogeneous	Homogeneous
SBW-26	18.5	Homogeneous	Homogeneous	Homogeneous
	25	Salt	Homogeneous	Homogeneous
SBW-27	18.5	Homogeneous	Homogeneous	Homogeneous
	25	Salt	Homogeneous	Homogeneous

Table 6.3. Comparison of Target and Measured SO_3 Concentrations

Glass	WL	SO_3 Target[(a)]	SO_3 Measured[(b)]	Retention % Relative	Salt Layer Observed[(c)]
SBW-11	18.5	0.83	0.749	90	No
	30	NM	NM	---	No
SBW-12	18.5	0.83	0.824	99	No
SBW-13	18.5	0.83	0.819	99	No
SBW-14	18.5	0.83	0.722	87	No
SBW-15	18.5	0.83	0.844	102	No
SBW-16	18.5	0.83	0.737	89	No
SBW-17	18.5	0.83	0.477	57	Yes
SBW-18	18.5	0.83	0.602	73	Yes
SBW-19	18.5	0.83	0.572	69	Yes
SBW-20	18.5	0.83	0.589	71	Yes
SBW-21	18.5	0.83	0.582	70	Yes
SBW-22	15	0.68	0.599	88	No
	18.5	0.83	0.737	89	No
	20	0.91	0.806	89	No
	25	1.14	1.074	94	Yes
SBW-23	15	0.68	0.694	102	No
	18.5	0.83	0.914	110	No
	20	0.91	1.01	111	No
	25	1.14	1.35	118	No
SBW-24	18.5	0.83	NM	---	No
SBW-25	15	0.68	0.654	96	No
	18.5	0.83	0.734	88	No
	20	0.91	0.734	81	No
	25	1.14	1.084	95	Yes
SBW-26	18.5	0.83	0.754	91	No
	25	1.14	1.08	95	Yes
SBW-27	18.5	0.83	0.782	94	No
	25	1.14	1.05	92	Yes

(a) Target SO_3 concentrations are precise to withing ± 1% (relative).
(b) Measured SO_3 concentrations are precise to within ± 10% (relative)
(c) Observations regarding the formation of a salt layer are after the second melt. Refer to Appendix B for more details on observations after both the initial and second melts.

6.2.3 1050°C Isothermal Heat Treatment

Table 6.4 summarizes the visual and optical microscopy results of select glasses. All samples tested were homogeneous, indicating that the T_L is less than 1050°C (meeting one of the processing constraints as listed in Table 3.1). It should be noted that for those glasses in which a salt layer was observed after initial fabrication, the salt layer may not have been observed after the second melt (refer to Appendix B). Therefore, the T_L results of these glasses should be viewed and used accordingly.

Table 6.4. Visual Observations of Homogeneity for Quenched and CCC SBW Glasses

Glass	WL	1050°C Heat Treatment	Glass	WL	1050°C Heat Treatment
SBW-11	18.5	Homogeneous	SBW-23	15	Homogeneous
	30	Homogeneous		18.5	Homogeneous
SBW-12	18.5	Homogeneous		20	Homogeneous
SBW-13	18.5	Homogeneous		25	Homogeneous
SBW-14	18.5	Homogeneous	SBW-24	18.5	Homogeneous
SBW-15	18.5	Homogeneous	SBW-25	15	Homogeneous
SBW-16	18.5	Homogeneous		18.5	Homogeneous
SBW-17	18.5	Homogeneous		20	Homogeneous
SBW-18	18.5	Homogeneous		25	Homogeneous
SBW-19	18.5	Homogeneous	SBW-26	18.5	Homogeneous
SBW-20	18.5	Homogeneous		25	Homogeneous
SBW-21	18.5	Homogeneous	SBW-27	18.5	Homogeneous
SBW-22	15	Homogeneous		25	Homogeneous
	18.5	Homogeneous			
	20	Homogeneous			
	25	Homogeneous			

6.2.4 Viscosity

High-temperature η was measured as a function of temperature (T) using a spindle viscometer for selected SBW glasses. Appendix C summarizes the measured viscosity data for each SBW glass. Table 6.5 summarizes the $\eta_{1150°C}$ for each glass as calculated from a Vogel-Tamman-Fulcher fit of the measured data.

Table 6.5. Calculated Viscosity at 1150°C (η1150°C) for Select Glasses Based on the Vogel-Tamman-Fulcher Fit of the Measured Data

Glass ID	Estimated $\eta_{1150°C}$ (Pa-s)	Glass ID	Estimated $\eta_{1150°C}$ (Pa-s)
SBW-11-18.5	3.90	SBW-22-25	5.14
SBW-15-18.5	3.07	SBW-23-18.5	5.66
SBW-17-18.5	15.76	SBW-25-15	7.94
SBW-20-18.5	5.56	SBW-25-18.5	6.28
SBW-21-18.5	6.86	SBW-25-20	5.67
SBW-22-15	8.18	SBW-25-25	4.28
SBW-22-18.5	6.81	SBW-26-18.5	6.55
SBW-22-20	6.47	SBW-27-18.5	6.32

6.2.5 Electrical Conductivity

The electrical conductivity (ε) of molten SBW-22-20 glass was measured. The ε – T – frequency data are listed in Table 6.6 and plotted in Figure 6.1. At 1-kHz frequency and above, the ε is relatively independent of frequency. At this frequency, the temperature effect on ε can be described by an Arrhenius function:

$$\ln[\varepsilon] = 8.696 - 7028.6/T \tag{6.1}$$

where T is in Kelvin and ε is in S/m. This would yield an ε of 38 S/m at the nominal melter operating temperature of 1150°C.

Table 6.6. Electrical Conductivity Data for SBW-22-20 Glass Melt

T (°C)	ε (S/m)	Freq (Hz)
1243	43.0	100
1144	31.8	100
1044	22.9	100
945	15.3	100
1243	56.9	1000
1144	42.4	1000
1044	29.4	1000
945	18.3	1000
1243	62.0	10000
1144	45.5	10000
1044	30.9	10000
945	18.9	10000

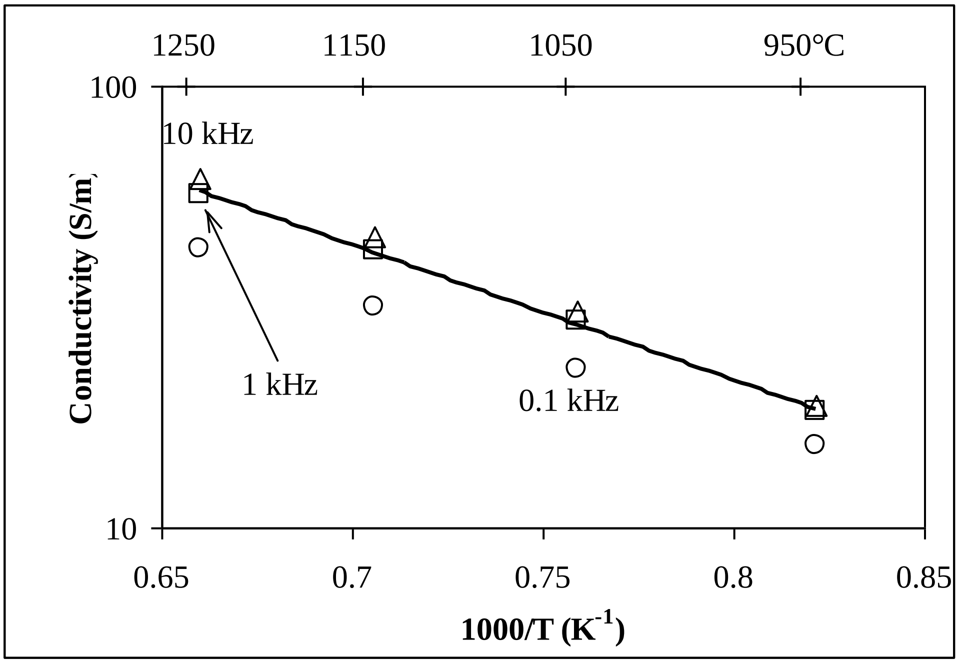

Figure 6.1. Electrical Conductivity of SBW-22-20 Melt as a Function of Inverse Temperature

6.2.6 PCT

Table 6.7 summarizes the PCT results for both quenched and CCC SBW glasses as a function of WL. Note that the PCT was not performed on the SBW-26 and SBW-27 glasses. With respect to the conservative acceptance criteria being used in this study (<1 g/m^2 for each element of interest), the only glasses (based on measurements) that are not acceptable are SBW-11 (18.5% WL), SBW-13 (18.5% WL), SBW-14 (18.5% WL), and SBW-23 (all WLs) glasses. If one were to move this conservative constraint to 1.5 g/m^2, only the SBW-13 and SBW-23 glasses would fail. Based on PCT releases, SBW-13 and SBW-23 are not considered potential candidates, given that they yield unacceptable PCT responses, even though the solubility of SO_3 appears to promising.

Table 6.7. Normalized B, Li, Na, and Si Releases (g/m^2) from PCT of Quenched and CCC SBW Glasses Samples

		Quenched				CCC			
Glass	**WL**	r_B	r_{Li}	r_{Na}	r_{Si}	r_B	r_{Li}	r_{Na}	r_{Si}
SBW-11	18.5	1.48	1.53	1.33	0.51	1.31	1.37	1.21	0.48
SBW-11	30	0.60	0.56	0.72	0.25	0.49	0.51	0.60	0.25
SBW-12	18.5	0.54	0.84	0.91	0.36	0.53	0.80	0.83	0.34
SBW-13	18.5	14.88	13.48	13.34	4.17	14.93	13.47	13.33	4.18
SBW-14	18.5	1.05	1.01	0.95	0.40	0.91	0.90	0.84	0.37
SBW-15	18.5	0.30	0.61	0.69	0.16	0.23	0.55	0.56	0.14
SBW-16	18.5	0.19	0.31	0.29	0.13	0.13	0.25	0.23	0.10
SBW-17	18.5	0.18	0.30	0.19	0.14	0.12	0.21	0.14	0.11
SBW-18	18.5	0.19	0.27	0.22	0.13	0.29	0.36	0.31	0.16
SBW-19	18.5	0.45	0.51	0.42	0.19	0.44	0.50	0.41	0.19
SBW-20	18.5	0.34	0.42	0.37	0.16	0.29	0.38	0.33	0.15
SBW-21	18.5	0.23	0.34	0.30	0.14	0.24	0.35	0.32	0.15
SBW-22	15	0.82	0.94	0.86	0.43	0.57	0.72	0.65	0.34
SBW-22	18.5	0.35	0.58	0.56	0.26	0.37	0.60	0.55	0.27
SBW-22	20	0.39	0.62	0.61	0.27	0.30	0.55	0.52	0.24
SBW-22	25	0.38	0.72	0.80	0.30	0.34	0.69	0.71	0.28
SBW-23	15	7.05	7.02	4.34	1.68	6.75	6.65	4.16	1.63
SBW-23	18.5	7.50	7.00	6.32	1.89	7.01	7.08	5.23	1.89
SBW-23	20	8.28	7.13	6.89	2.07	7.69	6.67	6.44	1.98
SBW-23	25	8.84	7.37	9.44	2.30	7.73	6.50	8.16	2.07
SBW-24	18.5	0.72	0.76	0.72	0.33	0.66	0.67	0.63	0.31
SBW-25	15	0.75	0.65	0.43	0.20	0.52	0.47	0.30	0.17
SBW-25	18.5	0.36	0.45	0.37	0.15	0.32	NR[a]	0.31	0.14
SBW-25	20	0.39	0.37	0.35	0.14	0.37	0.36	0.35	0.14
SBW-25	25	0.36	0.34	0.49	0.14	0.34	0.33	0.47	0.14

Figure 6.2 provides a visual comparison of r_i (g/m^2) values for quenched and CCC samples of the SBW glasses. These results were derived using the targeted compositions. For those glasses shown below the 1:1 correlation line in Figure 6.2, the CCC sample is more durable (lower r_i) than its quenched counterpart, for example, the SBW-23 based glasses. The durability criteria are not met for SBW-23 and the SBW-13 based glasses (the latter being the 18.5 mass% WL glass, which yielded an r_B of

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[a] Li value not reported (NR) for SBW-25-18.5ccc.

approximately 15 g/m^2). The low durabilities of these two groups of glasses are primarily a result of the high-alkali content of the glasses with SBW-13 and SBW-23 having 16.16 and 12.33 mass% sum of alkali, respectively.

The normalized releases from SBW-11 and SBW-14 at 18.5% WL also do not meet the conservative <1 g/m^2 acceptance criterion (although their releases are well below those of the EA glass).

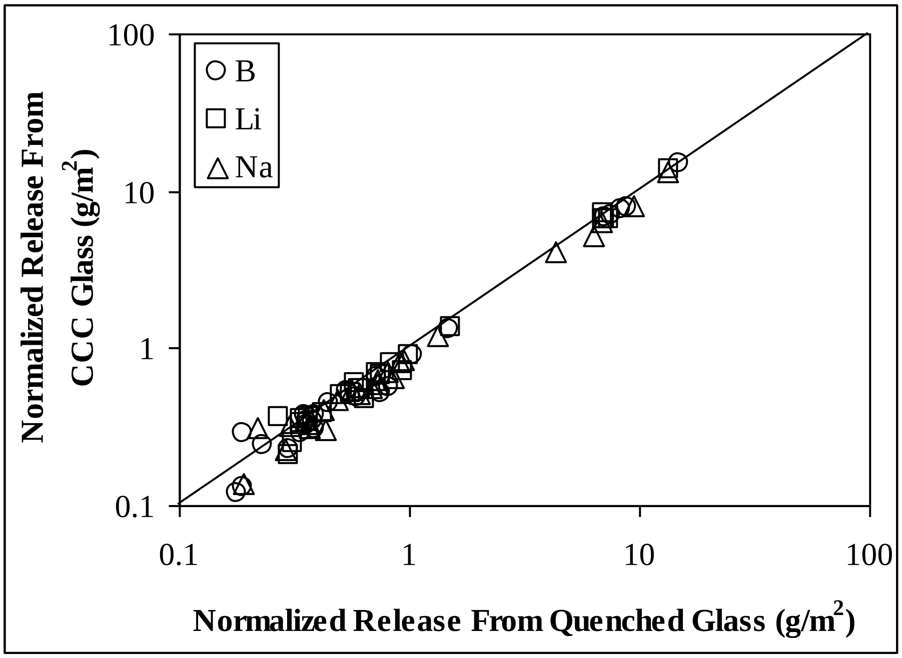

Figure 6.2. Comparison of r_i for Quenched and CCC Glasses

6.3 Summary of Batch-Chemical Testing

Based on the initial scoping tests using batch chemicals, various candidate additive compositions could be eliminated from further consideration based on the acceptance criteria. In terms of solubility, those glasses in which salt was observed provide an indication that the compositional changes were not conducive to maximizing SO_3 solubility. As with previous studies, these results suggested that high concentrations of alkali and alkali-earth elements would improve the incorporation of SO_3 in glass. In fact, it appears that combinations of alkali and alkali-earth are more effective at raising sulfur retention and avoiding salt formation than any single component. However, a quantitative relationship has not yet been developed. Based on this criterion, SBW-17, -18, -19, -20, -21, and -24 were excluded from further study.

With respect to the PCT response, SBW-13 and SBW-23 were not considered as potential candidates, given their high r_i values, even though both retained targeted SO_3 concentrations over the WL range evaluated. Again, the presence of high concentrations of alkali and/or alkali-earth enhances sulfur incorporation, but also increases r_i.

Viscosity measurements relative to acceptance criteria also aided in the down-selection process of potential candidates for recommendation. Given that the acceptance range for viscosity was 2 to 10 Pa-s (for JHCM) and 5 to 10 Pa-s (for CCIM), SBW-17 failed both criteria with SBW-11 (18.5% WL), -15 (18.5% WL), and -25 (25% WL), failing the CCIM criterion on the low side (i.e., < the 5 Pa-s lower limit).

None of the heat treatments at 1050°C indicated devitrification. Thus, this property did not eliminate any of the candidates from further consideration.

Based on the batch chemical testing results, only the following 6 of the 17 candidates produced glasses that met all of the constraints: SBW-12, -14, -16, -22, -26, and -27 (although it should be noted that the PCT was not performed on -26 and -27 based glasses).

7.0 Slurry Simulant Testing

To gain a better understanding of the effects of glass composition on sulfur partitioning from a slurry-fed melter operation, tests were performed to better simulate that process than was allowed by the crucible melts discussed in Section 6.0. This section discusses the fabrication of WM-180 simulant, the test experiments, and the results from these liquid simulant tests.

7.1 Fabrication of WM-180 Simulant and Melter Feed

Six liters of 2001 WM-180 simulant were prepared to support laboratory testing. Table 7.1 summarizes the chemicals and amounts used to fabricate the simulant (listed in order of addition). The simulant was prepared by mixing the dry salts in a beaker. Then just enough deionized water (DIW) was added to dissolve all of the salts with heating and stirring. The manganese nitrate and aluminum nitrate solutions were added to this solution. The resulting solution was continually heated and stirred as the boric acid was added. At this point, the heating and stirring were discontinued, and the solution was transferred to a plastic carboy. The hydrofluoric acid was then added to the slurry and stirred. Then the sulfuric acid was added to the slurry as it was stirred vigorously. About 1 L of DIW was added to the slurry, and then the hydrochloric, phosphoric, nitric, and molybdic acids were added to the slurry. Finally, DIW was added to bring the final volume of the slurry to 6 L. The resulting slurry was then thoroughly mixed by stirring. The composition of the resulting solution was analyzed using inductively coupled plasma (ICP) and ion chromatography (IC). The average concentrations from four ICP measurements are listed in Table 7.2.

Table 7.1. Chemicals Used in the Preparation of 2001 WM-180 Simulant

Chemical Used	Amount Used (g)	Chemical Used	Amount Used
$NaNO_3$	1050	$RuCl_3$	0.9674 g
KNO_3	119	$Sr(NO_3)_2$	0.149 g
$Ca(NO_3)_2\ 4H_2O$	66.8	TiO_2	0.0277 g
$Cd(NO_3)_2\ 4H_2O$	1.4003	$Zn(NO_3)_2\ 6H_2O$	1.89 g
$Ni(NO_3)_2\ 6H_2O$	2.59	KI	0.132 g
$Fe(NO_3)_3\ 9H_2O$	52.7	$H_5As_3O_{10}$	0.476 g
ZrF_4	0.0678	$Mn(NO_3)_2$ (50% soln)	30.2 g
$Cr(NO_3)_3\ 9H_2O$	8.05	2.2M $Al(NO_3)_3$ soln	1810 mL
$Ba(NO_3)_2$	0.089	H_3BO_3	4.59 g
$Ce(NO_3)_3\ 6H_2O$	0.1238	28.9M HF	7.0 mL
$Co(NO_3)_2\ 6H_2O$	0.0337	18M H_2SO_4	23.3 mL
$Cu(NO_3)_2\ 3H_2O$	1.013	12M HCl	14.6 mL
$Gd(NO_3)_3\ 5H_2O$	0.4623	14.6M H_3PO_4	5.6 mL
$Pb(NO_3)_2$	2.4057	15.4M HNO_3	197 mL
$LiNO_3$	0.14	H_2MoO_4	0.2 g
$Mg(NO_3)_2\ 6H_2O$	18.51	15.4M HNO_3	113 mL

Table 7.2. Chemical Analysis of SBW Simulant

Element	Target Concentration (g/mL)	Analyzed Concentration (g/mL)	% Difference	Element	Target Concentration (g/mL)	Analyzed Concentration (g/mL)	% Difference
Al	17,897	15,800	-12	K	7,669	6,600	-14
As	18.69	*		Li	2.355	3.60	53
B	133.0	130	-2	Mg	292.4	295	1
Ba	7.656	5.65	-26	Mn	774.6	810	5
Ca	1,891	1,600	-15	Mo	18.49	*	
Cd	84.76	81	-4	Na	47,308	42,000	-11
Ce	6.625	*		Ni	86.35	78.0	-10
Cl	1,042	*		P	424.4	680	60
Co	1.135	*		Pb	270.8	340	26
Cr	174.3	170	-2	Ru	79.61	*	
Cu	44.29	46.0	4	S	2,238	2,200	-2
F	648.4	*		Sr	10.44	14.0	34
Fe	1,214	1,100	-9	Ti	2.767	*	
Gd	27.87	*		Zn	68.60	*	
I	16.48	*		Zr	6.044	*	
* Analyte not measured							

A slurried melter feed is fabricated from the simulant with the addition of glass-forming chemicals (GFCs) (summarized in Table 7.3), sugar and/or other reductants, extra water and/or HNO_3 (to obtain acceptable slurry flow properties), and H_2SO_4 to adjust the target concentration of SO_3 in the glass. Sufficient slurry feed to fabricate between 50 and 200 g of glass was developed with most feeds designed to fabricate either 50 or 85 g of glass. The GFCs were each weighed in the proper proportions and then slowly stirred into the waste simulant in a beaker on a stir plate. When HNO_3 was used to obtain better flow properties, it was added before adding the GFCs. When water was added to obtain better flow properties, it was added after adding the GFCs. In later tests, the GFCs were premelted into a frit (either all GFCs or partial mixtures) as will be described below.

Table 7.3. Chemical Sources for Glass-Forming Additives

Glass Component	Typical Source Chemical	Alternative Chemical
B_2O_3	H_3BO_3	
CaO	$CaCO_3$	$Ca(OH)_2$
Fe_2O_3	Fe_2O_3	
K_2O	K_2CO_3	
Li_2O	Li_2CO_3	$LiOH \cdot H_2O$
MgO	MgO	$Mg(OH)_2$
Na_2O	Na_2CO_3	NaOH
SiO_2	SiO_2 (-240 mesh)	
V_2O_5	V_2O_5	
ZrO_2	ZrO_2 (-325 mesh)	

7.2 Summary of Ramp Heated Crucible Melts

Ramp-heating crucible melts were performed that were based on SBW-1, -2, -4, -9, -10, and -22 additive compositions. The tests with SBW-2, -4, and -9 used 2000 WM-180 simulant, and the tests with SBW-22 used 2001 WM-180 simulant. The goals of these tests were to determine the appropriate WL, the need for any acid additions or adjustments, and the appropriate additive compositions. Most of the tests were performed using two basic test methods. In the first set of experiments (referred to as Method 1), all melts were made by mixing enough waste simulant and additives to obtain 50 g of glass (after loss of all H_2O, HNO_3, $C_6H_{12}O_6$, and other volatile components). The dry additives were slowly mixed into the waste simulant in either the melting crucible (~500 mL alumina crucibles) or Teflon beakers with a Teflon-coated stir bar. Sugar was added to the mixture along with other solid additives. In many cases, concentrated HNO_3 was added using a pipette to adjust the total pH after solids were added. The mixtures were heated on a hot plate with continued stirring using a hot-plate face temperature of roughly 140°C until nearly dry. The mixtures were transferred to drying ovens to dry overnight (~16 h) at roughly 100°C. For samples dried directly in alumina crucibles, the tight-fitting crucible lids were coated with a glass frit and placed on the crucible in the heat-treatment furnace. For samples dried in Teflon beakers, the sample was first transferred to a high-SiO_2 (>98%) crucible. Heat treatments were performed by ramp heating from between room temperature and 150°C to 240°C at 3°C/min, from 240°C to 300 at 1°C/min, and then from 300°C to 1150°C at 6°C/min, held at 1150°C for 1 h, and quenched to room temperature. The lid was removed from the crucible (usually by breaking it off), and visual observations of the melt were made. A 20-mL sample of DIW was used to wash any salt off the top of the melt/crucible interior. The salt solutions were analyzed using inductively coupled plasma-optical emission spectrometry (ICP-OES) and/or IC. Glass samples were broken from the crucible and ground to roughly 1-μm median particle diameter in a tungsten-carbide mill. Glass powder (and occasionally dry feed samples) were fused in Na_2O_2 and KOH, dissolved in HNO_3, and analyzed by ICP-OES. The S concentration of glass samples was also analyzed by X-ray fluorescence spectroscopy (XRF).

The second set of tests (Method 2) was also performed with the 2000 WM-180 simulant, but differed slightly in the experimental procedure relative to Method 1. In this second set of tests, all melts were made by mixing 150 mL of waste simulant, which required varying quantities of additives to target between 40 to 60 g of glass. The dry additives were slowly mixed into the waste simulant in the melting crucible (~600 mL Pt/Au crucibles) for all tests. The sugar was added to the mixture along with other solid additives. In many cases, a predetermined quantity of concentrated HNO_3 was added using a pipette to adjust the total pH to that of the simulant before adding solids. The mixtures were heated on a hot plate with continued stirring using a hot-plate face temperature of roughly 140°C until nearly dry (during drying, a Pt/Au lid was placed on top of the crucible). The mixtures were transferred to a high-temperature furnace to dry. The furnace was then ramped at ~8°C/min to 150°C and held for 20 min and then to 225°C and held for 20 min. For select tests, a dry sample was obtained at this point for ICP and IC analysis. The furnace was then ramped at 8°C/min to 1150°C and then held for 1 h. The crucible was removed and placed in a pan of water. No lid was used during vitrification. Visual observations of the melt were made during heating. Glass samples were broken from the crucible and analyzed by ICP-OES. Other samples were taken for various property testing.

The results of crucible tests with SBW-1, -2, -4, -9, and -10 were reported by Peeler et al. 2001 and are partially repeated here along with results from tests with SBW-22, 2001 WM-180 simulant, using Method 1. Table 7.4 summarizes the tests performed and their results.

Table 7.4. Parameters, Visual Observations, and Results for the Liquid Simulant Crucible Tests

Glass	WL (mass%)	Sugar (g/L)	Target SO_3	Test	Salt(a)	S in Salt (g)(b)	SO_3 in Glass (%)(b)	Comments
SBW-1	25	141	0.89	2	3	NM	NM	---
SBW-4	20	141	0.71	2	No	NM	NM	---
SBW-4	25	141	0.89	2	No	NM	NM	---
SBW-4	25	141	0.89	2	No	NM	NM	2 $\underline{M}$ H^+
SBW-4	30	141	1.07	2	4	NM	NM	XRD and SEM/EDS Results
SBW-4	30	141	1.07	2	4	NM	NM	2 $\underline{M}$ H^+
SBW-4	30	141	1.07	2	4	NM	NM	3 $\underline{M}$ H^+
SBW-4	35	0	1.25	2	2	NM	NM	---
SBW-4	35	141	1.31	2	4	NM	NM	2 $\underline{M}$ H^+, 1999 SBW
SBW-9	25	141	0.89	2	No	NM	NM	---
SBW-9	25	141	0.89	2	No	NM	NM	---
SBW-9	30	141	1.07	2	No	NM	NM	---
SBW-9	30	141	1.07	2	1	NM	NM	2 $\underline{M}$ H^+
SBW-10	30	141	1.07	2	3	NM	NM	2 $\underline{M}$ H^+, SBW-4 with BaO for CaO
SBW-2	35	77	1.25	1	4	.082	NM	Sugar test FeII/Fe=7.07×10^{-4}
SBW-2	35	109	1.25	1	4	.093	NM	Sugar test FeII/Fe=3.86×10^{-4}
SBW-2	35	131	1.25	1	4	.094	NM	Sugar test FeII/Fe=1.32×10^{-3}
SBW-2	35	164	1.25	1	3	.093	NM	Sugar test FeII/Fe=2.71×10^{-1}
SBW-4Q	35	141	1.25	other	N/a	.002	0.80	2.5 g Q-furnace test
SBW-4D	35	141	1.25	other	4	NM	NM	Dryout test
SBW-4D	35	141	1.25	other	2	NM	NM	3 $\underline{M}$ H^+, Dryout test
SBW-4	35	141	1.25	1	3	.048	0.98	2 $\underline{M}$ H^+, Acid adjustment
SBW-4	35	141	1.25	1	3	.038	0.98	3 $\underline{M}$ H^+, Acid adjustment
SBW-9	30	141	1.07	1	1	.003	1.1	2.1 $\underline{M}$ H^+
SBW-9	30	141	1.07	1	1	NM	NM	2.1 $\underline{M}$ H^+
SBW-9	30	141	1.07	1	1	.025	0.85	---
SBW-9	25	141	0.89	1	2	.019	0.78	---
SBW-9	25	141	0.89	1	3	NM	NM	---
SBW-9	28	141	1.00	1	3	NM	NM	---
SBW-9	32	141	1.14	1	3	NM	NM	---
SBW-9	32	135	1.14	other	No	NM	0.43	oven dried in Al_2O_3 cruc, 5 cf/h N_2 sweep gas
SBW-9	32	135	1.14	other	No	NM	0.23	5 cf/h N_2 sweep gas
SBW-9	32	135	1.14	other	No	NM	0.15	Pt cruc, 5 cf/h N_2 sweep gas fast/exothermic reaction near completion of drying
SBW-22	20	160	0.91	1	No	NM	0.84	---
SBW-22	22.5	160	1.02	1	No	NM	1.00	---
SBW-22	25	160	1.14	1	1	NM	1.01	faint "finger-print" of salt on surface

(a) Salt – 1) faint patterns on surface, 2) hazy patterns cover surface, 3) accumulation of salt in areas, 4) gross accumulation of salt.

(b) NM – Not measured.

Table 7.5 summarizes the matrix of CSM tests performed. The CSM tests were performed in three series. The first series of tests was aimed at determining the effects of different physical and chemical parameters on the partitioning of sulfur between glass, off-gas, and salt phases. This series of tests; performed with SBW-9 based additive mixes (composed of carbonate and oxide source chemicals) and 2000 WM-180 simulant; varied feed rate, free acid, sugar content, reductant type, and SO_3 content. The results from the initial series of tests (CSM041201 through CSM070901) are summarized by Darab et al. (2001) and are partially repeated in this report.

The second series of tests was aimed at determining the influence of additive composition on partitioning of sulfur between the glass, off-gas, and salt phases. This series of tests (CSM070501 through CSM080901) based on 2001 WM-180 simulant varied additive composition and SO_3 content. The results of these tests along with other tests described in this report narrowed the baseline additive composition to SBW-22 and the baseline 2001 WM-180 concentration to 20 mass% on a glass oxide basis.

The third series of tests was aimed at understanding the effects of additive source chemical addition on sulfur behavior. This series of tests (CSM110601 through CSM011102), based on SBW-22 additive composition using 2001 WM-180 simulant at 20% WL, varied sugar content, HNO_3 addition, and chemical form of additives. The chemical form variants included additives comprised of oxide and carbonates, oxides and hydroxides, and full and partial frits. Since the feeds based on GFCs required the addition of water or HNO_3 to obtain acceptable rheology, HNO_3 and sugar concentrations were also systematically varied.

7.3 CSM Experimental Method

The centimeter scale melter (CSM) was developed to better simulate those processes that are important to determining the behavior of sulfur in a slurry-fed melter system with batch heating from the glass melt below (Darab et al. 2001). Figure 7.1 shows a schematic diagram of the CSM used for the study of WM-180 simulant vitrification in the laboratory. The steps and conditions used in CSM testing are described below.

Table 7.5. Summary of CSM Tests Performed

Set	Test ID(a)	Glass	Sim(b)	WL	SO$_3$(c)	HNO$_3$(d)	Sugar(e)	Rate(f)	Comment
1	041201	9	2000	32	1.14	---	135	0.8	Base run
1	042701	9	2000	32	1.14	---	135	1.5	Base run
1	050801	9	2000	32	1.14	---	135	1.5	High sweep gas rate
1	051401	9	2000	32	1.14	---	135	7.2	High feed rate
1	051601	9	2000	32	2.28	---	135	1.3	200% S as Na_2SO_4
1	052401	9	2000	32	1.14	111	135	1.5	240% H^+
1	053101	9	2000	32	1.14	---	---	1.3	No sugar
1	060101	9	2000	32	1.14	---	135	0.5	Low feed rate
1	060501	9	2000	32	1.14	---	135	0.8	Low feed rate
1	060601	9	2000	32	1.14	---	75	1.3	50% sugar
1	060801	9	2000	32	1.14	---	203	1.3	150% sugar
1	061201	9	2000	32	1.14	---	---	1.3	83% C as glycolic acid (13.39 g)
1	061401	9	2000	32	1.14	101	135	1.3	200% H^+
1	061801	9	2000	32	1.14	---	68	1.3	50% C from urea, 50% C from sugar
1	062101	9	2000	32	1.14	---	135	1.3	Base run, SO_2 spike in off-gas
1	062201	9	2000	32	1.14	---	135	1.3	Long run (200 mL feed)
1	062601	9	2000	32	1.14	166	203	1.3	150% sugar, 150% HNO_3
1	062701	9	2000	32	1.14	---	135	1.3	Base run, SO_2 spike in off-gas
1	070901	9	2000	32	1.14	---	---	1.3	100% C (as 16.2 g glycolic acid)
2	070501	11	2001	18.2	0.80	---	140	1.3	H_2O added for rheology
2	071001	11	2001	18.5	1.20	---	140	1.3	H_2O added for rheology
2	071101	11	2001	18.5	1.40	---	140	1.3	H_2O added for rheology
2	071301	22	2001	20	1.40	63	168	1.3	---
2	072001	22	2001	20	1.80	60	164	1.3	---
2	072301	24	2001	20	1.80	60	164	1.3	---
2	072401	23	2001	20	1.80	60	164	1.3	---
2	072501	25	2001	20	1.80	60	164	1.3	---
2	072601	26	2001	20	1.80	60	164	1.3	---
2	080901	27	2001	20	1.80	60	164	1.3	---
3	110601	22	2001	20	0.91	100	207	1.5	carbonates, high sugar
3	110801	22	2001	20	0.91	100	207	1.9	hydroxides, high sugar
3	111201	22	2001	20	0.91	100	207	1.5	frit, high sugar
3	111301	22	2001	20	0.91	---	160	2.8	frit, med sugar
3	111401	22	2001	20	0.91	100	207	1.5	frit, high sugar
3	111601	22	2001	20	0.91	---	160	1.5	frit, med sugar
3	121201	22	2001	20	0.91	100	160	1.5	hydroxides, med sugar
3	121301	22	2001	20	1.80	---	160	1.5	frit, med sugar, high S
3	121401	22	2001	20	0.91	100	160	1.5	frit, med sugar
3	122101	22	2001	20	0.91	100	160	---	hydroxides, med sugar, variable feed rate
3	010402	22	2001	20	0.91	100	127	2.4	hydroxides, low sugar
3	010802	22	2001	20	0.91	---	51	2.0	frit, low sugar
3	010902	22	2001	20	0.91	---	51	2.3	frit (-Ca, -V), low sugar
3	011002	22	2001	20	0.91	---	51	2.4	frit (-V), low sugar
3	011102	22	2001	20	0.91	---	51	2.2	frit (-Ca), low sugar

(a) Test identification numbers are CSMxxxxxx, where xxxxxx represents the date of the test.

(b) CSM test simulants include 2000 WM-180 (2000) and 2001 WM-180 (2001) described in Table 2.1.

(c) SO_3 is the concentration of sulfur targeted in the final glass (assuming no loss) on a mass% SO_3 basis.

(d) HNO_3 is the amount of concentrated nitric acid added to the feed prior to the test on a mili-liter nitric acid/L original SBW simulant basis.

(e) Sugar is the concentration of sucrose added to the feed on a grams of sucrose/L of original SBW simulant basis

(f) Rate is the slurry feed rate to the CSM on a mili-liter per minute basis. All tests described here were run in the 1 inch diameter CSM (5.07×10^{-4} m^2) so the specific feed rate in $L\cdot min^{-1}\cdot m^{-2}$ is rate × 1.974.

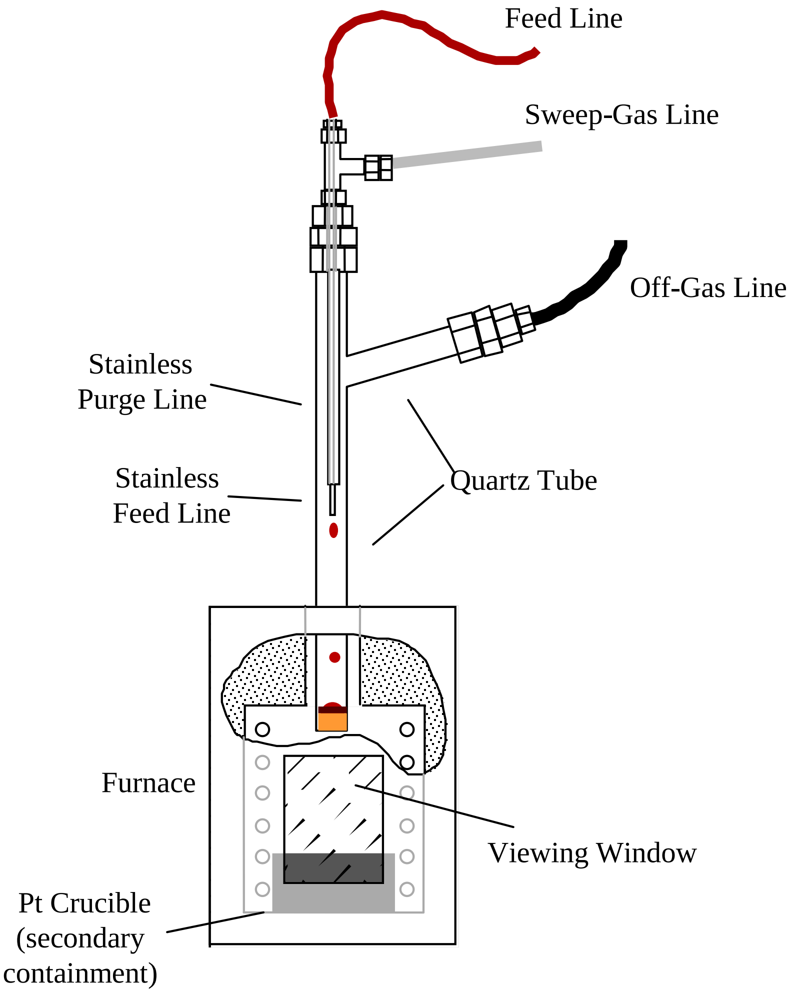

Figure 7.1. **Schematic Diagram of the CSM. Typically, the quartz tube assembly is gradually lowered further into the furnace as the melt level increases with time. For clarity, a cut-away view of part of the furnace is shown.**

The CSM consists of a main vessel of 1-in.-diameter fused-silica tubing as shown in Figure 7.1.(a) This tubing is sealed at the bottom, open at the top, and has a side-arm near the top for gas removal. The tube is suspended in a custom-designed box furnace equipped with a fused-silica window to observe the experiment. The melter feed is introduced through the top of the crucible through an ⅛-in. stainless steel tube that extends past the off-gas side-arm and is roughly centered in the crucible. The feed tube is surrounded by a sweep-gas flow tube that delivers roughly 50 to 100 ccm of helium to facilitate delivery of the gasses generated by the experiment to the off-gas analysis system. The off-gas treatment/analyses were performed using a number of different setups (described by Darab et al. 2001 in detail). Typically, gases exiting the side-arm went into a submerged bed scrubber, through a water-cooled condenser, and into a gas-chromatograph with a mass spectrometer (GC-MS). During the experiment, the concentrations

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(a) Later CSM designs include 1.5- and 3-in.-diameter crucible tubes that flare out from the main interface tube just below the feed tube.

of several fixed gasses were measured using the GC-MS. After each experiment was completed, each of the gas lines was flushed, and the resulting solution was combined with the scrubber solution and condensate for analyses. The combined solutions were analyzed by ICP-AES and IC. The glasses resulting from the testing were analyzed by fusion and dissolution followed by ICP-AES and/or directly by XRF.

To perform the CSM tests, slurried melter feed was stirred in a beaker on a stir-plate. A Tygon® tube was used to draw the feed directly from the beaker, through a systolic pump, and to the ⅛-in. stainless steel feed tube at a desired rate. The feed drops from the feed tube to the melt surface where it is heated from the glass melt below. Although the plenum temperature is not controlled, the crucible can be placed fully into the furnace (which is typically held at 1150°C) or more typically, adjusted so that the glass-melt/cold-cap interface is maintained at the furnace hot-zone chamber-top refractory interface to assure that the primary source of heat to the melting batch comes from the glass melt pool below.

7.4 CSM Results

The results from the first series of CSM tests are reported by Darab et al. (2001). We repeat many of those results here but primarily focus on the second two series of glasses. Table 7.6 summarizes the sulfur partitioning between the off-gas scrub solution, glass, and salt for the CSM tests listed in Table 7.5.

Of direct importance to formulating a glass for scaled melter testing with SBW is the partitioning of sulfur between the off-gas, glass, and salt. Of the set of three tests (i.e., SBW-11, -22, -23, -24, -25, -26, and -27), only SBW-22 and -27 were able to process with 1.8 mass% SO_3 without the formation of a salt phase. From these two tests, SBW-22 had slightly higher sulfur in the glass and slightly lower sulfur in the scrub solution. However, these differences were not significant. In comparison, SBW-9 formed a salt in most tests with 1.14 mass% SO_3. Since SBW-22–based glass was fully characterized while SBW-27–based glasses were not, and all other results appeared to be nearly equal, SBW-22 was chosen for further testing.

Table 7.6. Summary of CSM Test Results

Set	Test	Glass	WL	Comment	%SO_3 in Glass	Salt	Fraction of S Found in Each				Fe^{II}/Fe
							Glass	Scrub	Salt	Sum	
1	041201	9	32	Base run	0.84	1	NA(a)	NA	NA	NA	NA
1	042701	9	32	Base run	0.84	0.5	0.73	0.26	0.01	1.00	0.07
1	050801	9	32	High sweep gas rate	0.77	1.5	NA	NA	NA	NA	0.00
1	051401	9	32	High feed rate	0.81	4	0.71	0.45	0.16	1.31	0.03
1	051601	9	32	200% S as Na_2SO_4	0.96	2	NA	NA	NA	NA	0.00
1	052401	9	32	240% H^+	0.81	0	0.70	0.15	0.00	0.85	0.21
1	053101	9	32	No sugar	1.14	1	1.00	NA	NA	1.00	0.10
1	060101	9	32	Low feed rate	1.06	1	0.93	NA	NA	0.93	0.08
1	060501	9	32	Low feed rate	NA	0	NA	NA	NA	NA	NA
1	060601	9	32	50% sugar	1.10	1	0.96	0.21	0.02	1.18	0.02
1	060801	9	32	150% sugar	0.35	0	0.31	0.54	NA	0.85	0.80
1	061201	9	32	83% C as glycolic acid (13.39 g)	0.85	1	0.74	0.24	0.00	0.99	0.01
1	061401	9	32	200% H^+	0.88	0	0.77	0.21	NA	0.98	0.06
1	061801	9	32	50% C from urea, 50% C from sugar	0.45	1	0.40	NA	NA	0.40	0.30
1	062101	9	32	Base run, SO_2 spike in off-gas	0.71	0.5	0.62	0.26	NA	0.88	0.12
1	062201	9	32	Long run (200 mL feed)	0.85	1	0.74	0.20	0.00	0.94	0.10
1	062601	9	32	150% sugar, 150% HNO_3	0.58	0	0.50	0.21	NA	0.71	0.49
1	062701	9	32	Base run, SO_2 spike in off-gas	NA	NA	NA	NA	NA	NA	NA
1	070901	9	32	100% C (as 16.2 g glycolic acid)	0.84	0	0.73	0.20	NA	0.94	0.03
2	070501	11	18.2	---	0.63	0	0.75	NA	NA	0.75	NA
2	071001	11	18.5	---	1.12	0	0.92	0.06	NA	0.97	0.19
2	071101	11	18.5	---	1.01	0	0.70	0.08	NA	0.78	NA
2	071301	22	20	---	0.94	0	0.66	0.14	0.00	0.79	0.10
2	072001	22	20	---	1.17	0	0.61	0.15	0.00	0.76	NA
2	072301	24	20	---	1.12	2	0.59	0.18	0.02	0.79	NA
2	072401	23	20	---	1.57	3	0.82	0.11	0.00	0.94	NA
2	072501	25	20	---	1.25	3	0.65	0.18	0.01	0.84	NA
2	072601	26	20	---	1.16	3	0.60	0.14	0.06	0.80	NA
2	080901	27	20	---	1.12	0	0.59	0.17	NA	0.75	0.04
3	110601	22	20	carbonates, high sugar	NA	NA	NA	NA	NA	NA	NA
3	110801	22	20	hydroxides, high sugar	0.70	0	0.77	0.12	NA	0.89	0.15
3	111201	22	20	frit, high sugar	0.55	0	0.60	0.24	NA	0.84	0.63
3	111301	22	20	frit, med sugar	0.66	0	0.72	0.21	NA	0.93	0.37
3	111401	22	20	frit, high sugar	0.55	0	0.60	0.19	NA	0.79	0.49
3	111601	22	20	frit, med sugar	0.61	0	0.66	0.19	NA	0.86	0.48
3	121201	22	20	hydroxides, med sugar	0.80	0	0.88	NA	NA	0.88	0.04
3	121301	22	20	frit, med sugar, high S	0.89	0	0.53	NA	NA	0.53	0.42
3	121401	22	20	frit, med sugar	0.75	0	0.82	NA	NA	0.82	0.11
3	122101	22	20	hydroxides, med sugar, variable feed rate	NA	0	NA	NA	NA	NA	NA
3	010402	22	20	hydroxides, low sugar	0.82	0	0.90	0.06	NA	0.96	0.09
3	010802	22	20	frit, low sugar	0.88	0	0.97	0.03	NA	1.00	0.05
3	010902	22	20	frit (-Ca, -V), low sugar	0.87	0	0.95	0.02	NA	0.97	0.06
3	011002	22	20	frit (-V), low sugar	0.87	0	0.96	0.02	NAN	0.97	0.05
3	011102	22	20	frit (-Ca), low sugar	0.85	0	0.93	0.02	NA	0.95	0.06

(a) NA = Not Analyzed

The set-three tests were designed to determine the influence of the chemical form of the additives the addition of HNO_3, and sugar additions on the sulfur behavior of SBW-22-20 processed in the CSM. Five variations of additives were used in testing:

1. Hydroxides and oxides of all additives where used in tests CSM110801, CSM121201, CSM122101, and CSM010402.
2. The additives were prefritted into a glass that was ground and used in tests CSM111201, CSM111301, CSM111401, CSM111601, CSM121301, CSM121401, and CSM010802.
3. All of the additives except CaO and V_2O_5 were prefritted into a glass that was used in test CSM010902.
4. All of the additives except V_2O_5 were prefritted into a glass that was used in test CSM011002.
5. All of the additives except CaO were prefritted into a glass that was used in test CSM011102.

Those tests with hydroxide- and oxide-based feeds required the addition of HNO_3 to give the feed adequate rheological properties to allow for pumping through the feed system of the CSM. Table 7.7 summarizes the concentrations of sugar and nitrate and the glass redox. Oddly, there is not a clear relationship between redox (Fe^{II}/Fe or Fe^{II}/Fe^{III}) and the mole ratio of NO_3:C for these samples. Figure 7.2 shows that, generally, redox is decreased by increasing NO_3:C; however, clearly there are other physical and chemical parameters that affect redox in addition to the NO_3:C ratio. The appropriate amount of sugar to add to frit-based SBW-22 melter feeds is roughly 50 g/L of waste simulant. However, for hydroxide-based SBW-22 melter feeds, 100 g of HNO_3/L of simulant is used to obtain appropriate rheology, and this requires roughly 115 g of sugar/L of SBW to obtain a redox of roughly 5% reduced in CSM tests. It is yet unclear how this translates to other melter systems.

Table 7.7. Summary of Glass Redox and Sugar/NO_3 Concentrations

Test ID	Description	Sugar g/L SBW	HNO_3 g/L SBW	NO_3:C (mole ratio)	Fe^{II}/Fe
110801	hydroxides, high sugar	207	100	0.938	0.15
111201	frit, high sugar	207	100	0.938	0.63
111301	frit, med sugar	160	---	0.940	0.37
111401	frit, high sugar	207	100	0.938	0.49
111601	frit, med sugar	160	---	0.940	0.48
121201	hydroxides, med sugar	160	100	1.214	0.04
121301	frit, med sugar, high S	160	---	0.940	0.42
121401	frit, med sugar	160	100	1.214	0.11
010402	hydroxides, low sugar	127	100	1.530	0.09
010802	frit, low sugar	51	---	2.948	0.05
010902	frit (-Ca, -V), low sugar	51	---	2.948	0.06
011002	frit (-V), low sugar	51	---	2.948	0.05
011102	frit (-Ca), low sugar	51	---	2.948	0.06

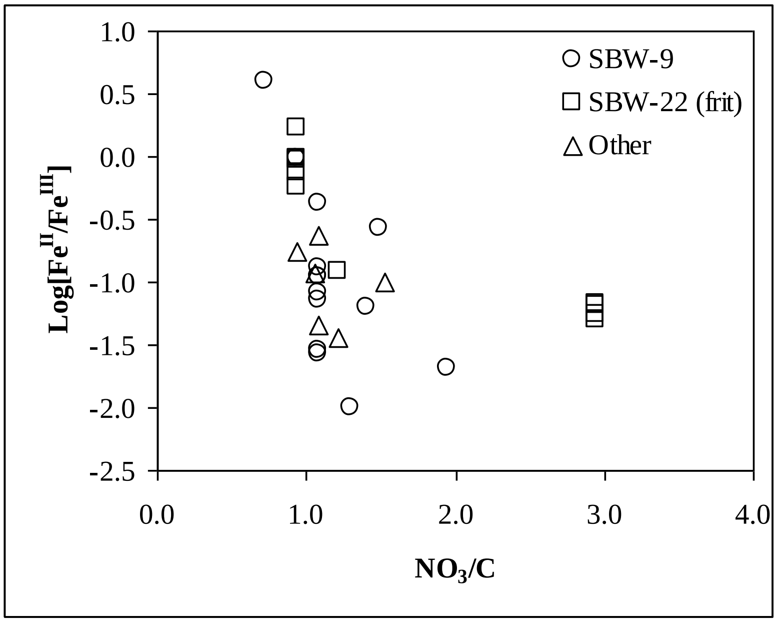

Figure 7.2. Redox as a Function of NO_3:C Ratio

No significant difference between the sulfur partitioning was seen in tests with CaO, V_2O_5, or both being removed from the frit or between tests with frit and the hydroxide-based feeds. It is therefore recommended that a prefabricated frit (containing all additive components) be used in testing SBW vitrification.

7.5 Summary of SMRF Tests

Before RSM or pilot-scale tests, it was desirable to evaluate various formulations (or melter feeds) for their potential to form salt layers using the slurry-fed melt-rate furnace (SMRF). Cozzi et al. (2002) provide a more detailed description of these tests. The SMRF has two chambers. As shown in Figure 7.3, the lower chamber is heated by three SiC heating elements, and the upper chamber is heated by four plate heaters. An 8-in.-diameter Inconel® crucible is positioned in the furnace so that the bottom of the crucible is flush with the top of the lower chamber, and the top of the crucible is flush with the top of the furnace. The SiC elements in the lower chamber heat the crucible from the bottom. This method of heating creates a vertical temperature profile in the crucible similar to the one-dimensional heat transfer from a glass pool to a cold cap. The plate heaters are used to maintain the plenum temperature at the selected setpoint.

The top of the crucible contains three ports: a thermocouple port to measure plenum temperature, a vent port to allow gases to escape, and a port to insert the feed tube. The furnace has a thermocouple inserted into the lower chamber so that the tip is positioned directly underneath the crucible bottom. The power (to the plates in the upper chamber) to maintain plenum temperature is controlled by a feedback loop from the thermocouple in the plenum. The glass-pool temperature is maintained by adjusting the power input to the SiC elements in the lower chamber to maintain a constant temperature in the lower chamber. The power to the SiC elements is controlled by a feedback loop from the thermocouple in contact with the Inconel® crucible.

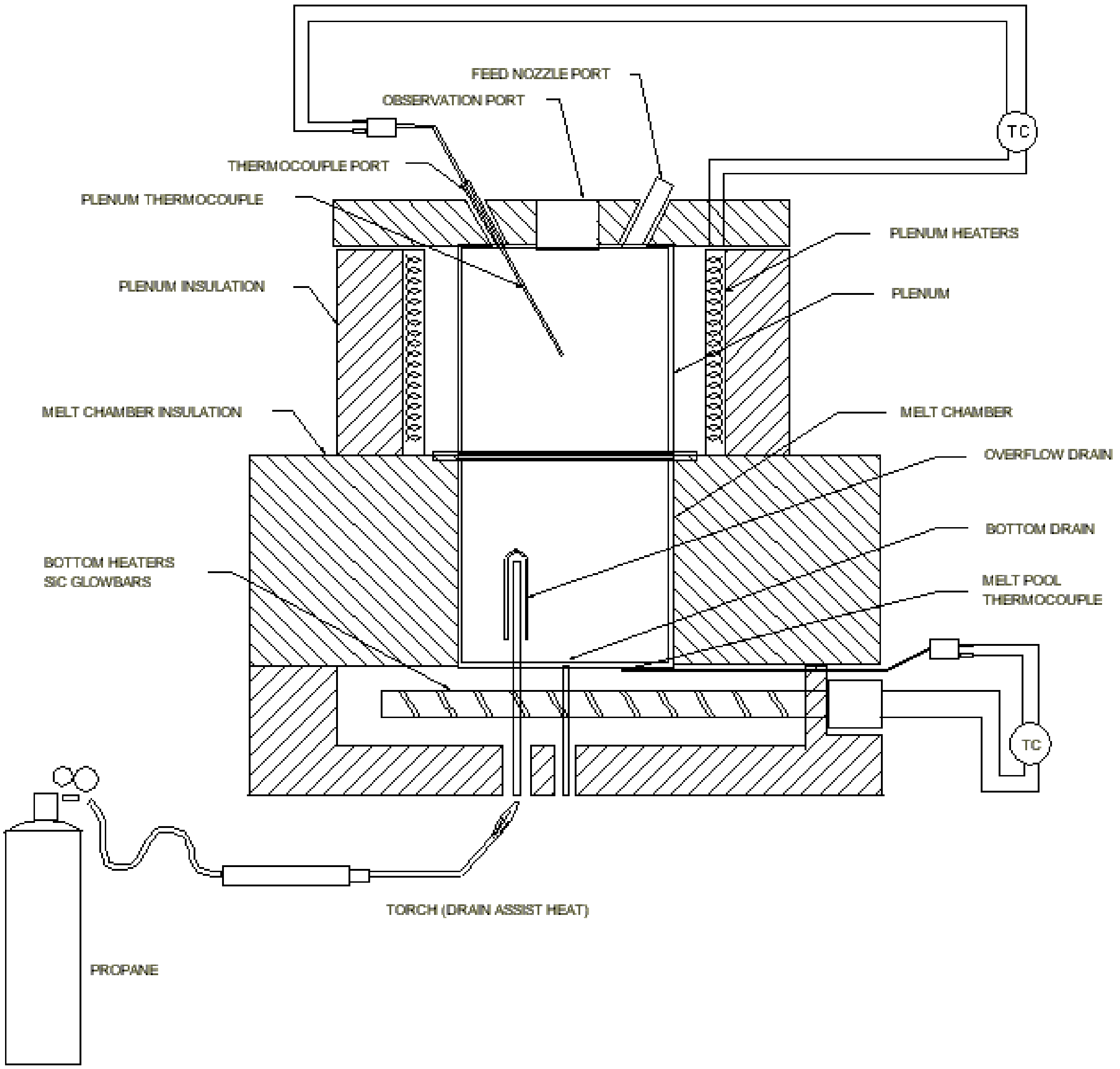

Figure 7.3. Schematic of the Melt-Rate Furnace

The feed system consists of a feed vessel with agitation set on a weigh scale, a dispensing peristaltic pump, and a water-jacketed feed tube, as shown in Figure 7.4. The pump is set up to dispense the same amount of feed each time the controller triggers the pump. The controller triggers the pump to run, based on a set-point plenum temperature. When the pump is triggered, the plenum drops 20 to 30°C as the feed is added. After the pump stops, the plenum temperature recovers as the feed burns off. When the plenum temperature rises back to the setpoint, the feed controller triggers the feed pump, and more feed is dispensed—setting up a semi-continuous feeding system. The controller was set to trigger feeding at 750°C during all four runs.

The glass is poured from the crucible to maintain a constant level using an overflow tube heated by a propane torch. A drain tube is also installed on the crucible to empty the vessel at the conclusion of a run, if desired. A scale is used to record the amount of glass poured.

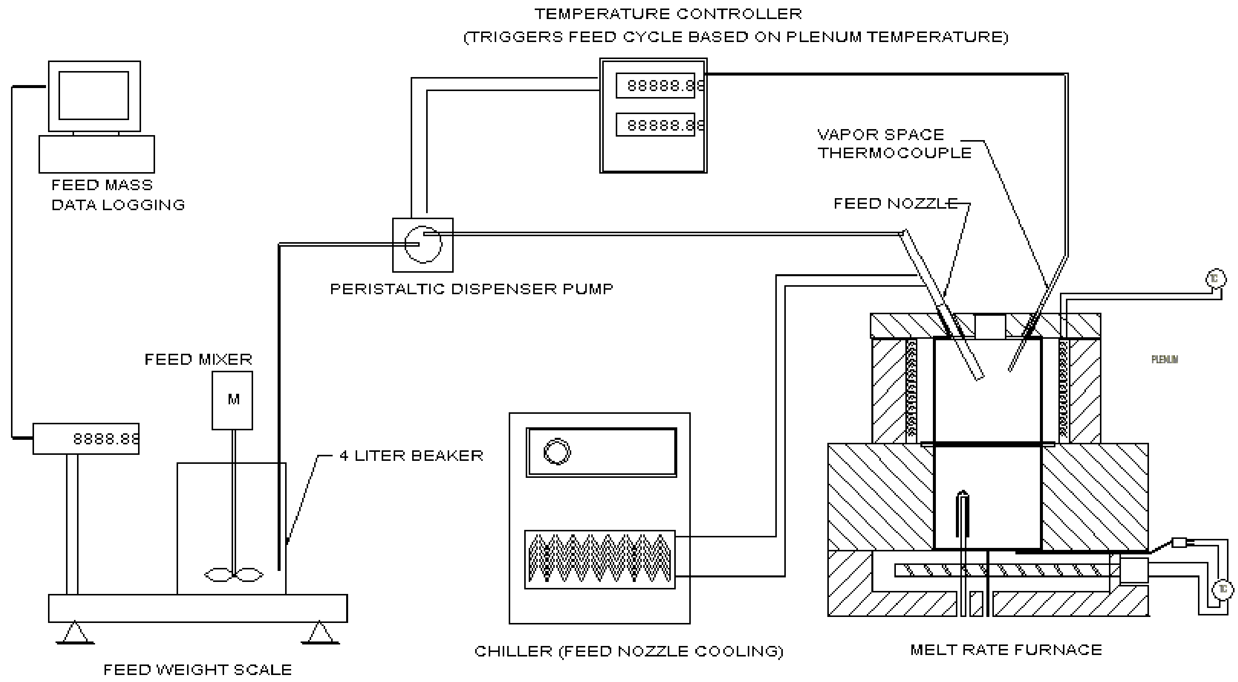

Figure 7.4. Schematic of the SMRF and feed system

Four runs were performed with the SMRF to assess the processability and sulfur behavior during the vitrification of SBW. These tests represent a significant scale-up of the vitrification process as compared to the CSM. The results of these four tests are described below.

7.5.1 Run 1: SBW-9-30

Run 1 was an 8-h run with operating parameters set to mimic to the extent possible the run conditions of the FY01 EV-16 run (Perry et al. 2001). Using melter feed from the EV-16 run (based on SBW-9 additives at a 30% WL), Run 1 resulted in a glass pool with a salt layer similar to that observed in the EV-16 melter. Over 17 kg of melter feed were processed. As previously mentioned, although laboratory-scale testing had not indicated the formation of a salt layer, the as-batched SO_3 concentration in the EV-16 melter feed was the equivalent of 1.35 mass%, significantly higher than the target value of 1.07 mass%.

7.5.2 Run 2: SBW-9-18.2

The primary objective for Run 2 (SBW-9 additives at 18.2% WL) was two-fold: 1) to assess the potential salt-layer formation at a lower WL and 2) to provide a compositional transition to the 2001 WM-180 simulate (glass turnover from Run 1 in anticipation of using the WM-180 2001 simulant for

Runs 3 and 4). Run 2 was an 8-h run based on the revised 2001 SBW simulant with melter operational parameters equivalent to Run 1. Over 18 kg of melter feed were processed during this run with no visible salt layer present. This indicated that by lowering the WL to 18.2%, the formation of a salt layer could be eliminated or at least minimized.

7.5.3 Run 3: SBW-22-18.2

This was an 8-h run with the 2001 SBW simulant composition and SBW-22 composition at 18.2% loading (Stone 2001a). Run 3 was performed to assess if a salt layer would form or accumulate using the glass composition being evaluated for recommendation for future RSM and EV-16 runs. Melter feed was made using the 2001 SBW simulant, and as in Run 2, it was mixed with glass formers. Sugar was used as the reductant at the levels tested in the CSM at PNNL (135 g sugar/L SBW simulant). Additional water and nitric acid were necessary to allow the feed to be pumped. The nitrate additions were compensated by trimming the feed with 33 g sugar/mole nitrate added as nitric acid. During the 8 h of furnace operation, over 15 kg of melter feed were added to the furnace, producing more than 4 kg of glass. Cold-cap, melt-pool, and glass-pour-stream samples were taken throughout the test.

No evidence of sulfur or salt accumulation was observed in the cold-cap samples or by visual observation after cold-cap burn off.

7.5.4 Run 4: SBW-22-20a, -22b

This run was a 48-h run with the revised 2001 SBW simulant composition and SBW-22 composition at 20% loading (Stone 2001b). Approximately 33 h into the run, the loading was increased to 22%. The increase in waste loading was implemented as there was no appreciable salt layer forming at a waste loading of 20 wt%. The increase was an attempt to determine if the 20 wt% waste loading was the maximum obtainable for this simulant composition, or if higher waste loadings were attainable. Run 4 was performed to determine if a salt layer forms during extended processing and, if it is stable, diminishes or expands. A cold-cap sample and a glass-pour sample were taken every 8 h of operation. During this time, feeding was suspended for 30 min, and a dip sample was taken.

As in the previous run, running SBW-22 at 20 and 22% WL showed no evidence of salt accumulation.

7.5.5 Summary

The results of the four tests are summarized in Table 7.8. The SMRF results are similar to crucible melts and CSM melts in that SBW-22-20 was easily processed without the formation of a salt and with nearly full incorporation of sulfur into the glass. This test revealed a barely detectable wisp of salt when 22% WL was processed in both SMRF and Method 1 crucible melts, which is another key similarity to previous tests.

Table 7.8. Summary of SMRF Test Runs with SBW Simulants

Run	Test ID	time (h)	Sim	Target SO_3 (%)	Measured SO_3 (%)	Salt	Comments
1	SBW-9-18.2	8	2001	0.83	0.77	None	Melter dip sample contained 0.88 mass% SO_3.
2	SBW-9-30	8	2000 from Perry et al. (2001)	1.35	0.95	Salt Layer	Although the target level of SO_3 was 1.07%, feed from a previous pilot melter test was used with significantly higher SO_3 than targeted. The melter-dip sample contained 1.17 mass% SO_3.
3	SBW-22-18.2	8	2001	0.84	0.80	None	
4a	SBW-22-20	33	2001	0.91	0.94	None	
4b	SBW-22-22	15	2001	1.00	0.99	None	

8.0 Conclusions and Recommendations

The results from the tests presented above suggest that SBW-22 frit (or additive mix) is appropriate for scale-up tests of the direct vitrification of 2001 WM-180 simulant at 20 mass% loading. This composition is summarized in Table 8.1 on a mass% oxide basis. The glass was formulated to be processable in both a JHCM and an ICCM with properties sufficient for disposal of the glass at the planned federal geologic repository.

Table 8.1. SBW-22-20 Composition

Component	2001 WM-180	SBW-22 (additives)	SBW-22-20
Ag_2O	0.001		0.000
Al_2O_3	27.524		5.505
As_2O_3	0.040		0.008
B_2O_3	0.349	6.035	4.898
BaO	0.007		0.001
CaO	2.154	5.020	4.447
CdO	0.079		0.016
Ce_2O_3	0.013		0.003
Cl	0.865		0.173
CoO	0.001		0.000
Cr_2O_3	0.207		0.041
Cs_2O	0.001		0.000
CuO	0.045		0.009
F	0.733		0.147
Fe_2O_3	1.412	1.520	1.498
Gd_2O_3	0.026		0.005
I	0.007		0.001
K_2O	7.525		1.505
Li_2O	0.004	6.110	4.889
MgO	0.395	1.750	1.479
MnO	0.814		0.163
MoO_3	0.023		0.005
Na_2O	51.911	4.295	13.818
Nb_2O_5	0.002		0.000
NiO	0.089		0.018
P_2O_5	0.791		0.158
PbO	0.237		0.047
PdO	0.002		0.000
ReO_2	0.001		0.000
RuO_2	0.014		0.003
Sb_2O_3	0.008		0.002
SeO_2	0.013		0.003
SiO_2		67.951	54.360
SO_3	4.548		0.910
SrO	0.010		0.002
TiO_2	0.004		0.001
V_2O_5	0.068	4.880	3.918
ZnO	0.069		0.014
ZrO_2	0.007	2.440	1.953
SUM	**100.000**	**100.000**	**100.000**

The main focus of formulation work and testing was minimizing the likelihood of salt accumulation and maximizing sulfur retention in the melt. Since these are both strong functions of the chemical and physical processes involved in the melter, it is difficult to extrapolate the results from smaller scale tests performed during this study to the melter. Therefore, it is recommended that the glass be fabricated in scaled melter tests to assure that processability is adequate at a larger scale.

9.0 References

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Appendix A

Target vs. Measured Chemical Composition Analysis

Appendix A: Target vs. Measured Chemical Composition Analysis

	SBW-11		**SBW-12**		**SBW-13**	
WL (mass%)	**18.5**		**18.5**		**18.5**	
Oxide	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**
Al_2O_3	5.091	5.33	5.091	5.16	5.091	5.05
B_2O_3	9.967	10.6	4.979	4.86	9.967	9.21
BaO	0.002		0.002		0.002	
CaO	4.489	4.65	4.489	4.56	0.398	0.391
CdO	0.015		0.015		0.015	
Cr_2O_3	0.039		0.039	0.043	0.039	0.041
CuO	0.009		0.009		0.009	
Fe_2O_3	1.500	1.63	1.500	1.50	1.500	1.44
Gd_2O_3	0.006		0.006		0.006	
K_2O	1.393	1.21	1.393	1.48	8.035	8.22
Li_2O	4.980	5.27	4.980	4.88	4.980	4.75
MgO	1.498	1.59	1.498	1.50	0.072	0.045
MnO	0.150	0.14	0.150	0.153	0.150	0.152
MoO_3	0.004		0.004		0.004	
Na_2O	11.152	11.0	15.292	15.4	11.152	11.1
NiO	0.017		0.017	0.025	0.017	0.024
P_2O_5	0.146		0.146		0.146	
PbO	0.044		0.044	0.048	0.044	0.044
RuO_2	0.002		0.002		0.002	
SO_3	0.842	0.749	0.842	0.824	0.842	0.819
SiO_2	52.347	54.1	53.195	54.1	51.223	52.4
V_2O_5	3.990	3.96	3.990	(a)	3.990	(a)
ZnO	0.002		0.002		0.002	
ZrO_2	1.990	1.89	1.990	1.97	1.990	1.96
Cl	0.161		0.161		0.161	
F	0.135		0.135		0.135	
Total	*100.00*	*102*	*100.00*	*96.0*	*100.00*	*95.2*

(a) Analysis of V_2O_5 was not request on the analytical submission form, therefore the sum of oxides should be < 100%.

	SBW-14		**SBW-15**		**SBW-16**		**SBW-17**	
WL (mass%)	**18.5**		**18.5**		**18.5**		**18.5**	
Oxide	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**
Al_2O_3	5.091	5.12	5.091	5.08	5.091	5.12	5.091	5.23
B_2O_3	11.842	11.3	5.770	5.54	11.475	10.8	9.845	10.0
BaO	0.002		0.002		0.002		0.002	
CaO	4.489	4.56	6.918	7.01	6.103	6.19	2.028	1.80
CdO	0.015		0.015		0.015		0.015	
Cr_2O_3	0.039	0.041	0.039	0.041	0.039	0.055	0.039	0.028
CuO	0.009		0.009		0.009		0.009	
Fe_2O_3	1.500	1.50	6.781	6.83	0.261	0.306	10.041	9.59
Gd_2O_3	0.006		0.006		0.006		0.006	
K_2O	1.393	1.49	1.393	1.45	1.393	1.45	1.393	1.24
Li_2O	4.980	4.86	4.890	4.77	4.890	4.73	3.260	3.21
MgO	1.498	1.50	0.072	0.045	0.072	0.053	0.072	
MnO	0.150	0.154	0.150		0.150	0.151	0.150	0.137
MoO_3	0.004		0.004		0.004		0.004	
Na_2O	11.152	11.2	15.308	14.9	11.233	11.3	9.603	9.87
NiO	0.017	0.024	0.017	0.023	0.017	0.025	0.017	
P_2O_5	0.146		0.146		0.146		0.146	
PbO	0.044	0.044	0.044	0.046	0.044	0.043	0.044	0.040
RuO_2	0.002		0.002		0.002		0.002	
SO_3	0.842	0.722	0.842	0.844	0.842	0.737	0.842	0.477
SiO_2	54.450	54.3	52.160	52.4	57.865	57.1	57.050	53.6
V_2O_5	0.013		0.013		0.013		0.013	0.09
ZnO	0.002		0.002		0.002		0.002	
ZrO_2	1.990	1.94	0.002	<0.015	0.002	<0.012	0.002	
Cl	0.161		0.161		0.161		0.161	
F	0.135		0.135		0.135		0.135	
Total	*100.00*	*98.3*	*100.00*	*98.5*	*100.00*	*97.6*	*100.00*	*95.0*

	SBW-18		SBW-19		SBW-20		SBW-21	
WL (mass%) Oxide	**18.5 Target**	**Meas.**	**18.5 Target**	**Meas.**	**18.5 Target**	**Meas.**	**18.5 Target**	**Meas.**
Al_2O_3	5.091	5.24	5.091	5.33	5.091	5.30	5.091	5.42
B_2O_3	9.845	9.89	9.845	9.86	9.845	9.85	8.215	8.29
BaO	0.002		0.002		0.002		0.002	
CaO	2.028	1.77	2.028	1.80	3.658	3.71	3.658	3.82
CdO	0.015		0.015		0.015		0.015	
Cr_2O_3	0.039	0.031	0.039	0.032	0.039	0.040	0.039	0.047
CuO	0.009		0.009		0.009		0.009	
Fe_2O_3	10.041	9.69	10.041	9.89	10.041	10.0	10.041	10.1
Gd_2O_3	0.006		0.006		0.006		0.006	
K_2O	1.393	1.27	1.393	1.28	1.393	1.27	1.393	1.27
Li_2O	2.445	2.18	2.445	2.14	2.445	2.13	2.445	2.10
MgO	0.072		0.072		0.072		0.072	
MnO	0.150	0.135	0.150	0.143	0.150	0.137	0.150	0.141
MoO_3	0.004		0.004		0.004		0.004	
Na_2O	12.863	12.4	12.863	12.6	12.863	12.6	12.863	12.6
NiO	0.017		0.017		0.017		0.017	
P_2O_5	0.146		0.146		0.146		0.146	
PbO	0.044	0.041	0.044	0.041	0.044	0.040	0.044	0.042
RuO_2	0.002		0.002		0.002		0.002	
SO_3	0.842	0.602	0.842	0.527	0.842	0.589	0.842	0.582
SiO_2	52.160	51.4	50.530	50.9	48.900	49.9	50.530	51.7
V_2O_5	1.643	1.64	3.273	3.24	3.273	3.31	3.273	3.29
ZnO	0.002		0.002		0.002		0.002	
ZrO_2	0.817		0.817		0.817	0.777	0.817	0.757
Cl	0.161		0.161		0.161		0.161	
F	0.135		0.135		0.135		0.135	
Total	*100.00*	*97.0*	*100.00*	*98.0*	*100.00*	*99.0*	*100.00*	*100*

WL (mass%)	**SBW-22**							
	15		**18.5**		**20**		**25**	
Oxide	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**
Al_2O_3	4.12	4.27	5.09	5.18	5.50	5.63	6.88	7.03
B_2O_3	5.17	5.28	4.97	4.89	4.89	4.67	4.61	4.35
BaO	0.00		0.00		0.00		0.00	
CaO	4.59	4.23	4.48	4.06	4.44	4.07	4.30	4.00
CdO	0.01		0.01		0.01		0.02	
Cr_2O_3	0.03	0.032	0.03	0.074	0.04	0.058	0.05	0.058
CuO	0.00		0.00		0.01		0.01	
Fe_2O_3	1.50	1.76	1.50	1.50	1.49	1.47	1.49	1.49
Gd_2O_3	0.00		0.00		0.00		0.00	
K_2O	1.13	1.25	1.39	1.49	1.50	1.66	1.88	2.10
Li_2O	5.19	4.97	4.98	4.79	4.88	4.62	4.58	4.30
MgO	1.54	1.61	1.49	1.52	1.47	1.51	1.41	1.46
MnO	0.12	0.12	0.15	0.14	0.16	0.15	0.20	0.20
MoO_3	0.00		0.00		0.00		0.00	
Na_2O	11.43	11.29	13.10	12.81	13.814	13.50	16.195	16.20
NiO	0.01	0.018	0.01	0.019	0.01	0.02	0.02	0.024
P_2O_5	0.11	0.13	0.14	0.15	0.15	0.17	0.19	0.21
PbO	0.03	0.04	0.04	0.04	0.04	0.05	0.06	0.06
RuO_2	0.00		0.00		0.00		0.00	
SO_3	0.68	0.599	0.84	0.737	0.91	0.806	1.13	1.074
SiO_2	57.76	58.42	55.387	53.93	54.36	55.00	50.97	52.22
V_2O_5	4.15	4.01	3.99	3.74	3.91	3.69	3.67	3.49
ZnO	0.00		0.00		0.00		0.00	
ZrO_2	2.07	1.958	1.99	1.823	1.95	1.823	1.83	1.755
Cl	0.13		0.16		0.17		0.21	
F	0.11		0.13		0.14		0.18	
Total	*100.00*	*100.09*	*100.00*	*97.01*	*100.00*	*99.01*	*100.00*	*100.12*

	SBW-23							
WL (mass%)	**15**		**18.5**		**20**		**25**	
Oxide	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**
Al_2O_3	4.128	4.12	5.091	5.16	5.504	5.54	6.880	6.75
B_2O_3	10.380	10.2	9.967	9.72	9.790	9.55	9.200	8.84
BaO	0.002		0.002		0.002		0.003	
CaO	0.323	<0.004	0.398	0.174	0.430	0.151	0.538	0.248
CdO	0.012		0.015		0.016		0.020	
Cr_2O_3	0.032		0.039		0.042		0.053	
CuO	0.008		0.009		0.010		0.013	
Fe_2O_3	1.504	1.67	1.500	1.63	1.498	1.67	1.493	1.66
Gd_2O_3	0.005		0.006		0.006		0.008	
K_2O	4.802	4.27	4.914	4.38	4.962	4.50	5.123	4.46
Li_2O	5.194	5.44	4.980	5.28	4.888	5.22	4.583	4.85
MgO	0.059	0.04	0.072	0.06	0.078	0.060	0.098	0.090
MnO	0.122	0.12	0.150	0.15	0.162	0.160	0.203	0.200
MoO_3	0.003		0.004		0.004		0.005	
Na_2O	9.402	9.32	11.152	11.1	11.902	11.8	14.403	14.1
NiO	0.014		0.017		0.018		0.023	
P_2O_5	0.119	0.166	0.146	0.196	0.158	0.207	0.198	0.243
PbO	0.036		0.044		0.048		0.060	
RuO_2	0.002		0.002		0.002		0.003	
SO_3	0.683	0.694	0.842	0.914	0.910	1.01	1.138	1.35
SiO_2	56.678	56.3	54.344	52.4	53.344	52.4	50.010	49.9
V_2O_5	4.159	3.77	3.990	3.57	3.918	3.57	3.678	3.25
ZnO	0.002		0.002		0.002		0.003	
ZrO_2	2.076	2.01	1.990	1.92	1.954	1.90	1.833	1.82
Cl	0.131		0.161		0.174		0.218	
F	0.110		0.135		0.146		0.183	
Total	*100.00*	*97.8*	*100.00*	*96.3*	*100.00*	*97.3*	*100.00*	*97.1*

	SBW-25$^{(a)}$							
WL (mass%) Oxide	**15 Target**	**Meas.**	**18.5 Target**	**Meas.**	**20 Target**	**Meas.**	**25 Target**	**Meas.**
Al_2O_3	4.128	4.18	5.091	5.16	5.504	5.59	6.880	6.86
B_2O_3	10.380	10.1	9.967	9.59	9.790	9.66	9.200	9.11
BaO	0.002		0.002		0.002		0.003	
CaO	4.590	4.94	4.489	4.73	4.446	4.84	4.303	4.73
CdO	0.012		0.015		0.016		0.020	
Cr_2O_3	0.032	0.031	0.039	0.053	0.042	0.142	0.053	0.067
CuO	0.008	0.027	0.009		0.010		0.013	
Fe_2O_3	10.412	10.3	10.041	9.98	9.882	10.0	9.353	9.78
Gd_2O_3	0.005		0.006		0.006		0.008	
K_2O	1.130	1.12	1.393	1.45	1.506	1.45	1.883	1.84
Li_2O	2.924	2.95	2.804	2.77	2.752	2.75	2.580	2.58
MgO	1.546	1.55	1.498	1.50	1.478	1.49	1.410	1.44
MnO	0.122	0.12	0.150	0.15	0.162	0.16	0.203	0.206
MoO_3	0.003		0.004		0.004		0.005	
Na_2O	7.787	7.60	9.603	9.52	10.382	10.6	12.978	12.4
NiO	0.014	0.014	0.017	0.027	0.018	0.020	0.023	0.024
P_2O_5	0.119	0.168	0.146	0.189	0.158	0.210	0.198	0.245
PbO	0.036	0.035	0.044	0.072	0.048	0.048	0.060	0.063
RuO_2	0.002		0.002		0.002		0.003	
SO_3	0.683	0.654	0.842	0.734	0.910	0.734	1.138	1.084
SiO_2	49.572	48.6	47.531	45.8	46.656	42.4	43.740	45.6
V_2O_5	4.159	3.81	3.990	3.57	3.918	3.59	3.678	3.40
ZnO	0.002		0.002		0.002		0.003	
ZrO_2	2.076	2.01	1.990	1.90	1.954	2.00	1.833	1.78
Cl	0.131		0.161		0.174		0.218	
F	0.110		0.135		0.146		0.183	
Total	*100.00*	*97.8*	*100.00*	*96.9*	*100.00*	*95.3*	*100.00*	*100.6*

(a) SBW-24 was not measured.

	SBW-26				**SBW-27**			
WL (mass%)	**18.5**		**25**		**18.5**		**25**	
Oxide	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**	**Target**	**Meas.**
Al_2O_3	5.091	5.31	6.880	7.16	5.091	5.41	6.880	7.18
B_2O_3	4.979	5.07	4.610	4.64	4.979	4.78	4.610	4.68
BaO	0.002		0.003		0.002		0.003	
CaO	11.963	12.07	11.180	11.8	9.974	10.8	9.350	9.87
CdO	0.015		0.020		0.015		0.020	
Cr_2O_3	0.039	0.028	0.053	0.043	0.039	0.045	0.053	0.062
CuO	0.009		0.013		0.009		0.013	
Fe_2O_3	1.500	1.50	1.493	1.47	1.500	1.52	1.493	1.45
Gd_2O_3	0.006		0.008		0.006		0.008	
K_2O	1.393	1.29	1.883	1.84	1.393	1.30	1.883	1.85
Li_2O	2.869	2.67	2.640	2.29	3.325	3.08	3.060	2.84
MgO	1.498	1.55	1.410	1.45	1.498	1.58	1.410	1.45
MnO	0.150	0.145	0.203	0.185	0.150	0.140	0.203	0.181
MoO_3	0.004		0.005		0.004		0.005	
Na_2O	11.152	11.0	14.403	14.0	11.152	11.2	14.403	14.2
NiO	0.017		0.023		0.017		0.023	
P_2O_5	0.146		0.198		0.146		0.198	
PbO	0.044	0.040	0.060	0.056	0.044	0.047	0.060	0.054
RuO_2	0.002		0.003		0.002		0.003	
SO_3	0.842	0.754	1.138	1.08	0.842	0.782	1.138	1.05
SiO_2	51.973	53.1	47.828	48.4	51.516	53.0	47.408	48.1
V_2O_5	3.990	4.01	3.678	3.65	5.979	6.10	5.508	5.43
ZnO	0.002		0.003		0.002		0.003	
ZrO_2	1.990	1.86	1.833	1.71	1.990	1.88	1.833	1.69
Cl	0.161		0.218		0.161		0.218	
F	0.135		0.183		0.135		0.183	
Total	*100.00*	*101.0*	*100.00*	*99.0*	*100.00*	*101*	*100.00*	*100.0*

Appendix B

Visual Observations of SBW Glasses

Appendix B: Visual Observations of SBW Glasses

Glass	WL	1st	2nd	CCC HT
SBW-11	18.5	Clean, medium brown glass, transparent	Clean, medium brown glass, transparent	Clean
	30	Clean, medium brown glass, transparent	Clean, medium brown glass, transparent	Clean
SBW-12	18.5	Clean, medium brown glass, transparent	Clean, medium brown glass, transparent	Clean
SBW-13	18.5	Light green, transparent, a few pits on surface	Clean, light/medium green glass, transparent	Clean
SBW-14	18.5	Clean, medium brown glass, transparent, possible undissolved material	Patty primarily clean, two light brown swirls on surface	Clean
SBW-15	18.5	Clean, medium brown glass, transparent	Clean, medium/dark brown glass, transparent	Clean
SBW-16	18.5	Clean, purplish/brown glass, transparent	Clean, purplish/brown glass, transparent	Clean
SBW-17	18.5	Film across pour patty surface, yellow spots on bottom of pour patty, salt ring on crucible wall above melt line, poured relatively slow	Dark brown, transparent glass, no salt observed	Clean
SBW-18	18.5	Film across pour patty surface, yellow spots on bottom of pour patty, salt ring on crucible wall above melt line, poured relatively slow	Dark brown, transparent glass, no salt observed	Clean
SBW-19	18.5	Film across top and bottom of pour patty surfaces, salt layer observed on crucible wall above melt line, poured relatively slow	Dark brown, transparent glass, no salt observed	Clean
SBW-20	18.5	Film across most of pour patty surfaces (top and bottom), salt observed on crucible walls above melt line, poured relatively slow	Dark brown, transparent glass, no salt observed	Clean
SBW-21	18.5	Film across top surface of pour patty, bottom of pour patty approx ½ covered, salt observed on crucible wall above melt line, poured relatively slow	Dark brown, transparent glass, no salt observed	Clean
SBW-22	18.5	Clean, light brown, transparent, a few swirls in bulk glass	Clean, light brown (perhaps green tint), transparent glass	Transparent, 2 – 3 swirls in bulk glass
	15	Clean, light green, transparent glass	Clean	Clean
	20	Clean, light green, transparent glass	Clean	Clean
	25	Clean, medium brown, transparent glass	Clean	Clean
	15	Light green/brown glass, transparent, 2 – 3 light brown swirls	Medium green color, transparent, clean	Light green glass, transparent, a few

Glass	WL	1st	2nd	CCC HT
		possible in bulk		swirls in bulk glass
	18.5	Light green/brown glass, transparent, a few swirls in bulk – consistent with previous melt	Medium green color, transparent, a few dark brown streaks in bulk glass	Light green glass, transparent, a few swirls in bulk glass
	20	Light brown, transparent, a few brown swirls	Medium green glass, transparent, light brown swirls in bulk glass	Medium green glass, transparent, a few swirls in bulk glass
	25	Medium brown glass, transparent, swirls in bulk, a few spots (islands) of dull / film on surface, salt film observed above melt line on crucible walls	Dark brown glass, shiny, no visible signs of salt in patty or residual crucible glass	Dark brown shiny glass, swirls in bulk
SBW-23	18.5	Clean, medium brown glass, transparent	Clean, medium brown/green glass, transparent	Transparent, 2 – 3 swirls in bulk glass
	15	Clean	Clean	Clean
	20	Clean	Clean	Clean
	25	Clean	Clean	Clean
SBW-24	18.5	Light brown, film covered both the pour patty surface and ½ of the bottom, salt observed above melt line on crucible wall	Clean, medium brown, transparent glass, residual crucible glass had a few brown streaks	Transparent, 2 – 3 swirls in bulk glass
SBW-25	18.5	Clean, dark brown glass	Clean, dark brown, shiny glass, a few pits on surface possible indicating a high viscosity	Dark brown, glass, appears clean, no swirls or crystallization
	15	Clean	Clean	Clean
	20	Clean	Clean	Clean
	25	Clean	Clean	Clean
SBW-26	18.5	Medium brown glass, transparent, a few darker brown swirls in bulk	Clean	Clean
	25	Dark brown glass, semi-transparent, film observed across surface of pour patty, salt observed above melt line	Clean	Clean
SBW-27	18.5	Medium brown glass, transparent, a few swirls in bulk	Clean	Clean
	25	Dark brown glass, semi-transparent, film covered portion of surface, salt observed above melt line	Clean	Clean

Appendix C

Viscosity Raw Data

Appendix C: Viscosity Raw Data

(Viscosity data presented in Poise; to convert to Pa-s divide by 10)

SBW-11@18.5		**SBW-15@18.5**	
Temp (°C)	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**
1150	39.20	1158.5	28.73
1095.5	57.76	1090	46.86
1043	87.05	1050	63.15
1150.5	38.80	1151	30.3
1206	26.04	1203.5	21.14
1151.1	38.99	1153	30.38

SBW-17@18.5		**SBW-20@18.5**		**SBW-21@18.5**	
Temp (°C)	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**
1150	163.92	1154.5	53.52	1148.5	69.86
1089	255.15	1100.5	84.35	1090	114.77
1046	373.96	1046	124.76	1052.5	139.43
1147	158.39	1150.5	55.19	1147	69.5
1199.5	105.22	1201	37.5	1201	46.3
1150	157.2	1150	55.79	1152	67.49

SBW-22@15		**SBW-22@18.5**		**SBW-22@20**		**SBW-22@25**	
Temp (°C)	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**
1150	82.55	1151.5	67.11	1154	62.01	1145.5	53.03
1094.5	133.66	1098.5	106.81	1098	100.98	1091.5	83.95
1048	211.84	1050	170.74	1052	157.24	1044.5	131.53
1152	80.02	1152	67.19	1151.5	64.14	1151	51.03
1200.5	54.14	1201	45.33	1207	41.45	1201	34.7
1151	81.04	1150	68.23	1150	65.17	1150	52.04

SBW-23@18.5	
Temp (°C)	**Viscosity (P)**
1150	56.38
1099	86.22
1043	137.90
1149.5	56.20
1204	37.45
1151	56.56

SBW-25@15		**SBW-25@18.5**		**SBW-25@20**		**SBW-25@25**	
Temp (°C)	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**
1153.5	76.12	1145	65.21	1151.1	55.96	1150.5	42.06
1098.5	130.01	1094.5	105.07	1094.5	93.45	1097	63.99
1051.5	209.08	1045.5	173.41	1043.5	154.89	1037	109.18
1151	78.08	1150	62.9	1150	56.78	1152	42.62
1202.5	51.48	1207	39.33	1216	34.96	1208	27.46
1152.5	77.93	1151.5	62.07			1149.5	44.15

SBW-26@18.5		**SBW-27@18.5**	
Temp (°C)	**Viscosity (P)**	**Temp (°C)**	**Viscosity (P)**
1149.5	65.78	1151	63.86
1082	128.04	1096	105.61
1047.5	187.38	1047.5	170.5
1151	65.05	1149	62.73
1202.5	42.60	1200	41.09
1150	65.07	1148	63.55

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