Immobilization Technology Section
Savannah River Technology Center
Westinghouse Savannah River Company

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Slurry Fed Melt Rate Furnace Runs to Support Glass Formulation Development for INEEL Sodium-Bearing Waste

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SAVANNAH RIVER SITE

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Approvals

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Summary

The Savannah River Technology Center (SRTC) in conjunction with the Pacific Northwest National Laboratory (PNNL) is developing frit compositions and adjusting waste loading to minimize the potential of forming a sulfate salt during melter processing. The overall objective of the glass formulation effort is to retain all of the sulfur from the melter feed into the glass. The objective of retaining all of the sulfur in the glass is being driven by the baseline flowsheet assumption that sulfur partitioned to the offgas will be recycled back to the melter. Therefore, there is no incentive to intentionally or unintentionally drive sulfur to the offgas via glass formulation techniques or melter operations. Pilot scale tests will be conducted in the Research Scale Melter (RSM) and the EV-16 melter systems. However, it is desirable to evaluate various glass formulations (or melter feeds) for their potential to form salt layers prior to the pilot scale tests. The Slurry-fed Melt Rate Furnace (SMRF) at the Clemson Environmental Technology Laboratory (CETL) is available to conduct scoping tests to assess different melter feeds with respect to the potential for form and/or accumulate salt layers.

A series of four runs were conducted in the SMRF to evaluate the potential for salt formation with different frit compositions and various amounts of SBW. Samples of the cold cap, dip samples and glass pour stream samples were analyzed to assess sulfur solubility and/or partitioning.

Using melter feed from the EV-16 FY01 run (based on frit SBW-9 at 30% waste loading), Run 1 resulted in a glass pool with a salt layer similar to that observed in the EV-16 FY01 run. Observation of the salt layer during this run indicated that the SMRF may provide a melter that produces similar results using less resources.

The primary objective for Run 2 (SBW-9 frit at 18.2% waste loading) was to provide a compositional transition to the 2001 WM-180 composition. However, the absence of a salt layer during this run was an indication that the sulfur content in the glass produced in the EV-16 run was at or near the solubility limit for sulfur in that particular glass composition.

Run 3 evaluated the SBW-22 frit at 18.2% waste loading prior to recommendation for use in the FY01 4Q RSM run at PNNL. No evidence of sulfur or salt accumulation was evident in the cold cap sample analysis or by visual observation after cold cap burn off.

Run 4 demonstrated that the SMRF could be used for an extended campaign. It was also shown that SBW-22 glasses at 20 and 22 wt.% waste loading did not accumulate a salt layer. Glass pool samples taken after the cold cap was allowed to burn off were free of salts for Runs 2, 3, and 4.

In addition to the demonstration of the ability of the SMRF to evaluate sulfate formation, insight into the effect of glass viscosity on melt rate can also be obtained. The SMRF can currently be used to evaluate the effect of the form of the glass formers on melt rate, redox and sulfur retention. There is also the potential to outfit the SMRF to measure off-gas speciation.

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Glossary

CETL	Clemson Environmental Technologies Laboratory
CSM	Centimeter Scale Melter
HLW	High-level waste
ICP-AES	Inductively coupled plasma atomic emission spectroscopy
INEEL	Idaho National Environmental and Engineering Laboratory
ITS-ML	Immobilization Technology Section – Mobile Lab
PNNL	Pacific Northwest National Laboratory
RCRA	Resource Conservation and Recovery act
RSM	Research Scale Melter
SBW	Sodium-bearing waste
SMRF	Slurry fed melt rate furnace
SRTC	Savannah River Technology Center
XRD	X-ray diffraction

Acknowledgments

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Introduction

The Idaho National Engineering and Environmental Laboratory (INEEL) is currently storing approximately one million gallons of sodium-bearing waste (SBW). SBW is a liquid radioactive waste produced from the second and third cycles of spent nuclear fuel reprocessing, waste calcination, and decontamination of High Level Waste (HLW) facilities. The SBW is an aqueous waste stream with proportionally high levels of sodium and potassium nitrates. The waste also contains approximately 0.07 M sulfur.

Currently, the waste loading of glass formulations being considered to immobilize the SBW is limited by the sulfur solubility in the glass. Historically, sulfur solubility in silicate glasses is governed by several factors: the sulfide/sulfate ratio (redox); the melt temperature; and the mol fraction of base (e.g. CaO, MgO and FeO)[1]. Exceeding the sulfur solubility of a glass leads to either the release of sulfur gasses (e.g., SO_2) or the dealkalinization of the glass through the formation of alkali sulfate salts (e.g., Na_2SO_4).

Bench[2,3] and pilot[4] scale tests have been conducted using simulated SBW. A discrete layer of sulfate salts on the surface of the melt pool was observed during the pilot scale test with SBW-9. The formation of sulfate salts was not observed during the bench scale tests using either batch chemicals or liquid feeds for the recommended glass formulation including frit and waste loading. A recent sample of the SBW has led to an updated estimate with a 40% increase of sulfur determined to be present[5]. In addition, the recommended offgas flowsheet recycles most of the sulfur that is partitioned to the offgas back to the waste stream. The additional sulfur estimated in the waste stream coupled with the incentive to retain all of the sulfur in the glass has initiated further research in the area of glass formulation. Specific areas of interest include the identification of components that increase sulfur solubility in glass, developing a fundamental understanding of sulfur partitioning as a function of various melter parameters or feed chemistry changes, and linking the observations of the bench scale tests to the large melter systems. One of the objectives of this report is to provide data that will form the technical basis from which bench scale tests can be linked to the larger melters. The methodology established could lower risks of recommending glass formulations for melter runs in which observations are inconsistent with those observed in the laboratory.

The Savannah River Technology Center (SRTC) in conjunction with the Pacific Northwest National Laboratory (PNNL) is developing alternative frit compositions and adjusting waste loading to eliminate salt accumulation while incorporating all of the sulfur from the melter feed into the glass and recycling the sulfur in the offgas back to the waste stream[2]. Pilot scale tests will be conducted in the Research Scale Melter (RSM) and the EV-16* melter systems; however, it is desirable to assess the recommended formulation on a scale larger the crucible scale used to develop the formulation for the formation of sulfate salts in the suggested flowsheets prior to the pilot scale tests. The Slurry-fed Melt Rate Furnace (SMRF) at the Clemson Environmental Technology Laboratory (CETL) is available to conduct scoping tests to assess different melter feeds with respect to the potential for form and/or accumulate salt layers.

The objectives of the tests performed on the SMRF were:

1) Run the same feed† used in the EV-16 to evaluate the SMRF as a smaller scale link to behavior in the EV-16 insofar as salt accumulation. A link between the SMRF and EV-16 would lower the risk of recommending frits for SBW vitrification.
2) Run the SBW-9 frit with a lower waste loading and the new simulant composition in Reference 5. This run will transition the SMRF glass inventory to both the new simulant and the lower waste loading recommended for Frit SB-22.
3) Run the SBW-22 frit with the simulant from Reference 5 at 18.2% waste loading. This represents 0.8% SO_4, the amount of sulfate measures in the glass produced in the EV-16.
4) Evaluate the relative melt rates for the different frit/simulant/waste loading tested.

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* Joule heated melter, model EV-16 EnVitCo, Toledo, OH

† SBW-9 with the 1999 SBW simulant (melter feed used in Reference 4).

Discussion

Simulant Composition

Two SBW simulant compositions were used during the testing: the original simulant (referred to as the 1999 simulant) remaining from the 2Q01 EV-16[4] run and a revised simulant (referred to as the 2001 simulant) developed from a more recent analysis of SBW[5,6]. The 2001 simulant has a sulfur content approximately 40% higher than the 1999 SBW simulant, but is otherwise similar. The 2001 simulant composition has been simplified for the tests to eliminate components making up less than 0.25% (oxide basis) of the waste stream, as documented in SRT-GDP-2001-00052*. Elimination of these trace components removed RCRA metals (including lead) from the simulant. The 2001 simulant composition was normalized to make up for the removed components. The required makeup chemicals for each of the simulants are shown in Table 1. Enough 2001 simulant was produced to provide an unvarying source for the planned experiments[7]. Subsequent Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) analysis of the simulant indicated that the elemental sulfur content was approximately ten percent greater than targeted. Table 2 is the analytical results comparing the targeted and measured values for components in the 2001 simulant.

Table 1. Reagent Chemical Used for the Revised 2001 Simulant Makeup.

Compound	2001 Simulant (Target)[5]	2001 Simulant (Revised)	Units
Cadmium Nitrate Tetrahydrate	0.220	-	gram/liter
Calcium Nitrate Tetrahydrate	10.7	11.1	gram/liter
Cerium Nitrate Hexahydrate	0.020	-	gram/liter
Chromium Nitrate Pentahydrate	1.43	-	gram/liter
Copper Nitrate Trihydrate	0.159	-	gram/liter
Cesium Nitrate	0.265	-	gram/liter
Gadolinium Nitrate Hexahydrate	0.076	-	gram/liter
Ferric Nitrate Nonahydrate	8.28	8.78	gram/liter
Lead Nitrate	0.408	-	gram/liter
Magnesium Nitrate Hexahydrate	2.91	3.09	gram/liter
Nickel Nitrate Hexahydrate	0.404	-	gram/liter
Potassium Nitrate	18.7	19.8	gram/liter
Sodium Nitrate	165	175	gram/liter
Strontium Nitrate	0.024	-	gram/liter
Zinc Nitrate Hexahydrate	0.295	-	gram/liter
Potassium Iodide	0.021	-	gram/liter
Boro-Fluoric Acid (48 wt.% solution)	2.11	-	gram/liter
Boric Acid	0.010	0.761	gram/liter
Manganese Nitrate (50 wt.% solution)	4.76	5.05	gram/liter
Aluminum Nitrate Nonahydrate (2.2M solution)	286	301	ml/liter
Sulfuric Acid (18M)	2.84*	3.88*	ml/liter
Hydrofluoric Acid (28.9M)	-	1.18	ml/Liter
Hydrochloric Acid (12M)	2.34	2.45	ml/liter
Nitric Acid (15.4M)	56.7	51.75	ml/liter
Molybdenate Dinitrate (0.1M solution)	1.81	-	ml/liter
Ruthenium Chloride	0.024	-	gram/liter
Phosphoric Acid (14.6M)	0.884	0.938	ml/liter

*Sulfur difference represents the ~40% change in sulfur content of SBW.

* M.E. Stone, "Simulant for Scoping Sulfate Tests with Alternative Frits for INEEL SBW Vitrification (U)," SRT-GDP-2001-00052, 6/2001.

Table 2. Analytical Results for 2001 Simulant (Revised) Used in the Slurry Fed Melt Rate Furnace.

Element	Sample Result	Sample Result	Target	Difference
	mg/L	Molar	Molar	Δ%
Al	15400	0.570	0.663	-14.0%
B	143	0.013	0.012	8.3%
Ca	1930	0.048	0.047	2.1%
Fe	1110	0.020	0.022	-9.1%
Mg	192	0.008	0.012	-33.3%
Mn	651	0.012	0.014	-14.3%
K	7480	0.191	0.196	-2.6%
Na	47000	2.04	2.06	-1.0%
Cl	Not measured	Not measured	0.030	-
F	Not measured	Not measured	0.047	-
P	414	0.013	0.014	-7.1%
S	2450	0.076	0.069	10.1%

Melter Feed Preparation

Melter feed was prepared by adding the glass formers as batch chemicals to the simulated waste along with sugar, as shown in Table 3. Runs 1-3 were made solely of one batch of feed (note that the composition of the feeds differed but the use of one feed batch per run minimized batch to batch compositional variations). For Run 1, the 1999 simulant remaining from the EV-16 run already contained the glass formers for Frit SBW-9 at 30% loading, therefore only the sugar was added during this testing to conform to the conditions run in the EV-16[4]. Due to the required run time, Runs 4a and 4b required several batches of feed (target composition constant for each run – SBW-22 frit with the 2001 simulant) to complete the duration of the runs. The Run Plans for the four tests are in Appendix A.

Table 3. Composition of the Feeds Used in the Slurry Fed Melt Rate Furnace.

Run	**1**	**2**	**3**	**4a**	**4b**
Simulant	1999	2001Revised	2001Revised	2001Revised	2001Revised
Frit	SBW-9	SBW-9	SBW-22	SBW-22	SBW-22
Loading (wt.% oxide)	30	18.2	18.2	20	22
For Each Batch					
Simulant	18.9 L	18.9 L	18.9 L	18.9 L	18.9 L
Silica	184.1 g/L	354.6 g/L	370.7	329.9	292.4
Sodium Hydroxide	-	-	30.20 g/L	26.87 g/L	23.82 g/L
Lithium Hydroxide	39.82 g/L	68.26 g/L	93.72 g/L	83.41 g/L	73.93 g/L
Calcium Hydroxide	18.70 g/L	32.06 g/L	36.17 g/L	32.19 g/L	28.53 g/L
Boric Acid	75.44 g/L	129.32 g/L	58.41 g/L	51.99 g/L	46.08 g/L
Magnesium Oxide	-	-	9.546 g/L	8.496 g/L	7.530 g/L
Ferric Oxide	28.319 g/L	48.547 g/L	8.291 g/L	7.379 g/L	6.541 g/L
Vanadium Oxide	-	-	26.62 g/L	23.69 g/L	21.00 g/L
Zirconia	-	-	13.31 g/L	11.85 g/L	10.50 g/L
Water Additions	-	300 g	300 g	200 g	-
Nitric Acid Additions	-	-	19.45 g	86.4 g	86.4 g
Sugar Addition	160 g/L	160 g/L	135 g/L	165 g/L	165 g/L

During the makeup of melter feed for Runs 2-4, the addition of calcium hydroxide to the 2001 simulant immediately caused large agglomerates to form in the feed. Continued agitation with an impeller mixer initially broke the agglomerates down. However, as stirring continued, the melter feed then began to set in a way comparable to cement. Dilution water, nitric acid or both were added to the batch to decrease the viscosity (see Table 3 for specifics regarding quantities added). It was also noted that the sugar addition also reduced the viscosity of the feed. A high-shear homogenizer was then used to break up any remaining agglomerates. The melter feed thickened noticeably as the agglomerates were dispersed. The temperature of the melter feed increased as the hydroxides and sugar were mixed with the nitric acid. The slurry thinned considerably as it cooled to room temperature but remained pumpable. To maintain the targeted redox ratio of the glass, sugar additions were used to offset the nitric acid additions used to reduce the viscosity of the melter feed (33g sugar/mol nitrate as agreed upon by PNNL and INEEL researchers).

During the makeup of two of the batches (Batches 3 and 4) for Run 4 (a and b), solids buildup on the bottom of the makeup vessel was noted. The impact on the Run 4 (a and b) is difficult to determine, however the analytical results of sulfate concentration for these batches match the results from earlier and later batches.

Test Equipment

Melt Rate Furnace

The SMRF was designed by D.F. Bickford of SRTC and fabricated at CETL. The furnace has two chambers. As shown in Figure 1, the lower chamber is heated by three SiC heating elements and the upper chamber is heated by four plate heaters. An eight-inch 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 for insertion of 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.

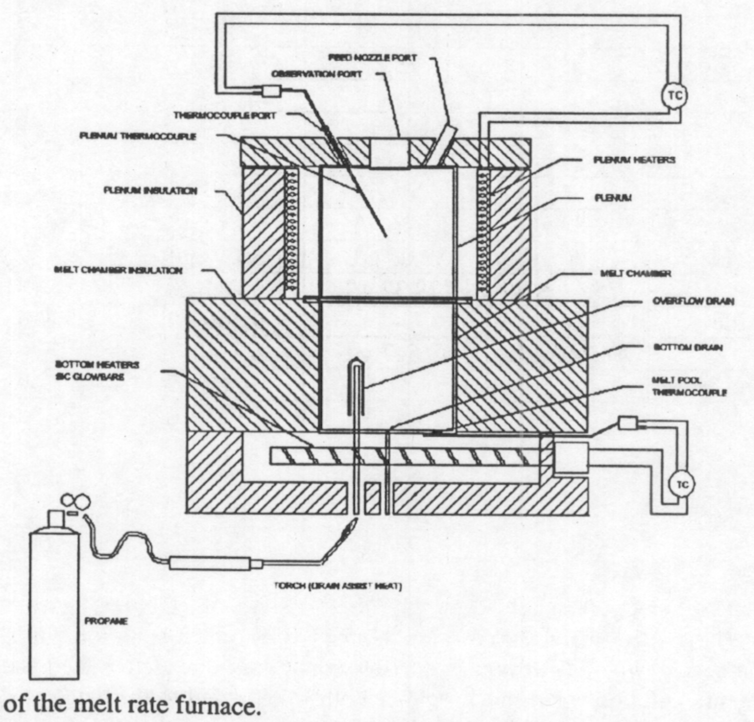

Figure 1. Schematic of the melt rate furnace.

Feed System

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 2. The pump is setup 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 –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 a heated tube the initiates pouring when the glass level overflows the height of the tube. A drain tube is also installed on the crucible to empty the vessel at the conclusion of a run, if desired. A weigh scale is used to record the amount of glass poured.

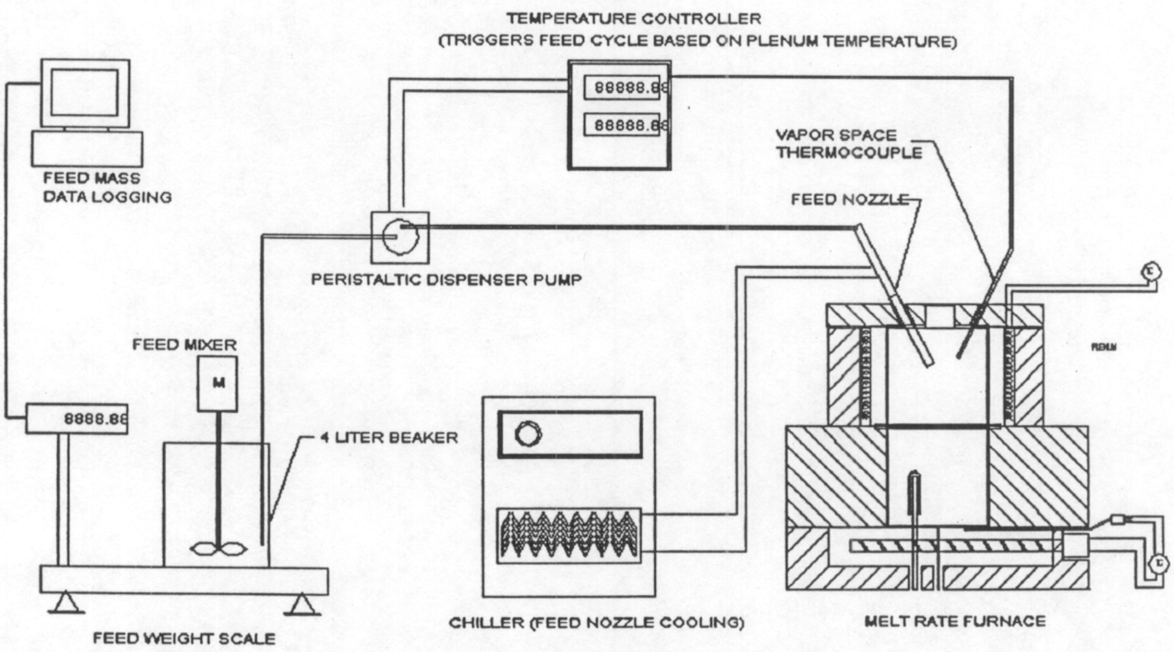

Figure 2. Schematic of the SMRF and feed system.

Experimental

Run Descriptions

Run 1 - SBW-9/1999 Simulant 30% Waste Loading

Run 1 was an 8-hour baseline run[8] with operating parameters as close as possible to the FY01, 2Q EV-16 run[4]. Table 4 is a summary of the operating conditions for Run 1. Melter feed remaining from the EV-16 run was used during the run. This feed utilized the original (1999) simulant and SBW-9 frit composition targeting a waste loading of 30%. Sugar (160 g/L) was added as the reductant. The set point plenum temperature was 750°C during the run. The feed pump speed, flow and pause times are initial settings and were varied throughout the run to maintain the plenum temperature and keep the feed tube clear. The feed pump speed must be sufficient to clear any partially dried feed in the feed tube to prevent blockage. The flow time must be brief enough to maintain plenum temperature.

Cold cap samples were taken four hours into the 8 hour run and at the end of the run. To obtain a dip sample (representative of the glass pool), at one point during the run, feeding was stopped to allow the cold cap to burn off and a dip sample was taken. Glass pour samples were taken four hours into the run and after the furnace had idled overnight at 900°C.

Table 4. Summary of Initial Operating Conditions Used in Run 1.

Parameter	Value	Units
Melter Temperature	1150	Celsius
Plenum Temperature	760	Celsius
Feed Pump Temperature Controller Setting	750	Celsius
Sugar Addition Amount	160	Grams/liter
Sulfur Content of Simulant	3.5	wt.% oxide
Frit Formulation	SBW-9	-
Waste Loading	30	wt.% oxide
Simulant Volume to be Made up	5 (18.9)	Gallons (L)
Feed Pump Setting	Single Dispense	-
Feed Pump Speed	20	percent
Feed Pump Flow Time	5	seconds
Feed Pump Pause Time	100	percent
Approximate Length of Run (@2.4 L/hr)	8	Hours

Run 2 - SBW-9/2001 Revised Simulant 18.2% Waste Loading

This run was performed to determine if additional salt would form if the waste loading was lowered to provide 0.8% SO_3 (the measured amount of SO_3 in the glass recovered from the EV-16 FY01 run). This glass composition was not designed or optimized. The composition was the result of a direct reduction in waste loading. Consequently, the glass cannot be expected to meet the processing limits that were achieved by the SBW-9 composition at a 30 wt.% waste loading. An additional purpose of this test is to transition the furnace to a glass with a lower sulfur content. The melter was not completely drained from the previous run, which was known to be saturated with SO_4. The melter feed for this run used the revised 2001 SBW simulant as described in Reference 7. This run was an 8-hour run with the revised simulant composition and SBW-9 frit composition at 18.2% loading[9]. Sugar was used as the reductant at 160 g/L. All operating parameters were kept the same as Run 1.

Run 3 - SBW-22/2001 Revised Simulant 18.2% Waste Loading

This run was an 8-hour run with the revised simulant composition and SBW-22 frit composition at 18.2% loading[10]. 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 revised 2001 SBW simulant, and as in Run 2, 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/ mol nitrate added as nitric acid. During the 8 h of furnace operation, over 15 kg of melter feed was 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. Table 5 is the operating conditions for Run 3.

Run 4 - SBW-22/2001 Revised Simulant 22% Waste Loading

This run was a 48-hour run with the revised 2001 SBW simulant composition and SBW-22 frit composition at 20% loading[11]. Approximately 33 hours 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 18.2 wt.%. The increase was an attempt to determine if the 18.2 wt.% waste loading was the maximum obtainable for this frit/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 the salt layer is stable, diminishing or expanding. A cold cap sample and a glass pour sample were taken every eight hours of operation. During this time, feeding was suspended for thirty minutes, and a dip sample was taken. Table 6 is a summary of the operating conditions used to start run 4 (a and b).

Table 5. Summary of Initial Operating Conditions Used in Run 3.

Parameter	Value	Units
Melter Temperature	1150	Celsius
Plenum Temperature	760	Celsius
Feed Pump Temperature Controller Setting	750	Celsius
Sugar Addition Amount	160	Grams/liter
Sulfur Content of Simulant (Target)	4.5	% oxide
Frit Formulation	SBW-22	
Waste Loading	18.2	% oxide
Simulant Volume to be Used	5 (18.9)	Gallons (L)
Feed Pump Setting	Single Dispense	
Feed Pump Speed	20	percent
Feed Pump Flow Time	5	seconds
Feed Pump Pause Time	100	percent
Approximate Length of Run (@2.4 L/hr)	8	Hours

Table 6. Summary of Initial Operating Conditions Used in Run 4.

Parameter	Value	Units
Melter Temperature	1150	Celsius
Plenum Temperature	760	Celsius
Feed Pump Temperature Controller Setting	750	Celsius
Nitric Acid Addition	60	ml/liter
Sugar Addition Amount	165	Grams/liter
Sulfur Content of Simulant (Target)	4.5	% oxide
Frit Formulation	SBW-22	
Waste Loading	20	% oxide
Approximate Simulant Volume to be Used	60	liters
Feed Pump Setting	Single Dispense	
Feed Pump Speed	20	percent
Feed Pump Flow Time	5	seconds
Feed Pump Pause Time	100	percent
Length of Run	48	Hours

Results

Run 1 - SBW-9/1999 Simulant 30% Waste Loading

Prior to the current runs, the SMRF had been run with the same feed that was used in Run 1. Figure 3 is a plot of the melter feeding operation. The plot shows the cumulative feed amount added to the furnace as well as the feed rate. The feed rate stabilizes approximately five hours into the run. This is a function of the amount of residual glass from the previous run . Over 17 kg of melter feed was added during the 9h 20min of processing. At the conclusion of the run, a discontinuous visible salt layer was present on the glass. The salt was concentrated near the crucible wall with small accumulations in the melt pool. This observation is consistent with the EV-16 melter run observations as reported by Perry et al.[4].

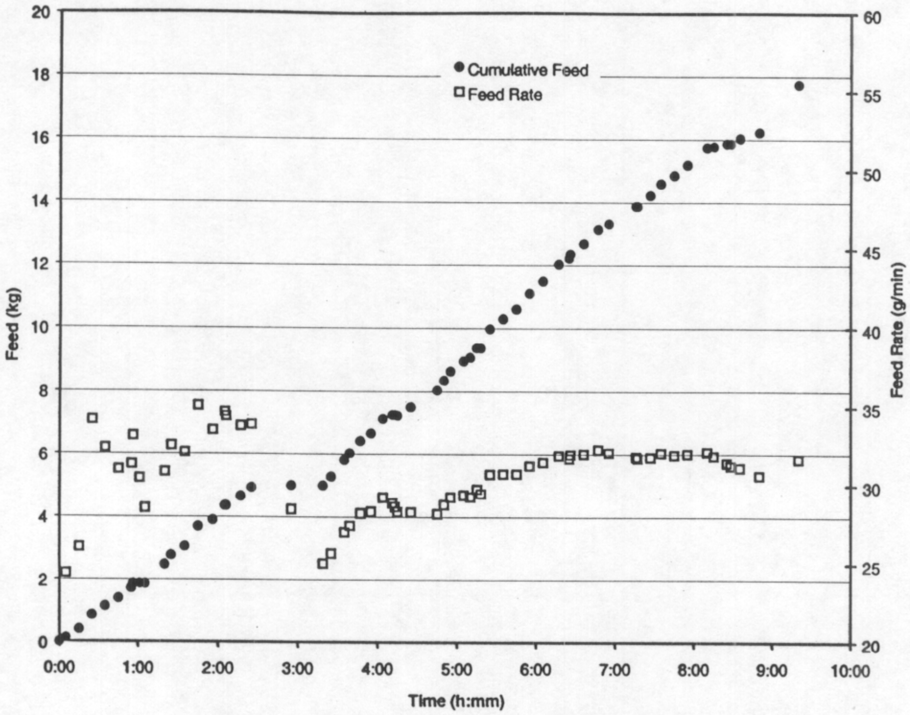

Figure 3. Melter feed processed in Run 1.

Table 7 contains the compositional analyses of the cold cap, dip, glass and salt samples from Run 1 as reported by the Immobilization Technology Section – Mobile Lab (ITS-ML). The composition of glass recovered from the EV-16 FY01 run is added for comparison. There does not appear to be a significant variation in the compositions of glass or cold cap samples taken throughout the run. The cold cap may be slightly enriched with silica with respect to glass poured. Samples taken from the melt pool (cold cap, dip, and salt layer) were chosen to deliberately maximize the amount of sulfur recovered. The glass pour sample taken ~4 h into the run had an SO_3 content of 0.95 wt.%. It should also be noted that the ITS-ML analyzed a sample of the residual salt layer. The results in Table 7 confirm that the salt layer is primarily composed of Na_2O and SO_3.

Table 7. Analyses of Samples from the SMRF during Run 1.

	Cold Cap	Cold Cap	Dip sample	Glass Pour	Dip sample	Salt Layer	Glass
	Mid-run	End of run	Mid-run	Mid-run	End of run	End of run	EV-16
Al_2O_3	8.16	8.26	7.92	8.44	8.11	0.06	9.58
B_2O_3	10.53	10.63	9.98	9.76	9.72	0.00	10.80
CaO	4.24	4.16	4.02	4.42	4.25	0.62	4.42
Fe_2O_3	6.78	6.78	6.48	7.48	7.15	0.02	7.58
K_2O	2.46	2.31	2.43	2.06	2.13	5.16	--
Li_2O	3.70	3.62	3.53	3.51	3.42	3.01	3.85
MgO	0.29	0.29	0.28	0.29	0.28	0.00	0.32
MnO	0.24	0.24	0.23	0.26	0.25	0.00	0.30
Na_2O	14.83	14.15	14.56	14.69	14.42	32.22	15.50
SO_3	3.05	1.74	3.70	0.95	1.17	53.52	0.77
SiO_2	41.71	43.85	43.64	46.85	47.92	0.52	43.90
ZrO_2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
V_2O_5	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Fe^{2+}/Fe^{tot}	--	--	--	0.1	0.1	--	--
Total	96.00	96.02	96.75	98.73	98.86	95.15	97.02

X-ray diffraction analysis of the cold cap and dip samples returned similar results. The crystalline portion of the cold cap and dip samples primarily consisted of sodium sulfate and quartz. Figure 4 is a representative XRD pattern for the cold cap and dip samples.

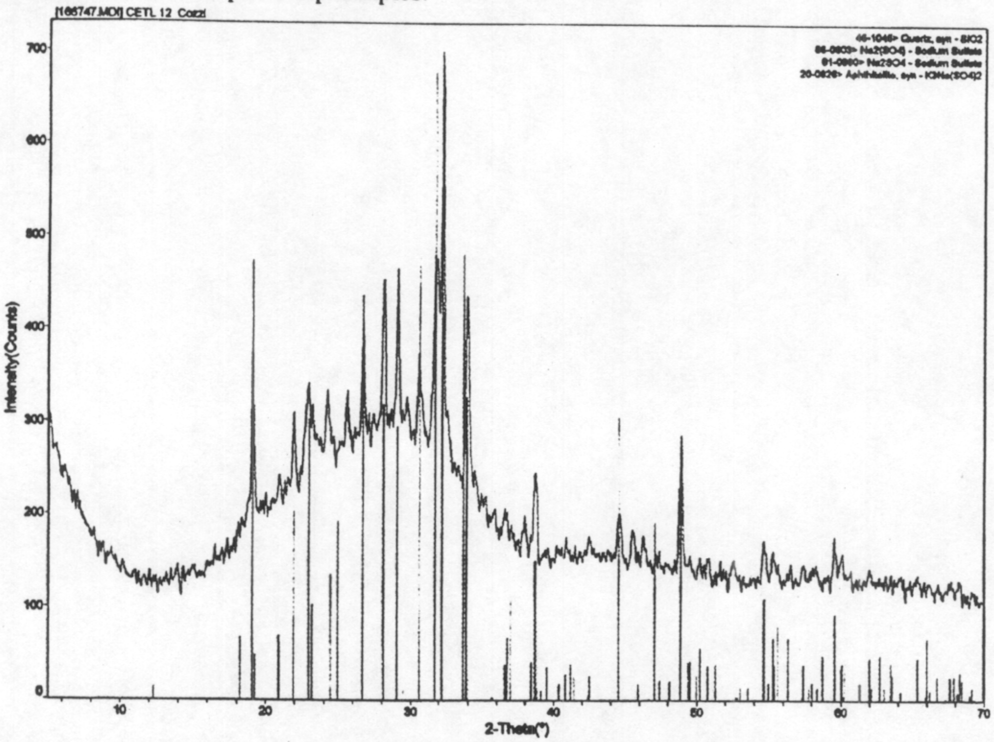

Figure 4. XRD pattern of cold cap sample taken after four hours of operation during Run 1. Pattern is typical of the cold cap and dip samples.

Run 2 - SBW-9/2001 Revised Simulant 18.2% Waste Loading

Run 2 began with a heel of approximately one inch of SBW-9/30wt.%/2000 SBW simulant glass in the furnace (see Table 7 for an estimate of the residual SBW-9/30% loading glass composition). This corresponds to slightly more than 720 grams of glass. Figure 5 is a plot of the melter feeding operation. The plot shows the cumulative feed amount added to the furnace as well as the feed rate. As in Run 1, the feed rate stabilized approximately five hours into the run. The plateau in the cumulative feed from 5:30-6:00 hours represents the suspension of feeding to allow the cold cap to burn off. Over 18 kg of melter feed was added during the 9h 15min of processing. At the conclusion of the run, no visible salt layer was present on the glass indicating that the reduction in waste loading is a possible mechanism to minimize the potential formation of a salt layer. The furnace was drained and idled at 500°C overnight in preparation for Run 3. No visible salt layer was detected after the 12-hour idle; however, patches of a thin haze were noticeable on the glass surface. A similar artifact was observed in the bench-scale work during the development of SBW-9[3], where it was determined that the haze was SO_3-based.

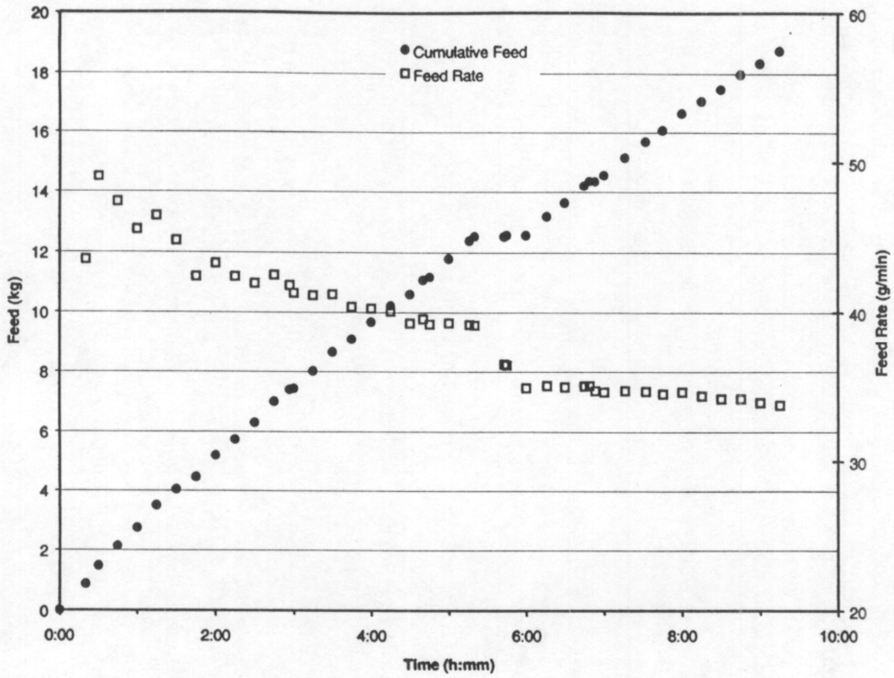

Figure 5. Melter feed processed in Run 2.

As shown in Table 8, the cold cap sample, dip sample, and glass pour sample were similar in composition. The Fe^{2+}/Fe^{tot} ratio for the glass sample taken at the end of the run was 0.3. Although the sugar to nitrate ratio was supposed to be the same as Run 1, the redox ratio measured in Run 1was 0.1 for both the glass pour sample and the dip sample taken at the conclusion of the run.

X-ray diffraction analysis of the cold cap sample indicates that silica is the only crystalline phase present. The presence of silica is consistent with batch to glass conversion process[12]. Figure 6 is the XRD pattern for the cold cap sample. Both XRD analysis and visual observation indicate that there is no evidence of any sulfates forming in the cold cap in the SBW-9/18.2% glass system.

Table 8. Analyses of Samples from the SMRF during Run 2.

	Cold Cap	Dip Sample	Glass Pour	SBW-9-18.2%
	Mid-run	After cold cap burn off	End of run	
Al_2O_3	4.40	5.39	5.69	5.01
B_2O_3	12.33	11.30	10.85	12.33
CaO	4.17	4.46	4.46	4.48
Fe_2O_3	8.01	8.41	8.04	8.44
K_2O	1.52	1.51	1.55	1.37
Li_2O	4.05	3.83	3.75	4.09
MgO	0.20	0.20	0.24	0.07
MnO	0.14	0.18	0.17	0.15
Na_2O	9.57	10.85	11.24	9.45
SO_3	1.03	0.88	0.77	0.83
SiO_2	51.98	51.98	52.62	53.17
ZrO_2	0.00	0.00	0.00	0.00
V_2O_5	0.00	0.00	0.00	0.00
Fe^{2+}/Fe^{tot}	--	--	0.3	--
Total	97.40	99.00	99.50	90.208

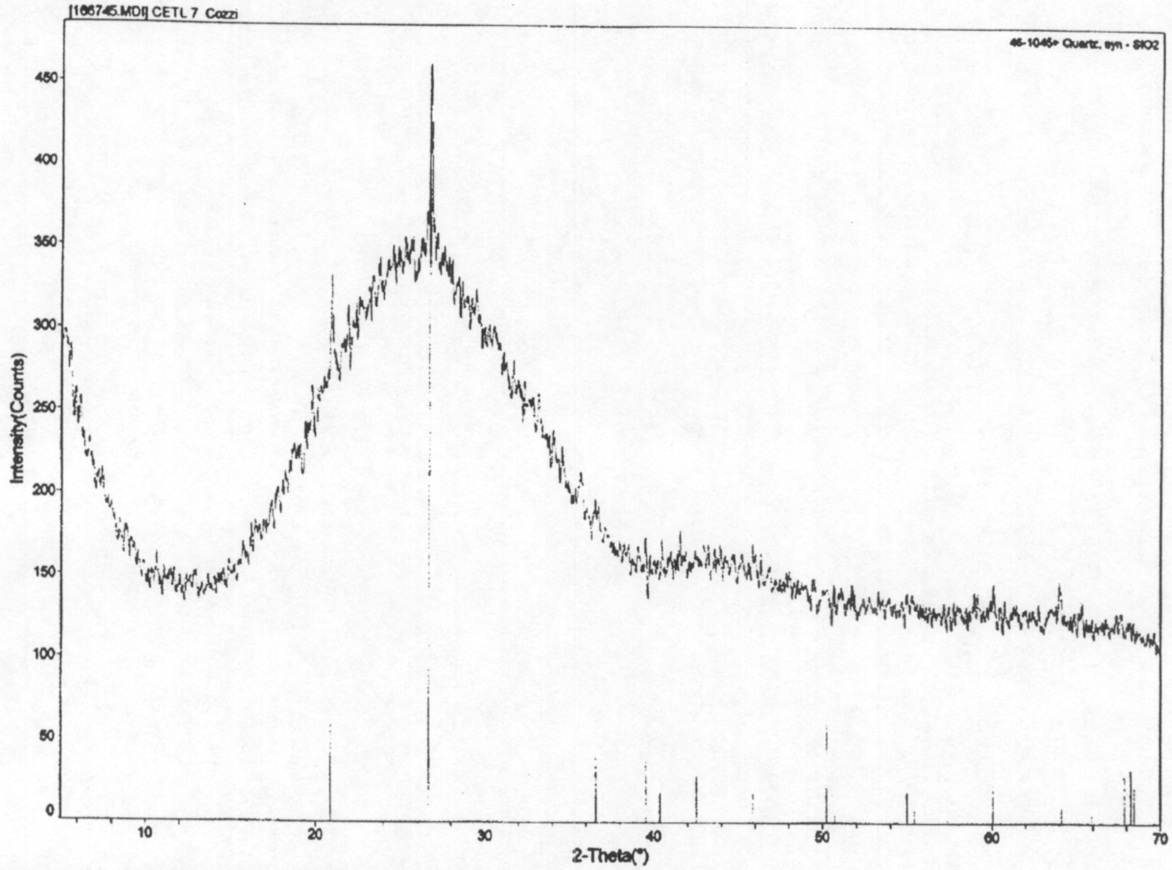

Figure 6. XRD pattern of cold cap sample taken during Run 2.

Run 3 - SBW-22/2001 Revised Simulant 18.2% Waste Loading

Table 9 is a plot of the melter feeding operation for Run 3. The plot shows the cumulative feed amount added to the furnace as well as the feed rate. The feed rate was consistent throughout the run. This can be attributed to the amount of heel remaining in the furnace from the previous run. The furnace contained almost three kilograms of glass from Run 2. The relatively large mass of glass in the furnace buffers the effect of the additions of melter feed. In Run 3, 14.9 kg of melter feed was fed to the furnace. This corresponds to ~4.2 kg of glass. The effect of the large heel of glass of a different composition is evident in
Table 9. The compositions of the samples taken from the glass pool (cold cap and dip samples) closely resemble the targeted composition, whereas the compositions of glass pour samples fall between the targeted Run 3 composition and the measured Run 2 composition, see Table 8 and
Table 9. As the waste loadings were adjusted to have identical quantities of sulfur, the measured sulfur composition remained constant throughout the transition from SBW-9 18.2% to SBW-22 18.2%. The Fe^{2+}/Fe^{tot} ratio for the glass samples taken throughout the run varied from 0.4 to 0.5.

As in Run 2, x-ray diffraction analysis of the cold cap sample indicates that silica is the only crystalline phase present. The presence of silica is consistent with batch to glass conversion process. Figure 8 is the XRD pattern for the cold cap sample. There was no evidence of any sulfates (or sulfur compounds or salts, in general) forming a second phase in the cold cap or observed on the surface of the glass during idling.

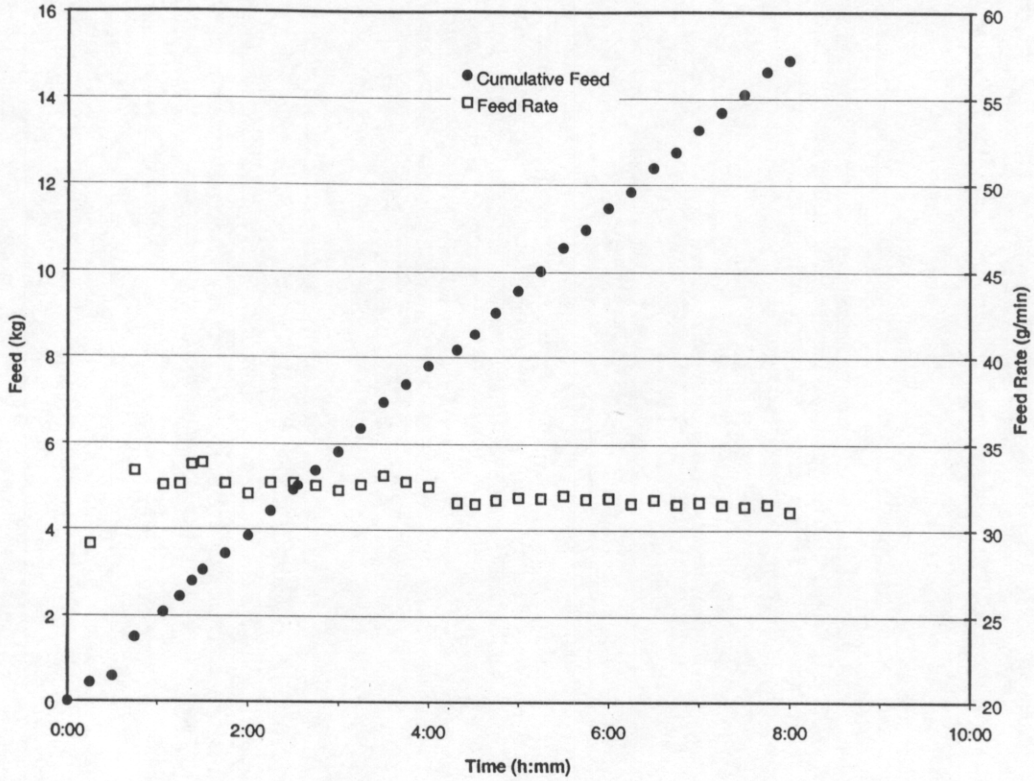

Figure 7. Melter feed processed in Run 3.

Table 9. Analyses of Samples from the SMRF during Run 3.

	Cold Cap	Cold Cap	Dip sample	Glass Pour	Glass Pour	Glass Pour	SBW-22-18.2%
	4 hr	End of run	End of run	2 hr 45 min	8 hr	End of run	Target
Al_2O_3	4.10	4.01	4.48	5.23	4.72	4.69	5.01
B_2O_3	5.67	5.25	6.70	10.92	7.57	7.79	5.00
CaO	4.17	4.14	4.53	4.35	4.39	4.44	4.50
Fe_2O_3	2.13	1.63	3.45	7.82	4.82	4.56	1.50
K_2O	1.46	1.46	1.23	1.45	1.33	1.36	1.37
Li_2O	5.06	5.25	4.46	3.98	4.18	4.46	5.00
MgO	1.41	1.51	1.30	0.33	0.99	1.04	1.50
MnO	0.13	0.12	0.14	0.17	0.15	0.14	0.15
Na_2O	11.92	12.23	11.34	10.88	11.40	11.59	12.96
SO_3	0.83	0.82	0.77	0.71	0.72	0.80	0.83
SiO_2	57.97	57.97	57.33	51.77	54.76	54.98	55.58
ZrO_2	1.78	1.95	2.14	0.21	1.51	1.62	2.00
V_2O_5	2.80	3.05	1.55	0.18	1.10	1.17	4.00
Fe^{2+}/Fe^{tot}	--	--	--	0.4	0.5	0.4	--
Total	99.42	99.38	99.39	97.99	97.65	98.64	99.40

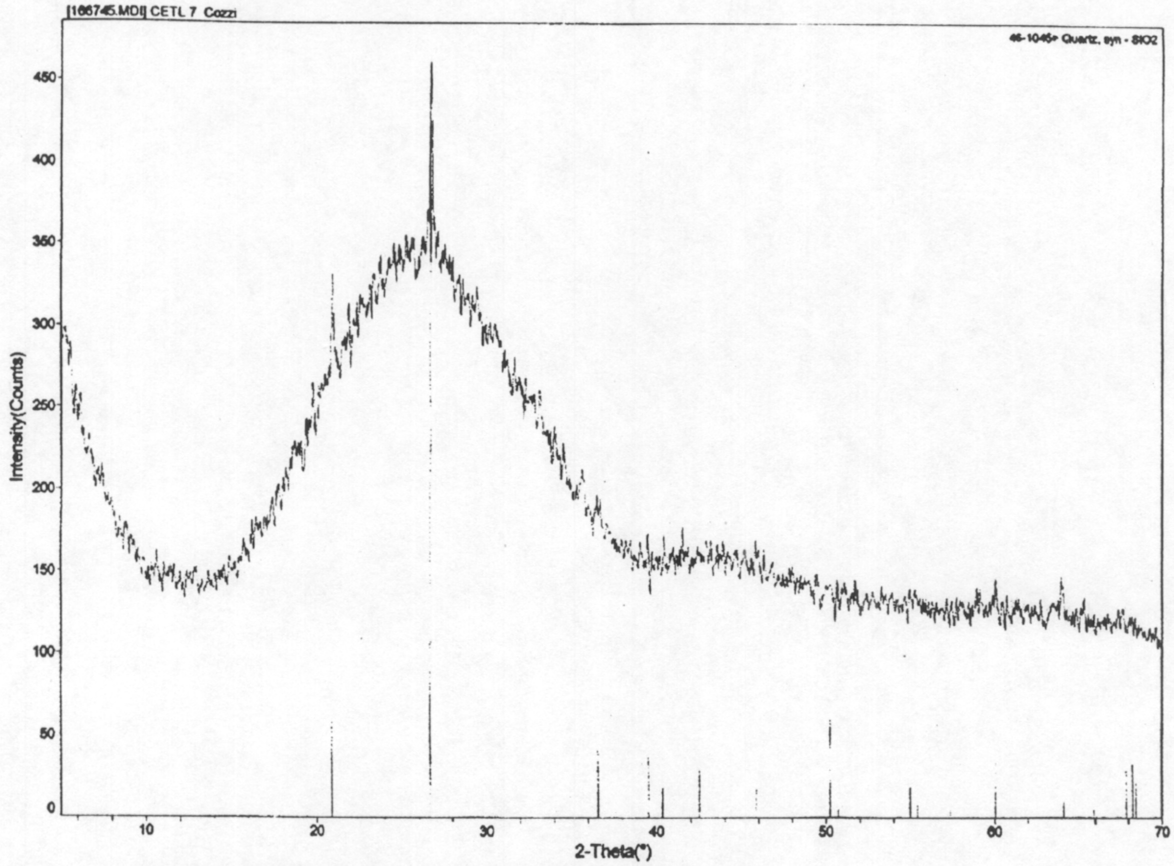

Figure 8. XRD pattern of cold cap sample taken during Run 3.

Run 4 - SBW-22/2001 Revised Simulant 22% Waste Loading

Figure 9 is a plot of the melter feeding operation for Run 4. The plot shows the cumulative feed amount added to the furnace as well as the feed rate. The heel in the furnace was the composition used in Run 1. Reductant testing using the Run 1 composition was performed between Run 3 and Run 4. Approximately five hours of operation was necessary to stabilize the feed rate. In Run 4, more than 90 kg of melter feed was fed to the furnace. This corresponds to more than 26 kg of glass. The circled areas in Figure 9 identify the sampling points where the cold cap was sampled, feeding was halted for thirty minutes, the glass pour stream was sampled, a dip sample was taken, and feeding was resumed. The cold cap and pour stream were also sampled at the conclusion of the run. Figure 10 shows the cumulative glass poured and pour rate during Run 4. Pouring was initiated after 3h 45min of operation. The onset of pouring is a function of the heel in the furnace and the feed rate. For the duration of the run, 19.8 kg of glass was poured. The amount of glass poured is less than the amount of glass (as melter feed) added to the melter because 3h and 45min of feeding was required before the glass level reached the pour spout. The pour rate stabilized at approximately twenty grams per minute after twenty-two hours of operation. Eight hours after the feed composition was changed to a higher waste loading, the pour rate began to increase. Figure 11 shows the effect of waste loading on viscosity in SBW-22 glasses. The difference in overall glass composition between 20 and 22 wt% waste loading is small, however, the effect on viscosity (pour rate) is significant. The change in glass composition in the furnace is evident in Table 10 and Table 11.

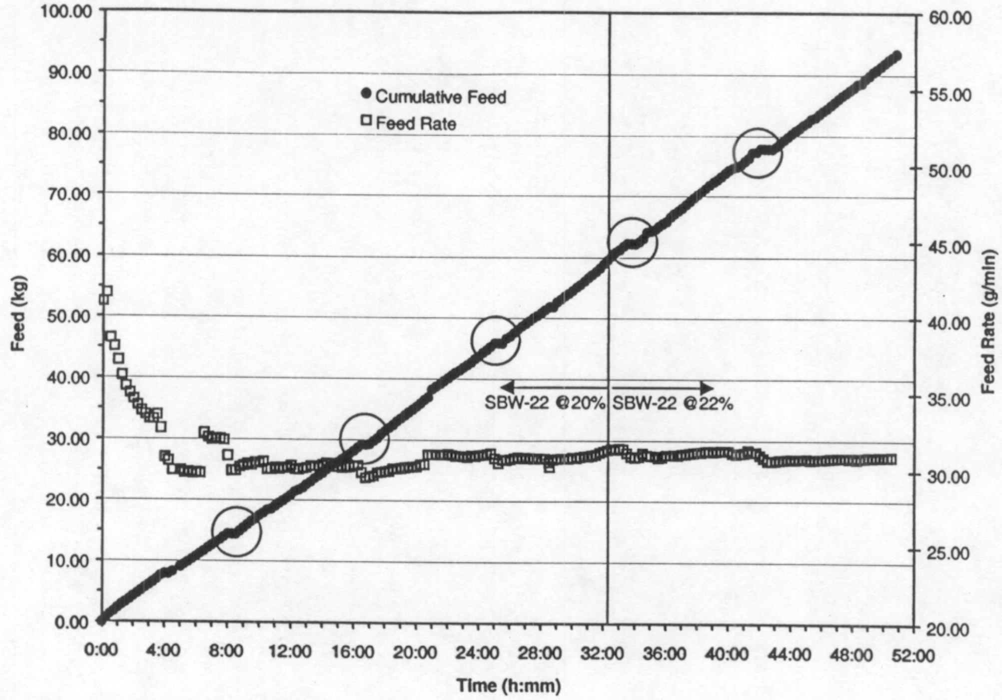

Figure 9. Melter feed processed in Run 4.

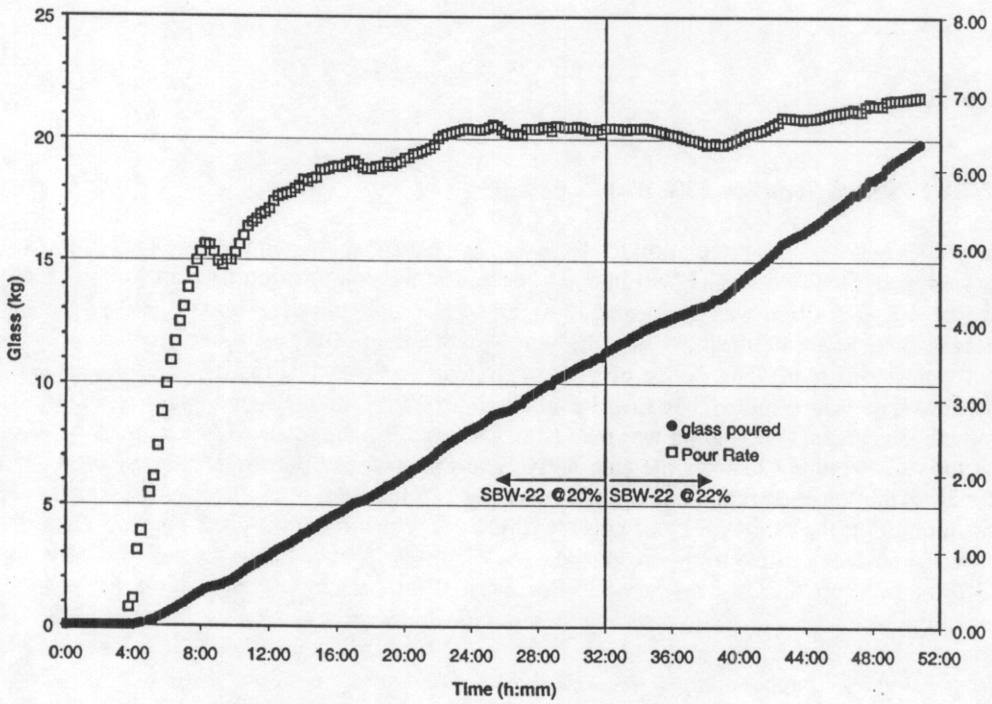

Figure 10. Glass produced in Run 4.

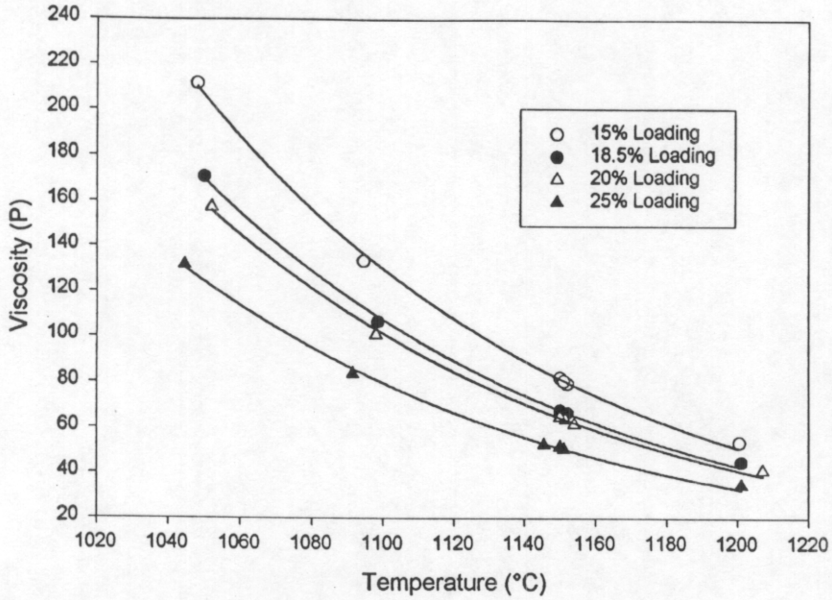

Figure 11. Viscosity of SBW-22 glasses as a function of waste loading.

Table 10 is the compositions of the samples taken when the SBW-22 20% composition was in use. The furnace had previously been run with SBW-9, 30 wt% loaded glass (from the EV-16 run). The transition of glass compositions can be followed by the alumina concentration. The heel had an alumina concentration greater than eight percent. The alumina concentration in the SBW-22, 20 wt% melter feed was targeted at 5.5 wt%. The alumina concentration of the pour sample taken after more than eight hours of operation indicates that a significant quantity of the heel is still in the furnace (alumina concentration in SBW-9-30 was 9.58 wt.%). In the next pour sample taken at 16h 50min of operation, the transition to the feed composition is almost complete. Table 11 is the analysis of the samples taken during the SBW-22, 22 wt% segment of operation.

Table 10. Analyses of Samples from the SMRF during the SBW-22 20% (4a) Segment of Run 4.

	Cold Cap	Cold Cap	Dip sample	Glass Pour	Glass Pour	Glass Pour	Glass Pour	SBW-22-20%
	8 hr 20 min	25 hr	25 hr 30 min	3 hr 45 min*	8 hr 20 min	16 hr 50 min	25 hr	Target
Al_2O_3	8.41	5.21	5.26	7.77	7.03	5.85	5.27	5.50
B_2O_3	5.09	5.06	5.57	9.79	8.37	6.54	5.64	4.90
CaO	3.97	3.45	3.64	4.23	4.27	4.22	4.22	4.45
Fe_2O_3	1.54	1.94	1.88	7.00	5.25	3.23	2.12	1.50
K_2O	1.61	1.70	1.81	2.25	2.09	1.91	1.72	1.51
Li_2O	4.56	4.43	4.63	3.57	3.92	4.26	4.59	4.89
MgO	1.57	1.55	1.50	0.38	0.76	1.21	1.50	1.48
MnO	0.15	0.17	0.17	0.30	0.23	0.18	0.16	0.16
Na_2O	13.15	13.74	13.62	14.58	14.04	13.69	13.42	13.82
SO_3	0.95	0.98	1.02	0.75	0.98	0.97	0.94	0.91
SiO_2	55.19	57.34	55.27	44.98	50.78	53.29	54.76	54.36
ZrO_2	1.82	1.90	1.81	0.15	0.68	1.33	1.73	1.95
V_2O_5	3.00	2.13	2.15	0.19	1.07	2.07	2.46	3.92
Fe^{2+}/Fe^{tot}	--	--	--	0.7	0.2	0.2	0.2	--
Total	101.02	99.59	98.32	95.95	99.48	98.75	98.52	99.34

*pour initiated

Table 11. Analyses of Samples from the SMRF during the SBW-22 22% (4b) Segment of Run 4.

	Cold Cap	**Cold Cap**	**Dip sample**	**Glass Pour**	**Glass Pour**	**SBW-22-22%**
	42 hr 45 min	51 hr 30 min	End of run	33 hr 45 min	51 hr	Target
Al_2O_3	5.21	5.76	5.12	5.24	5.27	6.06
B_2O_3	4.80	4.80	4.57	5.44	4.96	4.78
CaO	3.91	4.47	3.92	3.79	3.97	4.39
Fe_2O_3	1.45	1.52	1.56	1.94	1.78	1.50
K_2O	1.88	1.80	1.72	1.72	1.82	1.66
Li_2O	4.35	4.41	4.22	4.48	4.50	4.77
MgO	1.47	1.66	1.51	1.51	1.53	1.45
MnO	0.16	0.17	0.16	0.16	0.17	0.18
Na_2O	13.59	14.09	13.37	13.34	13.78	14.77
SO_3	1.44	1.16	1.19	0.91	0.99	1.00
SiO_2	53.27	53.90	53.56	55.88	54.80	53.00
ZrO_2	1.74	1.82	1.76	1.81	1.80	1.90
V_2O_5	2.81	2.94	2.69	2.27	2.71	3.82
Fe^{2+}/Fe^{tot}	--	--	--	0.4	--	--
Total	96.07	98.51	95.37	98.50	98.08	99.28

X-ray diffraction analysis of the cold cap sample taken at 42h 45min of operation indicates the presence of undissolved quartz and that sulfate salts had formed. The next cold cap sample, taken 45 minutes after feeding ended, indicated that the glass was free of crystalline material. Figure 12 and Figure 13 are the XRD patterns for the cold cap samples.

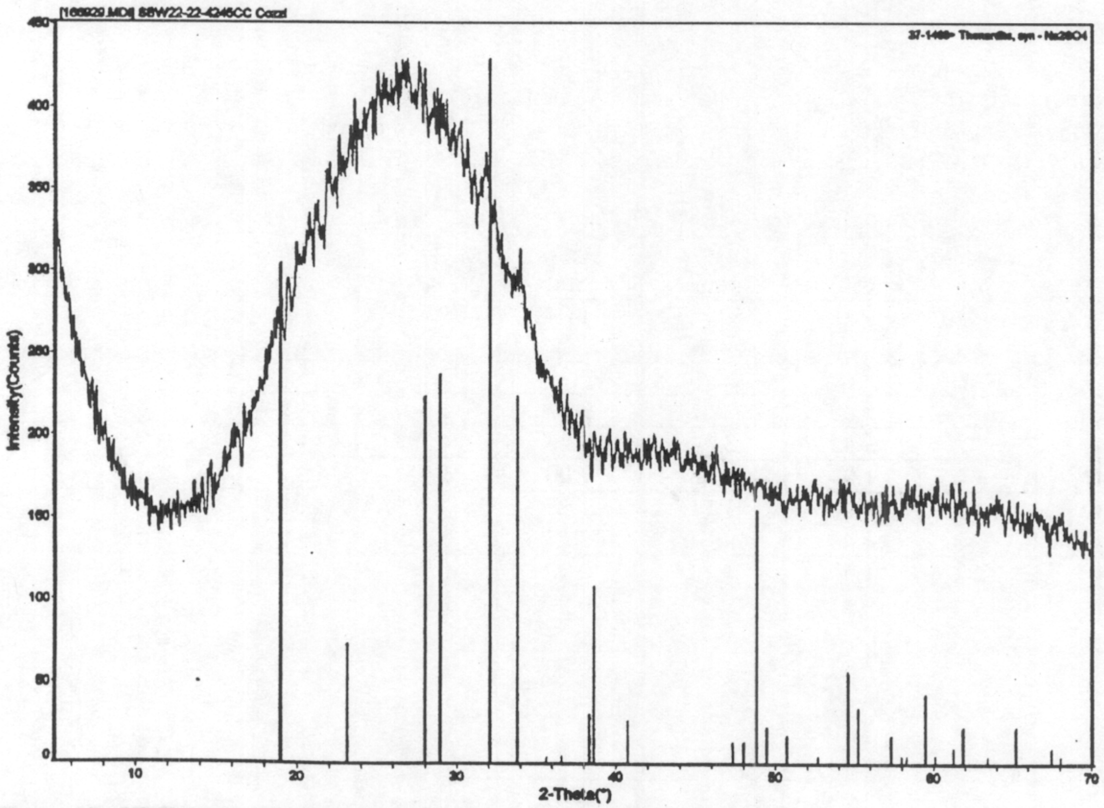

Figure 12. XRD pattern for cold cap sample taken at 42h 45min of Run 4.

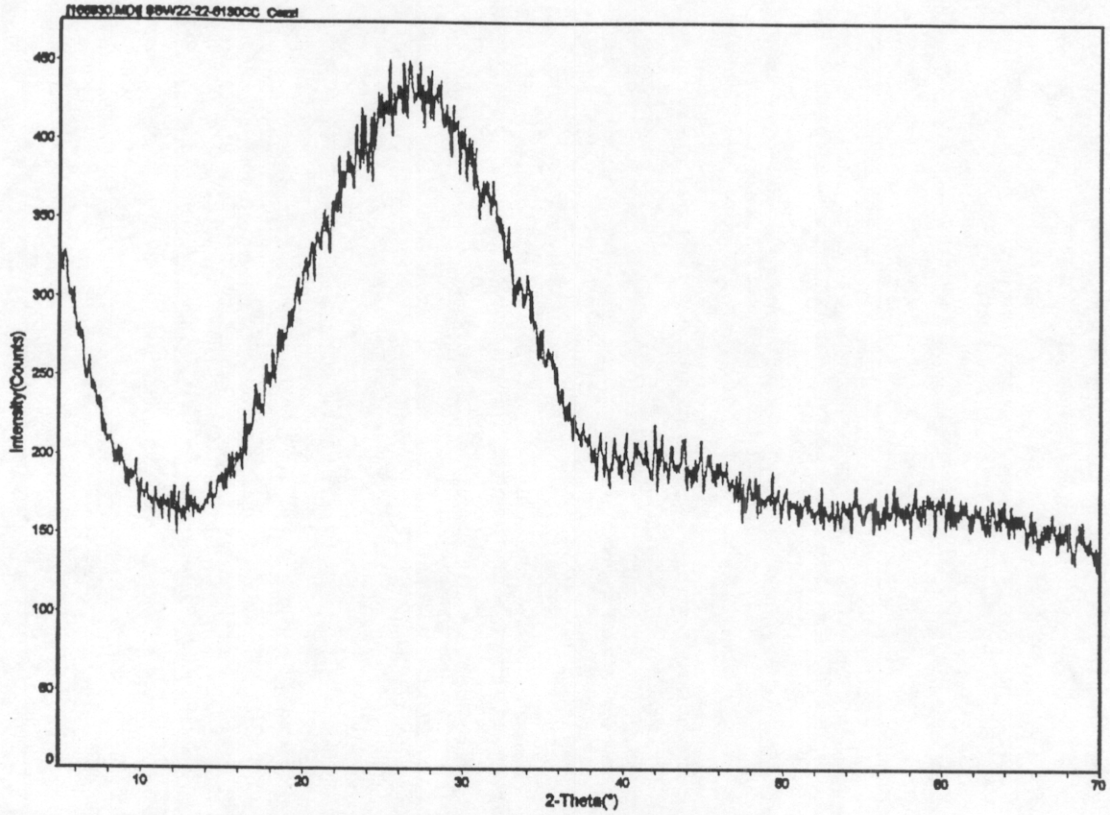

Figure 13. XRD pattern for cold cap sample taken at 51h 30min of Run 4.

Conclusions

A series of four runs were conducted in the SMRF to assess the potential formation of a sulfate salt layer with different glass compositions and various amounts of SBW. Samples of the cold cap, dip samples and glass pour stream samples were analyzed. Using melter feed from the EV-16 FY01 run, Run 1 resulted in a glass pool with a salt layer similar to that observed in the EV-16 FY01 run. Run 2 provided a transition to the updated WM-180 composition. Run 3 evaluated the SBW-22 frit (@18.2% waste loading) prior to recommendation for use in the FY01 4Q RSM run at PNNL. Run 4 demonstrated that the SMRF could be used for an extended campaign. It was also shown that SBW-22 glasses at 20 and 22 wt% waste loading did not accumulate a salt layer. Glass pool samples taken after the cold cap was allowed to burn off were free of salts.

Table 12 is melt rate for each of the four runs performed in the SMRF. The melt rate was calculated by the amount of melter feed processed over the duration of the run per melt surface area. Table 13 is a summary of the conditions and observations for the four runs performed in the SMRF.

Table 12. Melt Rate for SBW Runs.

	Run 1	Run 2	Run 3	Run 4a	Run 4b
Composition	SBW9-30	SBW9-18.2	SBW18-18.2	SBW18-20	SBW18-22
Feed Rate (lb/h/ft^2)	12.0	12.8	12.1	11.6	11.6
Feed Rate (kg/h/m^2)	58.6	62.5	59.3	56.7	56.7
Run Duration (h:mm)	9:20	9:15	8:00	32:00	18:45
Plenum Temperature	750°C	750°C	750°C	750°C	750°C

Table 13. Summary of Melter Paramters for SBW Runs.

	Run 1	Run 2	Run 3	Run 4a	Run 4b
Composition	SBW9-30	SBW9-18.2	SBW18-18.2	SBW18-20	SBW18-22
Run Duration (h:mm)	9:20	9:15	8:00	32:00	18:45
Melt Temperature	1150°C	1150°C	1150°C	1150°C	1150°C
Plenum Temperature	750°C	750°C	750°C	750°C	750°C
% SO_4 in Feed	1.05	1.00	1.00	1.09	1.20
% SO_4 in Glass	0.92	0.92	0.96	1.19	1.19
Melt Pool Observations	visible salt layer	No salt layer Thin haze	No salt layer	No salt layer	No salt layer

In addition to the demonstration of the ability of the SMRF to evaluate sulfate formation, insight into the effect of glass viscosity on melt rate can also be obtained. The SMRF can currently be used to evaluate the effect of the form of the glass formers on melt rate, redox and sulfur retention. There is also the potential to outfit the SMRF to measure off-gas speciation.

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APPENDIX A
RUN PLANS FOR THE SLURRY FED MELT RATE FURNACE RUNS TO SUPPORT GLASS FORMULATION DEVELOPMENT FOR INEEL SODIUM-BEARING WASTE

Westinghouse Savannah River Company

Interoffice Memorandum

SRT-GDP-2001-00065

June 28, 2001

To: D. K. Peeler, 773-43A

From: M. E. Stone, 704-1T

Run Plan for Frit SBW-9 and Revised Simulant (U)

References: SRT-GPD-2001-00061, SRT-GDP-2001-00052 Revision 1

The Slurry-fed Melt Rate Furnace (SMRF) will be utilized to study sulfate salt formation and/or accumulation during vitrification of the Idaho National Engineering and Environmental Laboratory (INEEL) Sodium Bearing Waste (SBW). A run will be performed with Frit SBW-9 and the revised simulant composition to provide a baseline for tests with alternative frits.

Approximately 50 gallons of the revised simulant was madeup on 6/20/01. This simulant does not contain glass formers or reductant. A portion of the simulant will be combined with the appropriate glass formers and reductant to produce melter feed for this run, as shown in Attachment A.

The plenum temperature will be held at the same temperature (750° C) as the run with SBW-9 and the original simulant. The feed will be fed using automatic control: a dispensing pump will add a known amount at intervals determined by the vapor space temperature controller. Glass level in the melter is controlled by an overflow. Glass poured from the melter during the run will be collected and shipped to SRTC for analysis in the SRTC Mobile Laboratory.

Operating Parameters

Parameter	Value	Units
Melter Temperature	1150	Celsius
Plenum Temperature	760	Celsius
Feed Pump Temperature Controller Setting	750	Celsius
Sugar Addition Amount	160	Grams/liter
Sulfur Content of Simulant (Target)	4.5	% oxide
Frit Formulation	SBW-9	
Waste Loading	18.2	% oxide
Simulant Volume to be Used	5	gallons
Feed Pump Setting	Single Dispense	
Feed Pump Speed	20	percent
Feed Pump Flow Time	5	seconds
Feed Pump Pause Time	100	percent
Approximate Length of Run (@2.4 L/hr)	8	Hours

The amount of sulfate salt on the surface will be qualitatively assessed using visual observations of the melt pool. An assessment of a salt layer accumulation will also be made. Dip stick measurements of the salt layer and dip samples of the melt pool will be taken as specified by the researcher. The glass poured from the melter will be sampled at intervals during the run.

If specified by researcher, the melter will be shutdown at the completion of the feed addition and allowed to cool. The sulfate present on the surface of the quenched glass pool will be evaluated. The melter would be reheated to drain the glass.

Draining

The melter will contain residual glass at the start of the test program. This glass will be removed from the melter by draining as much glass as possible from the melter. The melter will be drained at the completion of the run as required in preparation for the next run.

c:	A. D. Cozzi, 773-43A	S. L. Marra, 704-1T
	D. F. Bickford, 773-43A	D. C. Witt, 704-1T
	GPD Files	INEEL SBW Sulfate Tests Laboratory Notebook

Attachment A. Makeup of Melter Feed

Glass Former Additions

Add 23.85 kg (18.93 liters or 5 gallons) of revised simulant to makeup vessel.

Amount added _______________ kg

While maintaining constant mixing, add the following glass formers to the vessel:

Glass Former	MW	Amount per Liter	**Addition Amount**	Amount Added
	g/mol		**Kilograms**	kilograms
SiO2	60.1	354.57	**6.71**	
LiOH Hydrate	41.9	76.70	**1.45**	
Ca(OH)2	74.1	36.03	**0.68**	
H3BO3	61.8	145.31	**2.75**	
Fe2O3	159.6	54.549	**1.03**	

Add 3.03 kilograms (160 g/L) of sugar to vessel while maintaining mixing.

Maintain mixing and transfer as required to melter feed vessel.

Westinghouse Savannah River Company

Interoffice Memorandum

SRT-GDP-2001-00065

June 28, 2001

To: D. K. Peeler, 773-43A

From: M. E. Stone, 704-1T

Run Plan for Frit SBW-9 and Revised Simulant (U)

References: SRT-GPD-2001-00061, SRT-GDP-2001-00052 Revision 1

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Approximately 50 gallons of the revised simulant was madeup on 6/20/01. This simulant does not contain glass formers or reductant. A portion of the simulant will be combined with the appropriate glass formers and reductant to produce melter feed for this run, as shown in Attachment A.

The plenum temperature will be held at the same temperature (750° C) as the run with SBW-9 and the original simulant. The feed will be fed using automatic control: a dispensing pump will add a known amount at intervals determined by the vapor space temperature controller. Glass level in the melter is controlled by an overflow. Glass poured from the melter during the run will be collected and shipped to SRTC for analysis in the SRTC Mobile Laboratory.

Operating Parameters

Parameter	Value	Units
Melter Temperature	1150	Celsius
Plenum Temperature	760	Celsius
Feed Pump Temperature Controller Setting	750	Celsius
Sugar Addition Amount	160	Grams/liter
Sulfur Content of Simulant (Target)	4.5	% oxide
Frit Formulation	SBW-9	
Waste Loading	18.2	% oxide
Simulant Volume to be Used	5	gallons
Feed Pump Setting	Single Dispense	
Feed Pump Speed	20	percent
Feed Pump Flow Time	5	seconds
Feed Pump Pause Time	100	percent
Approximate Length of Run (@2.4 L/hr)	8	Hours

The amount of sulfate salt on the surface will be qualitatively assessed using visual observations of the melt pool. An assessment of a salt layer accumulation will also be made. Dip stick measurements of the salt layer and dip samples of the melt pool will be taken as specified by the researcher. The glass poured from the melter will be sampled at intervals during the run.

If specified by researcher, the melter will be shutdown at the completion of the feed addition and allowed to cool. The sulfate present on the surface of the quenched glass pool will be evaluated. The melter would be reheated to drain the glass.

Draining

The melter will contain residual glass at the start of the test program. This glass will be removed from the melter by draining as much glass as possible from the melter. The melter will be drained at the completion of the run as required in preparation for the next run.

c: A. D. Cozzi, 773-43A
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GPD Files
S. L. Marra, 704-1T
D. C. Witt, 704-1T
INEEL SBW Sulfate Tests Laboratory Notebook

Attachment A. Makeup of Melter Feed

Glass Former Additions

Add 23.85 kg (18.93 liters or 5 gallons) of revised simulant to makeup vessel.

Amount added ______________ kg

While maintaining constant mixing, add the following glass formers to the vessel:

Glass Former	MW	Amount per Liter	**Addition Amount**	Amount Added
	g/mol		**Kilograms**	kilograms
SiO2	60.1	354.57	**6.71**	
LiOH Hydrate	41.9	76.70	**1.45**	
Ca(OH)2	74.1	36.03	**0.68**	
H3BO3	61.8	145.31	**2.75**	
Fe2O3	159.6	54.549	**1.03**	

Add 3.03 kilograms (160 g/L) of sugar to vessel while maintaining mixing.

Maintain mixing and transfer as required to melter feed vessel.

Westinghouse Savannah River Company

Interoffice Memorandum

SRT-GDP-2001-00067

June 28, 2001

To: D. K. Peeler, 773-43A

From: M. E. Stone, 704-1T

Run Plan for Frit SBW-22 and Revised Simulant (U)

References: SRT-GPD-2001-00061, SRT-GPD-2001-00065, SRT-GDP-2001-00052 Revision 1

The Slurry-fed Melt Rate Furnace (SMRF) will be utilized to study sulfate salt formation and/or accumulation during vitrification of the Idaho National Engineering and Environmental Laboratory (INEEL) Sodium Bearing Waste (SBW). A run will be performed with Frit SBW-22 and the revised simulant composition to provide a baseline for tests with alternative frits.

Approximately 50 gallons of the revised simulant was madeup on 6/20/01. This simulant does not contain glass formers or reductant. A portion of the simulant will be combined with the appropriate glass formers and reductant to produce melter feed for this run, as shown in Attachment A.

The plenum temperature will be held at the same temperature (750° C) as the run with SBW-9 and the original simulant. The feed will be fed using automatic control: a dispensing pump will add a known amount at intervals determined by the vapor space temperature controller. Glass level in the melter is controlled by an overflow. Glass poured from the melter during the run will be collected and shipped to SRTC for analysis in the SRTC Mobile Laboratory.

Operating Parameters

Parameter	Value	Units
Melter Temperature	1150	Celsius
Plenum Temperature	760	Celsius
Feed Pump Temperature Controller Setting	750	Celsius
Sugar Addition Amount	160	Grams/liter
Sulfur Content of Simulant (Target)	4.5	% oxide
Frit Formulation	SBW-22	
Waste Loading	18.2	% oxide
Simulant Volume to be Used	5	gallons
Feed Pump Setting	Single Dispense	
Feed Pump Speed	20	percent
Feed Pump Flow Time	5	seconds
Feed Pump Pause Time	100	percent
Approximate Length of Run (@2.4 L/hr)	8	Hours

The amount of sulfate salt on the surface will be qualitatively assessed using visual observations of the melt pool. An assessment of a salt layer accumulation will also be made. Dip stick measurements of the salt layer and dip samples of the melt pool will be taken as specified by the researcher. The glass poured from the melter will be sampled at intervals during the run.

If specified by researcher, the melter will be shutdown at the completion of the feed addition and allowed to cool. The sulfate present on the surface of the quenched glass pool will be evaluated. The melter would be reheated to drain the glass.

Draining

The melter will contain residual glass at the start of the test program. This glass will be removed from the melter by draining as much glass as possible from the melter. The melter will be drained at the completion of the run as required in preparation for the next run.

c:
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GPD Files
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INEEL SBW Sulfate Tests Laboratory Notebook

Attachment A. Makeup of Melter Feed

Glass Former Additions

Add 23.85 kg (18.93 liters or 5 gallons) of revised simulant to makeup vessel.

Amount added ______________ kg

While maintaining constant mixing, add the following glass formers to the vessel:

Glass Former	Frit Composition	MW	Amount per Liter	**Addition Amount**	Amount Added
	Wt% oxide	g/mol	grams	**kilograms**	kilograms
SiO2	67.95	60.1	370.7	**7.015**	
NaOH	4.29	40	30.20	**0.571**	
LiOH Hydrate	6.11	41.9	93.72	**1.774**	
Ca(OH)2	5.02	74.1	36.17	**0.685**	
H3BO3	6.03	61.8	58.41	**1.105**	
MgO	1.75	40.3	9.546	**0.181**	
Fe2O3	1.52	159.6	8.291	**0.157**	
V2O5	4.88	181.8	26.62	**0.504**	
ZrO2	2.44	123.2	13.31	**0.252**	

Add 3.03 kilograms (160 g/L) of sugar to vessel while maintaining mixing.

Maintain mixing and transfer as required to melter feed vessel.

Label container as

INEEL SBW Revised Simulant for Sulfate Tests
160 g/L sugar
18.2% SBW-22

Westinghouse Savannah River Company

Interoffice Memorandum

SRT-GDP-2001-00075

July 24, 2001

To: D. K. Peeler, 773-43A

From: M. E. Stone, 704-1T

Run Plan for 48 Hour Tests with Frit SBW-22 and Revised Simulant (U)

References: SRT-GPD-2001-00061, SRT-GPD-2001-00067, SRT-GPD-2001-00065, SRT-GDP-2001-00052 Revision 1

The Slurry-fed Melt Rate Furnace (SMRF) will be utilized to study sulfate salt formation and/or accumulation during vitrification of the Idaho National Engineering and Environmental Laboratory (INEEL) Sodium Bearing Waste (SBW). A run will be performed with Frit SBW-22 and the FY01 simulant composition.

Approximately 50 gallons of the revised simulant was madeup on 6/20/01. This simulant does not contain glass formers or reductant. A portion of the simulant will be combined with the appropriate glass formers and reductant to produce melter feed (SBW-22 at 20% waste loading) for this run, as shown in Attachment A.

The plenum temperature will be held at the same temperature (750°C) as the previous run with SBW-22 (SRT-GPD-2001-00067). The feed will be fed using automatic control: a dispensing pump will add a known amount at intervals determined by the vapor space temperature controller. Glass level in the melter is controlled by an overflow. Glass poured from the melter during the run will be collected and shipped to the Savannah River Technology center (SRTC) for analysis in the SRTC Mobile Laboratory.

Operating Parameters

Parameter	Value	Units
Melter Temperature	1150	Celsius
Plenum Temperature	760	Celsius
Feed Pump Temperature Controller Setting	750	Celsius
Nitric Acid Addition	60	ml/liter
Sugar Addition Amount	165	Grams/liter
Sulfur Content of Simulant (Target)	4.5	% oxide
Frit Formulation	SBW-22	
Waste Loading	20	% oxide
Approximate Simulant Volume to be Used	60	liters
Feed Pump Setting	Single Dispense	
Feed Pump Speed	20	percent
Feed Pump Flow Time	5	seconds
Feed Pump Pause Time	100	percent
Length of Run	48	Hours

The presence of a salt layer on the surface will be qualitatively assessed using visual observations of the melt pool. An assessment of whether the salt layer is accumulating as the test progresses will also be made. Dip stick measurements of the salt layer and dip samples of the melt pool will be taken as specified by the researcher. The glass poured from the melter will be sampled at intervals during the run.

If specified by researcher, the melter will be idled periodically to allow the cold cap to burn off to allow the amount of sulfate present on the surface of the glass pool to be evaluated.

Draining

The melter will contain residual glass at the start of the test program as a result of incomplete drainage after the previous tests (complete draining is almost impossible and/or impractical). After the completion of the proposed tests, the melter will be drained to the extent possible in preparation for the next run.

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Attachment A. Makeup of Melter Feed (SBW-22, 20%)

The melter feed will be madeup in small batches to allow for homogenization to remove clumps. Each batch will be based on 10 liters of simulant and will be madeup in a 5 gallon bucket.

Add 12.6 kg (10 liters) of revised simulant to a 5 gallon bucket.

While maintaining constant mixing, add 600 ml (864 grams) of 70% nitric acid to bucket.

While maintaining constant mixing, add the following glass formers to the bucket in the order listed.

Glass Former	Frit Composition	MW	Amount per Liter	**Addition Amount**
	Wt% oxide	g/mol	grams	**kilograms**
SiO_2	67.95	60.1	329.9	**3.299**
V_2O_5	4.88	181.8	23.69	**0.2369**
ZrO_2	2.44	123.2	11.85	**0.1185**
MgO	1.75	40.3	8.496	**0.08496**
Fe_2O_3	1.52	159.6	7.379	**0.07379**
H_3BO_3	6.03	61.8	51.99	**0.5199**
NaOH	4.29	40	26.87	**0.2687**
LiOH Hydrate	6.11	41.9	83.41	**0.8341**
$Ca(OH)_2$	5.02	74.1	32.19	**0.3219**

Add 1.65 kilograms (165 g/L) of sugar to bucket while maintaining mixing.

Remove mixer and install homogenizer on feed vessel. Run homogenizer until clumps are removed.

Label container as:

INEEL SBW Revised Simulant for Sulfate Tests
60 ml/L Added HNO_3
165 g/L sugar
20% SBW-22
Makeup Date: XX/XX/XX
Michael Stone 803-557-7288
Alex Cozzi 803-725-3250