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SLUDGE BATCH 5 (SB5): SELECTION OF CANDIDATE FRITS AND CHARACTERIZATION OF PRELIMINARY GLASS SYSTEMS

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Prepared for the U.S. Department of Energy Under
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EXECUTIVE SUMMARY

Six potential frits were identified as candidates for processing the February 2007 projected SB5 composition (i.e., no implementation of aluminum dissolution) from an array of frit formulations based upon composition-property model predictions. Test glasses were fabricated in the laboratory to verify the applicability of the product performance models to glasses produced with these frits.

Characterization of the glasses fabricated with the selected frits showed that all of the glasses had durability responses that are considered very acceptable at a waste loading of 36%. The durability responses were predictable by the free energy of hydration models. No crystallization was identified in the quenched glasses. Samples of the glasses that were slowly cooled following the canister centerline cooled (ccc) thermal profile were found to contain small amounts of magnetite. This crystalline phase had little impact on the durability of the glasses, and therefore is not an issue for concern based on the February 2007 projections. Note that revised versions of the SB5 flowsheet, including those incorporating aluminum dissolution, are expected, which will require additional frit development work when received.

Initial melt rate testing results showed that the previously identified trend of increasing melt rate with increasing concentration of B_2O_3 for SB4 may be extended to this SB5 system. A complete report on melt rate testing with these frits will be issued at a later date.

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LIST OF ABBREVIATIONS

AD	Analytical Development
ARM	Approved Reference Material
ARP	Actinide Removal Process
ccc	canister centerline cooled
CSSX	Caustic Side Solvent Extraction
del Gp ΔG_P	Free Energy of Hydration
DWPF	Defense Waste Processing Facility
EA	Environmental Assessment
LWO	Liquid Waste Organization
MAR	Measurement Acceptability Region
MCU	Modular CSSX Unit
MRF	Melt Rate Furnace
MST	monosodium titanate
PCCS	Product Composition Control System
PCT	Product Consistency Test
PSAL	Process Science Analytical Laboratory
SB4 / SB5	Sludge Batch 4 / Sludge Batch 5
SRAT	Slurry Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
WL	Waste Loading
XRD	X-ray Diffraction

1.0 Introduction

Sludge Batch 5 (SB5) is the next sludge batch to be qualified for processing at the Defense Waste Processing Facility (DWPF). A sludge batch is defined as a single tank of sludge or a combination of sludges from different tanks that has been or will be qualified for transfer to DWPF. SB5 will consist primarily of Tank 51 – Sludge Batch 4 (SB4), which has already been qualified, and smaller portions of material from Tanks 5, 6, and 7. SB5 is also anticipated to contain monosodium titanate (MST)/sludge solids from the Actinide Removal Process (ARP) and cesium strip effluent from the Modular Caustic Side Solvent Extraction (CSSX) Unit (MCU). However, the work described in this report does not take the addition of these latter two waste streams into consideration.

Prior to the processing of a new sludge batch in DWPF, the Savannah River National Laboratory (SRNL) must analyze and confirm that the sludge batch produces an acceptable glass via durability models of the Product Composition Control System (PCCS). An integral part of this qualification process is the development of a frit, which when coupled with the sludge, produces an acceptable glass and maximizes waste throughput (which is dependent upon waste loading and melt rate).

The objectives of this task are scoping in nature and are intended to provide guidance for frit development efforts at SRNL as the SB5 flowsheet is further refined. A collection of several key criteria will provide the basis for the frit development and selection process. These include identifying frits that: provide reasonable projected operational windows over the anticipated SB5 composition region, are robust to anticipated sludge composition variation, improve or maintain high waste loadings, improve or maintain high melt rates, and have "frittable" compositions. Of particular interest will be the determination of waste throughput factors (melt rate versus waste loading trends) for select SB5 glass systems of interest. The primary mechanism to assess candidate SB5 frits will be a paper study using model-based predictions and current PCCS constraints.

Later frit development efforts will assess the viability of using the current 0.6 wt% SO_4^{2-} limit (in glass) and/or the possibility of increasing the SO_4^{2-} solubility limit in PCCS to account for anticipated sulfur concentrations in SB5. If warranted, later studies may also assess the need to increase the TiO_2 limit in PCCS to accommodate the MST streams. Other factors such as the potential formation of nepheline will also be assessed through paper study assessments and experimental studies. Melt rate assessments (utilizing both dry-fed and slurry-fed systems) will also be assessed for select systems of interest to support the frit recommendation process and to evaluate melt rate as a function of waste loading.

The preliminary study described in this report identifies candidate frits using the February 2007 SB5 composition projection from the Liquid Waste Organization (LWO), considering a sludge-only operational mode and without the implementation of aluminum dissolution. A paper study was used to select a small series of candidate frits for further study. In addition, the intent is to measure melt rates for these glass systems using SRNL's dry-fed melt rate furnace (MRF). Glass compositions are developed for the selected frits using the projected SB5 composition. These glasses are fabricated and characterized in the laboratory to verify that the durability and nepheline crystallization models used in the paper study are applicable to these glass systems.

This work is undertaken in response to a Technical Task Request issued by DWPF[1] and is performed under a Task Technical and Quality Assurance Plan.[2]

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2.0 Experimental Procedure

This section describes the strategy used to select the glasses for the study, including the target sludge and frit compositions. The target glass compositions are then given, followed by a discussion of the techniques used to fabricate and analyze the glasses.

2.1 Frit Selection Strategy

A projected composition for SB5 was received from LWO in February 2007.[3] This projection did not include the impacts of implementing the aluminum dissolution process or the addition of ARP/MCU feed streams. The data were converted to an oxide basis and are shown in Table 2-1. Projected washing data were provided when the LWO report was issued. These data were used to project a SO_4^{2-} concentration for the sludge,[a] which is also included in Table 2-1.

Table 2-1. Projected SB5 composition (in wt% oxides), renormalized to exclude the radioactive components.

Sludge Component	Concentration (wt%)
Al_2O_3	30.58
B_2O_3	0.00
BaO	0.11
CaO	2.09
Ce_2O_3	0.23
Cr_2O_3	0.20
CuO	0.07
Fe_2O_3	24.30
K_2O	0.16
La_2O_3	0.03
Li_2O	0.00
MgO	1.41
MnO	5.20
Na_2O	22.65
NiO	2.31
PbO	0.10
SO_4^{2-}	1.16
SiO_2	1.82
ThO_2	0.01
TiO_2	0.51
U_3O_8	6.75
ZnO	0.07
ZrO_2	0.23

An array of frit compositions was next developed to be combined with this sludge at waste loadings (WLs) from 25% to 60%. The frit components and their range of concentrations were selected based on previous experience with DWPF processing, and are listed in Table 2-2. Every combination of B_2O_3, CaO, Fe_2O_3, Li_2O, MgO, Na_2O and ZrO_2 within the concentration ranges given in Table 2-2 was used to develop frit compositions, with the SiO_2 concentration making up the remainder of the mass. This resulted in a total of 14,580 frit compositions to be assessed.

[a] See WSRC-NB-2006-00017, pages 122-123 for details of the SO_4^{2-} calculation.

Table 2-2. Frit components and their concentration ranges used for MAR assessments.

Frit Component	Concentration Range (wt%)	Increment (wt%)
B_2O_3	12-20	1
CaO	0-2	1
Fe_2O_3	0-2	1
Li_2O	8-11	1
MgO	0-2	1
Na_2O	0-4	1
SiO_2	57-80	1
ZrO_2	0-2	1

The paper assessment paired the sludge composition in Table 2-1 with the 14,580 frit compositions described by Table 2-2 and applied the PCCS Measurement Acceptability Region (MAR) criteria to determine which combinations would produce acceptable glasses (based on model predictions) over the range of WLs from 25% to 60%. The SO_4^{2-} limit of 0.6 wt% in glass was not activated in the MAR. Other constraints are expected to limit the achievable WL before the SO_4^{2-} limit is reached for this SB5 projection (1.24 wt% SO_4^{2-} in sludge) since the 0.60 wt% SO_4^{2-} limit would not be exceed until a WL of 48%. The MAR assessment results for six selected frits are shown in Table 2-3. These six frits were selected based on their relatively wide range of waste loadings over which an acceptable glass was predicted, their relatively high concentrations of B_2O_3 (which is likely to both improve melt rate and suppress nepheline crystallization), and in the case of Frit 510, its use in processing Sludge Batch 4.

Table 2-3. Composition of the six frits selected for MRF testing (in wt% oxides) and their MAR assessment results.

Frit ID	B_2O_3	Li_2O	Na_2O	SiO_2	WL Range (wt%)	Limiting Constraint
503	14	8	4	74	25 – 42	Nepheline
510	14	8	8	70	25 – 38	Nepheline
516	14	11	2	73	25 – 43	T_L, Nepheline
517	17	10	3	70	25 – 42	T_L, Nepheline
518	20	10	1	69	25 – 39	T_L
519	20	9	3	68	25 - 41	T_L, Nepheline

For the six selected frits, the upper end of the WL range was limited by either predictions of nepheline crystallization, a high liquidus temperature (T_L), or both. Frit 518 was the only system that was not limited by a wasteform-affecting constraint (nepheline crystallization was not a limiting constraint until 44% WL).[a] The six frits cover a wide range of B_2O_3 concentrations (14-20 wt%) and Na_2O concentrations (1-8 wt%), which should provide the ability to evaluate the influence of these components on the measured melt rate for the February 2007 SB5 system.

[a] Nepheline crystallization is considered a wasteform-affecting constraint due to its adverse impact on durability of the glass. Liquidus temperature is considered a process-affecting constraint due to concerns over the accumulation of crystalline material within the melter.

2.2 Target Glass Compositions

Crucible-scale glass melts were used to measure the durability of glasses produced with the six selected frits as well as to determine whether any crystallization issues exist for glasses that are slowly cooled. The frits were combined with the projected SB5 composition at a WL of 36% (slightly above the DWPF target of 34% for SB5) to develop the target test glass compositions, which are given in Table 2-4. Note that the sludge composition given in Table 2-4 was renormalized to remove the radioactive components (U and Th) in order to simplify laboratory experiments.

Table 2-4. Target compositions of the six test glasses (in wt% oxides).

Glass ID	SB5MR-503	SB5MR-510	SB5MR-516	SB5MR-517	SB5MR-518	SB5MR-519
Frit	503	510	516	517	518	519
WL	36	36	36	36	36	36
Al_2O_3	11.81	11.81	11.81	11.81	11.81	11.81
B_2O_3	8.96	8.96	8.96	10.88	12.80	12.80
BaO	0.04	0.04	0.04	0.04	0.04	0.04
CaO	0.81	0.81	0.81	0.81	0.81	0.81
Ce_2O_3	0.09	0.09	0.09	0.09	0.09	0.09
Cr_2O_3	0.08	0.08	0.08	0.08	0.08	0.08
CuO	0.03	0.03	0.03	0.03	0.03	0.03
Fe_2O_3	9.38	9.38	9.38	9.38	9.38	9.38
K_2O	0.06	0.06	0.06	0.06	0.06	0.06
La_2O_3	0.01	0.01	0.01	0.01	0.01	0.01
Li_2O	5.12	5.12	7.04	6.40	6.40	5.76
MgO	0.55	0.55	0.55	0.55	0.55	0.55
MnO	2.01	2.01	2.01	2.01	2.01	2.01
Na_2O	11.30	13.86	10.02	10.66	9.38	10.66
NiO	0.89	0.89	0.89	0.89	0.89	0.89
PbO	0.04	0.04	0.04	0.04	0.04	0.04
SO_4^{2-}	0.45	0.45	0.45	0.45	0.45	0.45
SiO_2	48.06	45.50	47.42	45.50	44.86	44.22
ThO_2	0.00	0.00	0.00	0.00	0.00	0.00
TiO_2	0.20	0.20	0.20	0.20	0.20	0.20
U_3O_8	0.00	0.00	0.00	0.00	0.00	0.00
ZnO	0.03	0.03	0.03	0.03	0.03	0.03
ZrO_2	0.09	0.09	0.09	0.09	0.09	0.09
Total	100.00	100.00	100.00	100.00	100.00	100.00

2.3 Glass Fabrication

Each study glass was prepared from the proper proportions of reagent-grade metal oxides, carbonates, H_3BO_3, and salts in 150 g batches.[4] The raw materials were thoroughly mixed and placed into a 95% platinum / 5% gold, 250 ml crucible. The batch was placed into a high-temperature furnace at the target melt temperature of 1150 °C.[5] The crucible was removed from the furnace after an isothermal hold at 1150 °C for 1 hour. The glass was poured onto a clean, stainless steel plate and allowed to air cool (quench). The glass pour patty was used as a sampling stock for the various property measurements, including durability testing.

Approximately 25 g of each glass was heat-treated to simulate cooling along the centerline of a DWPF-type canister[6] to gauge the effects of thermal history on the product performance. This cooling schedule is referred to as the ccc curve. Visual observations on both quenched and ccc glasses were recorded.

2.4 Property Measurements

This section provides a general discussion of the Product Consistency Test results and the X-ray diffraction analyses of the melter test glasses.

2.4.1 *Product Consistency Test*

The Product Consistency Test (PCT)[7] was performed in triplicate on each quenched and ccc glass to assess chemical durability. Also included in the experimental test matrix was the Environmental Assessment (EA) glass,[8] the Approved Reference Material (ARM) glass, and blanks from the sample cleaning batch. Samples were ground, washed, and prepared according to the standard procedure.[7] Fifteen milliliters of Type I ASTM water were 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, where the samples were maintained at temperature for 7 days. Once cooled, the resulting solutions were sampled (filtered and acidified), then labeled and analyzed by the Process Science Analytical Laboratory (PSAL) using inductively coupled plasma – atomic emission spectroscopy (ICP-AES). Normalized release rates were calculated based on target compositions using the average of the common logarithms of the leachate concentrations.

2.4.2 *X-Ray Diffraction Analysis*

Visual observations for crystallization were performed and documented for all of the glasses. Since some of the ccc glasses were observed to contain small amounts of crystals, representative samples for all of the ccc glasses were submitted to SRNL Analytical Development (AD) for X-ray diffraction (XRD) analysis. Samples were analyzed under conditions providing a detection limit of approximately 0.5 vol%. That is, if a crystalline phase were present at 0.5 vol% or greater, the diffractometer would not only be capable of detecting the crystals but would also allow a qualitative determination of the type of crystal(s) present. Otherwise, a characteristically high background devoid of crystalline spectral peaks indicates that the glass product is amorphous, suggesting either a completely amorphous product or that the degree of crystallization is below the detection limit.

3.0 Results and Discussion

This section discusses visual observations of the glasses after fabrication, the results of XRD analyses and the results of the PCT for each glass, both quenched and ccc. The initial results of dry-fed melt rate testing are briefly presented.

3.1 Homogeneity

Visual observations of each glass were recorded after melting and quenching, as well as at the completion of the ccc heat treatment. These observations are listed in Table 3-1. The term ‘clean’ means that no crystallization was visually observed in the glass. A typical DWPF composition glass will also appear ‘black and shiny,’ which again indicates the lack of any visible crystallization.

Table 3-1. Visual observations and XRD results for the quenched and ccc version of each glass.

Glass ID	Heat Treatment	Visual Observations	XRD
SB5MR-503	quenched	clean, black and shiny	-
	ccc	slight haze with scattered surface crystals; bulk clean	magnetite
SB5MR-510	quenched	clean, black and shiny	-
	ccc	slight haze, small crystals on surface; bulk clean	magnetite
SB5MR-516	quenched	clean, black and shiny	-
	ccc	some crystals on surface; bulk clean	magnetite
SB5MR-517	quenched	clean, black and shiny	-
	ccc	crystals on surface; bulk clean	magnetite
SB5MR-518	quenched	clean, black and shiny	-
	ccc	crystals on surface; bulk clean	magnetite
SB5MR-519	quenched	clean, black and shiny	-
	ccc	crystals on surface; bulk clean	magnetite

The quenched versions of each glass appeared visually amorphous. Some crystallization was visible on the surface of each of the ccc versions of the glasses, but no crystallization was visible along the cross-section of the ccc samples (i.e., the ‘bulk’ of the glass). XRD was used to determine the type of crystallization present in each of the ccc glasses. The crystalline phase was identified as magnetite for all six of the test glasses. The small amount of magnetite present in each glass is unlikely to impact the glasses’ durability after the ccc heat treatment. This will be confirmed by the results of the PCTs.

3.2 Product Consistency Test

The PCT was completed for each of the six test glasses, both quenched and ccc. The ARM and EA standard glasses were also included in the tests. The results of the PCTs, normalized to the target glass compositions, are given in Table 3-2.

Table 3-2. PCT results for each of the test glasses and the standards.

Glass ID	Normalized Release (g/L)			
	Li	**B**	**Na**	**Si**
ARM	0.53	0.45	0.45	0.26
EA	9.46	17.76	14.82	4.01
SB5MR-503	0.59	0.51	0.44	0.37
SB5MR-503ccc	0.56	0.46	0.44	0.36
SB5MR-510	0.57	0.53	0.63	0.40
SB5MR-510ccc	0.59	0.53	0.61	0.40
SB5MR-516	0.66	0.58	0.53	0.43
SB5MR-516ccc	0.66	0.57	0.54	0.44
SB5MR-517	0.63	0.56	0.52	0.40
SB5MR-517ccc	0.65	0.58	0.55	0.43
SB5MR-518	0.67	0.61	0.46	0.40
SB5MR-518ccc	0.61	0.55	0.46	0.38
SB5MR-519	0.63	0.57	0.48	0.38
SB5MR-519ccc	0.63	0.58	0.50	0.39

The measured values for the ARM glass fall within the specified control limits.[9] Note that the normalized release for boron for the EA glass is slightly above the typical value of 16.695 g/L.[8]

The PCT results for the six study glasses show that each glass has a durability that is considered very acceptable, with normalized releases for boron that are better than an order of magnitude below that of the EA glass. There is little difference between the PCT responses of the quenched and ccc versions of each glass, indicating that the small amount of crystallization identified in the ccc glasses by visual observation and XRD has no measurable impact on durability. In terms of durability, any of the six frits tested would produce an acceptable glass with the given sludge composition and waste loading.

3.3 Model Applicability

The PCT results were compared with the durabilities predicted by the free energy of hydration, or ΔG_P model[9] to evaluate the applicability of the model (which is used as part of the DWPF process control system) to the SB5 composition projection and frits used in this study. The results of this comparison are shown in Figure 3-1.

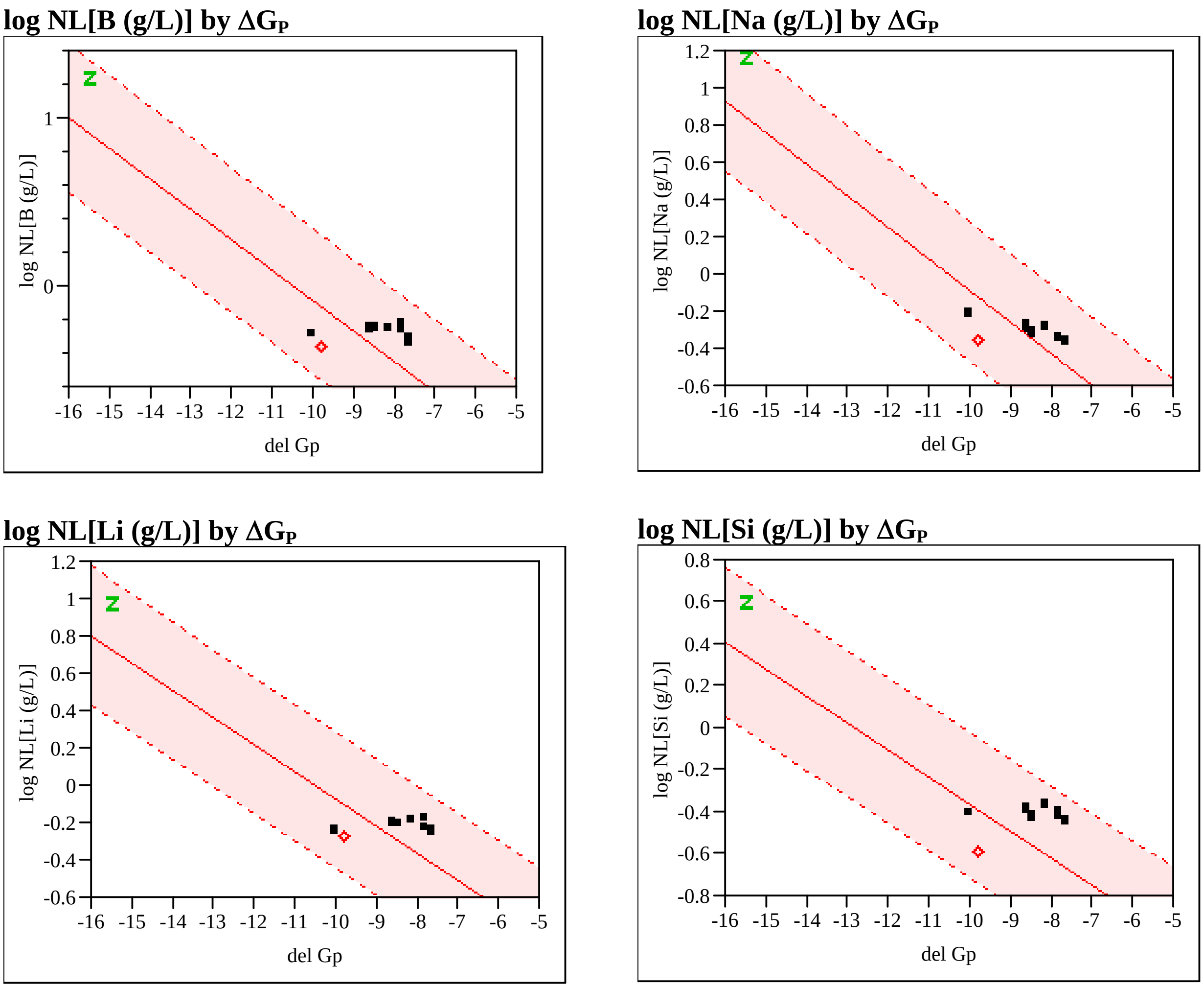

Figure 3-1. Comparison of the Measured PCT Responses and the Durabilities Predicted by the Free Energy of Hydration Model.

The solid red lines in the plots of Figure 3-1 indicate the predicted durability with respect to a particular element (B, Na, Li or Si) based on the calculated ΔG_P. The red shaded area indicates the 95% confidence bounds on the prediction for an individual glass. The measured durabilities of the ARM and EA standard glasses are indicated on these plots by the red and green data points, respectively. The durabilities of the SB5 glasses in this study, measured by the PCT, are indicated by the black data points in Figure 3-1. All of these points fall within the bounds of the model predictions, indicating that the ΔG_P model is applicable to the evaluated frit/sludge systems.

3.4 Initial Melt Rate Results

A full report on the dry-fed MRF results is forthcoming; however, the initial results will be described briefly here. Each of the six frits was combined with a dry Slurry Receipt and Adjustment Tank (SRAT) product corresponding to the sludge composition listed in Table 2-1 at 35% WL. The feeds were then melted in the MRF for 50 minutes at 1150 °C. The crucibles were then cut in cross section, and the thickness of the melted glass layer was measured. Two MRF runs were completed. The first included all six of the test frits. The results are given in Table 3-3. A second round of MRF testing was run using the best performing frits from the first run. These results are also given in Table 3-3.

Table 3-3. Melt rate results for the six frits.

Glass ID	Melt Rate (in/hr)		Components in Frit (wt%)	
	Run #1	Run #2	B_2O_3	Na_2O
SB5MR-503	0.52	0.40	14	4
SB5MR-510	0.47	-	14	8
SB5MR-516	0.47	-	14	2
SB5MR-517	0.56	0.48	17	3
SB5MR-518	0.49	-	20	1
SB5MR-519	0.53	0.60	20	3
Frit 418 Standard, First Layer	1.48	1.53	8	8
Frit 418 Standard, Second Layer	3.18	3.23	8	8

In general, the MRF testing results show that as the concentration of B_2O_3 in the frit increases, melt rate also increases. This trend is consistent with that observed in dry-fed and slurry-fed melt rate tests with SB4 – another high Al_2O_3 waste stream.[10] The impact of Na_2O concentration on melt rate is not clear from these results. Note that the Frit 418 Standard is a frit-only test (i.e., without any sludge addition) and therefore shows a considerably higher melt rate. Further data analysis for the MRF testing is underway, and the results will be more thoroughly discussed in a subsequent report.

4.0 Summary

Six potential frits were identified as candidates processing the February 2007 projected SB5 composition based upon MAR assessments of this sludge and an array of frit compositions. Test glasses were fabricated in the laboratory to verify the applicability of the product performance models to glasses produced with these frits.

Characterization of the glasses fabricated with the selected frits showed that all of the glasses had durability responses that are considered very acceptable and predictable by the free energy of hydration model. No crystallization was identified in the quenched glasses. Samples of the glasses that were slowly cooled following the canister centerline cooled (ccc) thermal profile were found to contain small amounts of magnetite. This crystalline phase had little impact on the durability of the glasses, and therefore is not an issue for concern.

Initial melt rate testing results showed that the previously identified trend of increasing melt rate with increasing concentration of B_2O_3 may be extended to this SB5 system. A complete report on melt rate testing with these frits will be issued at a later date.

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5.0 Path Forward

- The results of the MRF testing will be more thoroughly discussed in a subsequent report.
- Subsequent MAR assessments will be performed on revised SB5 compositional projections as washing or blending strategies change or as the Al-dissolution flowsheet evolves.
- Additional melt rate testing should be undertaken when an updated SB5 composition projection is received from LWO.
- If the aluminum dissolution process will be implemented for SB5 processing, additional melt rate testing should be performed based on the estimated impacts of aluminum dissolution to the SB5 flowsheet.

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