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THE IMPACT OF FEED PREPARATION ACID STOICHIOMETRY AND REDOX ON MELT RATE FOR THE SB3-FRIT 418 FEED SYSTEM

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EXECUTIVE SUMMARY

In March 2004, the Defense Waste Processing Facility (DWPF) transitioned to a blend of Sludge Batch 2 (SB2) and Sludge Batch 3 (SB3), and implemented a frit change from Frit 320 to Frit 418. This blended sludge batch has been designated SB3 although previously SRNL has called this new sludge batch SB2/3. A series of dry-fed tests (using the Melt Rate Furnace or MRF) and slurry fed tests (using the Slurry-Fed Melt Rate Furnace or SMRF) have been performed to investigate the effect of feed preparation acid stoichiometry and REDOX (REDuction/OXidation) on the melt rate of the SB3 – Frit 418 system. With regards to acid stoichiometry, the current DWPF target of 155% gave a higher melt rate than the 185% when tested in the SMRF. This contradicted the MRF results for the first time that both melt systems were used to evaluate melt rate with various feeds. The SMRF results should be used as slurry-fed results are more representative of what would occur in the DWPF, especially since the variable tested did not change the final glass composition. With regard to changes in REDOX (0.0, 0.1, and 0.2), the MRF tests indicated no difference while the SMRF tests showed that melt rate was not negatively impacted until a REDOX below 0.1 was used. In addition, all SMRF acid stoichiometry and REDOX feeds tested were diluted from about 50 to 45 weight percent total solids after melt rates had been determined at 50 weight percent. In all cases the lower weight percent solids resulted in lower melt rates. Based on this testing, the Immobilization Technology Section (ITS) recommends that the DWPF continue to operate the feed preparation processing for SB3 with Frit 418 with a targeted acid stoichiometry of 155% and a targeted REDOX of 0.2. If needed for other process concerns, REDOX targets approaching 0.1 could be used at DWPF before melt rate would be negatively impacted.

Because all of this testing was performed on small-scale equipment with dried or slurried, non-radioactive simulant, the exact impact of feed acid stoichiometry and REDOX on the radioactive sludge in a DWPF-sized melter could not be quantified.

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LIST OF ACRONYMS

ACTL	Aiken County Technology Laboratory
DWPF	Defense Waste Processing Facility
EDS	Energy dispersive spectroscopy
ITS	Immobilization Technology Section
LMR	Linear Melt Rate
MRF	Melt Rate Furnace
REDOX	REDuction/OXidation
SEM	Scanning electron microscopy
SB2	Sludge batch 2
SB3	Sludge batch 3
SME	Slurry Mix Evaporator
SMRF	Slurry-fed Melt Rate Furnace
SRAT	Sludge Receipt and Adjustment Tank
SRS	Savannah River Site
SRNL	Savannah River National Laboratory
WL	Waste Loading
WSRC	Westinghouse Savannah River Company
XRD	X-ray diffraction

1.0 INTRODUCTION

In March 2004 the Defense Waste Processing Facility (DWPF) transitioned to a blend of Sludge Batch 2 (SB2) and Sludge Batch 3 (SB3), and implemented a frit change from Frit 320 to Frit 418. This blended sludge batch has been designated SB3 although previously SRNL has called this new sludge batch SB2/3. A series of dry-fed tests (using the Melt Rate Furnace or MRF) and slurry fed tests (using the Slurry-Fed Melt Rate Furnace or SMRF) have been performed to investigate the effect of feed acid stoichiometry and REDOX (REDuction/OXidation) on the melt rate of the SB3 – Frit 418 system. The targeted waste loading for all of the feeds was 35%. The impact of waste loading had been previously investigated (Lorier and Smith, 2004) in the MRF. This previous report also documented the impact of acid stoichiometry in the MRF, but Frit 432 was used as there was no Frit 418 available at the time of the tests.

Tests were performed with non-radioactive, simulated SB3 material. Due to the small-scale of the test equipment and the design of the equipment, as well as the use of dry or slurry simulant feed, the behavior of the actual radioactive feed in the DWPF melter cannot be fully proven.

2.0 EXPERIMENTAL

2.1 4-L SRAT and 22-L SRAT/SME Feed Preparation

All Sludge Receipt and Adjustment Tank (SRAT) products for the MRF runs were fabricated at the Aiken County Technology Laboratory (ACTL). All feeds made for the MRF and SMRF tests used the same simulated sludge and they all had a targeted waste loading (WL) of 35%. The sludge was made at the Clemson Environmental Technologies Laboratory. It was a mixture of 40% SB2 and 60% SB3. Trim chemicals were added to achieve the final desired composition. The SRNL Mobile Lab sample ID for the SB2/SB3 simulated sludge is 04-0241 and the average results of two samples are given below in Table 2-1.

Table 2-1. SB3 Sludge Composition for all MRF and SMRF Acid Stoichiometry and REDOX Tests

Element	Weight % Calcined @ 1100 C		Element	Weight % Calcined @ 1100 C
Al	9.57		Mn	4.07
Ba	0.139		Na	14.05
Ca	2.37		Ni	1.06
Cr	0.153		Pb	0.01
Cu	0.157		S	0.35
Fe	28.35		Si	1.04
Gd	0.075		Zn	0.323
K	0.122		Zr	0.486
Mg	2.15			

Anion	Mg/Kg
HCO_2	< 100
NO_2	19050
NO_3	14150
SO_4	2215
C_2O_4	< 1000

Total % Solids	Insoluble % Solids	Wt% Calcined
22.6	15.4	16.3

For the MRF acid stoichiometry tests, one 4-L SRAT run was performed at the targeted acid stoichiometry (155 or 185%) and a REDuction/OXidation (REDOX) (expressed as $Fe^{2+}/\Sigma Fe$) target of 0.2 with Frit 418 (nominal and measured compositions listed in Table 2-2) later added for the targeted waste loading (WL). This same Frit 418 lot was used in the feeds used for all of the SMRF tests reported in this document as well.

For the MRF REDOX tests, three 4-L SRAT runs were performed at the target acid stoichiometry of 155% and a targeted REDOX of 0.0, 0.1, or 0.2. In addition, one 4-L SRAT batch was made for a 100% nitric MRF run and a 40% nitric MRF run (at a target of 155% acid stoichiometry as well). The predicted REDOX's of the 100% and 40% nitric acid runs were -0.35 and -1.3 respectively. For reference, a 155% acid stoichiometry feed for targeted REDOX's of 0.2, 0.1, and 0.0 have about 10, 12 and 15% nitric respectively for SB3. In other words, the 100% and 40% nitric acid feeds tested in the MRF were very high in the percentage of nitric acid used. It should be noted as well that DWPF would not be allowed to process 185% acid stoichiometry SB3 feed as it would exceed hydrogen generation safety limits.

Table 2-2. Nominal and Measured Compositions of Frit 418 Used for Melt Rate Tests

	FRIT 418*	
	Nominal	**Measured**
Oxide	**(wt%)**	**(wt)**
B_2O_3	8	8.05
Li_2O	8	7.91
Na_2O	8	7.99
SiO_2	76	75.32
Impurities	---	0.707
Total	100	99.977

*all Frit 418 used for the MRF and SMRF tests was from DWPF Lot 418 L2U

The feeds for the SMRF tests were made in the ACTL 22-L SRAT/SME (Slurry Mix Evaporator). Two 22-L runs were needed for each type of feed made. The same targeted acid stoichiometries and REDOX's from the MRF tests were used. A 0.2 REDOX feed was not made for the set of SMRF REDOX tests. This was because the SMRF 155% acid stoichiometry feed test previously discussed in this report had the required composition and feed preparation cycle (0.2 REDOX). 100% and 40% nitric acid feeds were not made for the SMRF after the 100% and 40% MRF tests indicated excessive off-gassing. Analyses of the SRAT and SME products used in the SMRF acid stoichiometry and REDOX tests are given in Appendix A. The calculated waste loadings for the 155% and 185% SMRF acid stoichiometry feeds were close to the targeted 35% (36.2 and 35.9% respectively). The calculated waste loadings for the SMRF 0.0 and 0.1 REDOX runs were close to target as well (36.6 and 36.2% respectively). All waste loadings were calculated by using the normalized lithia (Li_2O) values in the Frit 418 (see Table 2-2) and SME feeds tested.

The pH was monitored during the 22-L SRAT/SME REDOX feed preparation runs. The 0.0, 0.1, and 0.2 REDOX feeds had values in the 5-6 range. The pH of the 4-L 100% and 40% nitric acid run feeds had respective pH's of 0.8 and 4.

Additional details of the various feed preparation runs are given in Savannah River National Laboratory (SRNL) notebook WSRC-NB-2003-00046. Run plans for the 22-L SRAT/SME runs for the four feeds made are given in SRNL inter-office memorandums SRT-GPD-2004-00037, SRT-GPD-00038, SRT-GPD-2004-000040, and SRT-GPD-2004-00041.

2.2 Melt Rate Furnace Testing

The dry-fed MRF utilizes has a cylindrical inner chamber approximately 0.5 cubic feet in size, with heating coils winding around the chamber walls. The diameter of the chamber is ~7", and an insulating sleeve and a 1200 mL stainless steel beaker were inserted from the top. The tests were conducted with 6" deep stainless steel beakers inserted with the sleeve so that the beaker bottom was approximately flush with the top of the uppermost chamber coil. An insulating block was used to cover the beaker. The furnace was heated to 1150°C with the top opening covered. Once the furnace reached the setpoint, the cover was removed and the beaker containing sufficient dried, sieved material to produce 500 grams of glass was inserted. After 50 minutes, the beaker was removed from the furnace and allowed to slowly cool to room temperature. This residence time in the furnace was determined during testing in 2002 to establish a standard test time for melt rate comparison for this dry-fed furnace (Lorier et al. 2002).

The relative melt rate is determined by measuring the height of the glass layer in the bottom of the beaker at ¼" intervals. The average height and duration in the furnace is used to yield a relative linear melt rate number, with units of inches/hour. General observations of the sectioned beaker are also used to describe differences between runs. A volumetric estimate of melt rate is also calculated, but the linear method is the basis for comparison in this and other reports. In general, the volumetric and linear values show similar results.

2.3 SMRF Testing

The SMRF was heated up with 8 kg of a prefabricated SB3 Frit 418 glass targeting 35% waste loading. The run plans used for the SMRF tests were SRT-GPD-2004-00033 ("Run Plan for the SB2/SB3 Frit 418 Acid Stoichiometry Runs in the Slurry Fed Melter Rate Furnace") and SRT-GPD-2004-00062 ("Run Plan for the SB2/SB3 Frit 418 REDOX Runs in the Slurry Fed Melter Rate Furnace"). The SRNL log notebook used was WSRC-NB-2003-00163. After the acid stoichiometry tests were completed, the SMRF was drained and shut down. The glass drained from this test was used to charge the SMRF for the REDOX runs.

Details about the SMRF are documented elsewhere by Smith et al. (2003). The melt pool and vapor space setpoints were 1125°C and 750°C respectively. The time for each feed cycle after the vapor space had reached the vapor space feed initiation setpoint of 750°C was 20 seconds (37 seconds was needed for the 155% acid stoichiometry run at 50 weight percent solids to allow for enough of this thicker feed to be delivered to the SMRF in one feed cycle). Each feed was diluted from 50 to 45 weight percent solids to determine the impact of percent solids on melt rate. This was also done so that direct melt rate comparisons could be made for the same feeds at a lower weight percent solids. The measured amps for the melt pool and vapor space heaters were about 20 each for all of the tests. The tests were run basically the same as those for the SB2/SB3 Frit 418 (Case 6b – 250 canisters) 40% waste loading SMRF test performed in August 2003 (Smith et al. 2003).

2.3.1 SMRF Acid Stoichiometry Test Details

The SMRF was charged with 8 kg of startup glass on 4/12/04 and heated up to operating temperatures. The 155% acid stoichiometry test was performed on 4/13/04. The feed was fairly thick (see section on feed rheologies) and the feed cycle time had to be increased from 20 to 37 seconds to get about 70 grams/feed cycle (normally get 90-100 grams/20 second feed cycle). After about five hours of feeding the cold cap (basically dried out feed) had formed a mound that blocked the feed tube in the SMRF. This mound was knocked down as well as two others in the next 1.5 hours. At about 1500 the feed line plugged and the test was stopped. The feed was diluted from 50 to 45 weight percent solids (later analysis gave a measured weight percent solids of 45.5%). The cold cap was allowed to burn off for 1.5 hours. During this cold cap burn off time, a top of glass sample was taken (SMRF-0156). Feeding with the 45 weight percent solids feed was resumed at about 1630. The feed was easier to pump and the feed cycle time was reduced back to 20 seconds. Due to problems with plugging of the feed tank suction line inlet, the stable run time for this diluted feed was only about 2 hours.

The next day a top of glass sample was taken (SMRF-0160) which appeared to be foamy (many small bubbles). Feeding of the 185% acid stoichiometry feed (50 weight percent solids) was then started. Multiple suction line pluggages were experienced until the suction line slots were enlarged. No cold cap mounding occurred. At 1630 the feed line plugged and the cold cap was then allowed to burn off while the feed was diluted to 45 weight percent solids (measured at 45.2% after the test). No cold cap mounding was observed but one feed line plug occurred at 2010. The test was stopped at 2300. The next day a top of glass pool sample (SMRF-0164) was taken which was also observed to be foamy. The SMRF was then drained that same day.

2.3.2 SMRF REDOX Test Details

The SMRF was charged with about 6 kg of glass drained from the SMRF at the end of the acid stoichiometry tests and brought up to temperature on 5/10/04. Feeding of the 0.0 REDOX feed at 50 weight percent solids was started the next day at 0721 with 20 second feed cycles that delivered about 90-100 grams/feed cycle. The feed line plugged at 1050 and slight feed mounding (1" high) was noted at 1206. At 1354 the feed mound had reached the feed tube. It was knocked down, but then the mound built back up to the feed tube at 1420. After being knocked down again the mound was almost up to the feed tube at 1450. Feeding was stopped at 1500 while the feed was diluted to 45 weight percent solids (measured after test as 46.2%) and the cold cap allowed to burn off. Feeding was resumed at 1622 and the test stopped at 2205. No cold cap mounding was observed for this diluted feed but there was a feed line plug at 1820 that took 15 minutes to fix.

The next day a top of glass sample (SMRF-0169) was taken. The top of the glass pool appeared foamy and the top of glass sample had multiple small bubbles in it. Feeding of the 0.1 REDOX material at 50 weight percent solids was started the same day at 0720. At 1250 the cold cap had mounded up to the tip of the feed tube and at 1345 feeding was stopped. At 1555 the cold cap had mounded up to the feed tube again. At 1620 the pour stream was surging at high pour rates. Due to this and cold cap mounding, feeding was stopped at 1632 to allow the cold cap to burn off and dilute the remaining feed to 45 weight percent (measured after test as 46.0%). Feeding was then resumed at 1730. No mounding problems were observed after the feed dilution but a vapor space thermocouple failure resulted in about 30 minutes of not feeding. At 2152 the test was stopped.

On the next day top of glass pool temperature measurements were taken that showed that the top of glass pool temperature ranged from 850 to 940 ºC. A top of melt pool sample (SMRF-0173) was taken. This sample, like the others taken in these tests, had multiple small bubbles. The SMRF was drained and then powered down.

3.0 RESULTS AND DISCUSSION

3.1 Effects of Acid Stoichiometry – MRF Tests

Testing in the MRF indicated an increase in melt rate with increasing acid stoichiometry. For Frit 418 the estimated linear melt rate rose from 0.58 in/hr to 0.78 in/hr when the acid addition was increased from 155% to 185%. Using Frit 432, the linear melt rate rose in a similar fashion (Lorier and Smith, 2004). If, however, the results of the MRF 0.2 REDOX test discussed in section 3.3 (0.72 in/hr) are used (run at 155% acid stoichiometry and therefore should be same as the 155% acid stoichiometry discussed above), then there was no impact observed in the MRF tests with regards to acid stoichiometry. Due to these conflicting results, no conclusions can be made from the MRF tests with regards to the impact of acid stoichiometry on melt rate for the SB2/SB3 Frit 418 feed system.

3.2 Effects of Acid Stoichiometry – SMRF Tests

Details of the SMRF acid stoichiometry runs are given in section 2.3.1. Feeds with acid stoichiometries of 155% and 185% at 50 and 45 weight percent solids were tested. Table 3-3 summarizes the melt rates for these feeds.

Table 3-3. Melt Rates for SB3 Frit 418 Feeds at 155 and 185% Acid Stoichiometries

Targeted Acid Stoichiometry/Wt % Solids	Melt Rate (g/min)	Run Times Used to Determine Melt Rates
155/50	14.1	4/13/04 (1100-1500)
185/50	11.5	4/13/04 (2030-2247)
155/45	12.7	4/14/04 (1330-1630)
185/45	9.0	4/14/04 (1900-2300)

Unlike the MRF results, the SMRF results were fairly conclusive. The 155% acid stoichiometry feed resulted in higher melt rates than the 185% acid stoichiometry feed at both 50 and 45 weight percent solids. In addition, the 50 weight percent solids feeds had higher melt rates for both acid stoichiometries than at 45 weight percent solids. The SMRF results should be considered to be a better indicator of the impact of feed acid stoichiometry on DWPF melt rate because small scale slurry feeding is a better test method (versus dry fed tests) for melt rate for DWPF, especially when the targeted glass composition is not being altered.

As discussed in section 2.0 (SMRF Testing), glass and top of glass pool samples were taken during this test. A discussion of the top of glass pool samples is given in section 3.6. Analyses of startup glass, as well as various glass pour and drain samples are given in Appendix B**.** Table B-1 in Appendix B also shows that the REDOX values for the glasses produced were close to the target of 0.2.

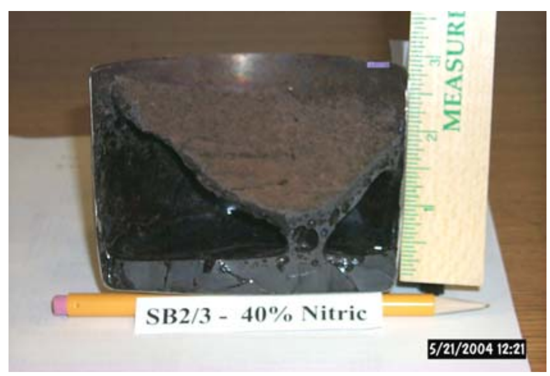
3.3 Effects of REDOX – MRF Tests

Three MRF runs were conducted at 0.0, 0.1, and 0.2 REDOX. There was no difference in the linear melt rate, with the rates being 0.73, 0.69, and 0.72 in/hr respectively. These tests were performed on product that had an elevated sulfate concentration. The sectioned beakers were similar in appearance. A second test was performed with 0.0 and 0.1 REDOX using the same sludge as used for the SMRF acid stoichiometry and REDOX tests (nominal sulfate concentration) and again there was no difference in melt rate between the two samples (0.59 in/hr for both). The reason for the lower melt rates cannot be attributed fully to the lower sulfate concentration but no other differences in the two sets of tests could be determined. If each set is looked at independently, the same conclusion can be made that melt rate was not impacted by REDOX in these MRF tests.

In addition to the above REDOX tests, 100% nitric and 40% nitric MRF tests were performed as well using nominal sulfate concentration SB3 sludge. As stated previously, the predicted REDOX's of the 100% and 40% nitric acid runs were -0.35 and -1.3 respectively. When the 100% nitric batch was placed in the MRF, brown smoke immediately began coming out the vent hole in the top. As the beaker heated, the smoke increased and was escaping from underneath the entire top cover of M-board. The crusty top layer rose to nearly the top of the beaker. Figure 3-1 shows that a very small layer of glass was formed in the bottom of the beaker during the heating cycle. Most of the material was incorporated in the layers of foam above the glass. The melt rate for the 100% nitric acid run (0.40 in/hr) was ~ 35% less than the 0.0and 0.1 REDOX from the second set of tests. This is to be expected with the large amount of material incorporated into the crusty layer in the top of the beaker.

The 40% nitric acid run also generated more brown smoke than the SB3-Frit 418 MRF runs with REDOX's targeted in the 0.0 to 0.2 range, but less than the 100% nitric test. The crusty layer rose in the beaker, but not to the extent that occurred in the 100% nitric run. Figure 3-1 below shows an improvement in the amount of glass formed compared to the 100% nitric run. The estimated melt rate was about the same (0.57 in/hr) that was achieved with the 0.0 and 0.1 REDOX testing (0.59 in/hr for both). Based on the behavior of the material in the MRF, there was no SMRF testing conducted with the 100% and 40% acid strategies.

Figure 3-1. Cross-Sectional Views of 100 and 40% Nitric Acid SB2/SB3 Frit 418 MRF Beakers



3.4 Effects of REDOX – SMRF Tests

Details of the SMRF REDOX runs are given in section 2.3.2. Feeds with targeted REDOX's of 0.0 and 0.1 (both at 155% acid stoichiometry) at 50 and 45 weight percent solids were tested. The melt rates from the previously run 155% acid stoichiometry SMRF test (see section 3.2) were used as the 0.2 REDOX test. Table 3-4 summarizes the melt rates for these feeds.

Table 3-4. Melt Rates for SB3 Frit 418 SMRF REDOX Tests

Targeted Feed REDOX/ Wt % Solids	Melt Rate (g/min)	Run Times Used to Determine Melt Rates
0.0/50	12.4	5/11/04 (1000-1330)
0.0/45	11.4	5/11/04 (2000-2300)
0.1/50	14.2	5/12/04 (1130-1530)
0.1/45	11.5*	5/12/04 (1900-2000)
0.2/50	14.1	4/13/04 (1100-1500)
0.2/45	12.7	4/14/04 (1330-1630)

*short run time of only one hour due to vapor space thermocouple failure

The melt rates for the 0.2 and 0.1 REDOX feeds (at 50 weight percent solids) were about the same (14.1 versus 14.2 g/min respectively). The melt rate was somewhat lower (12.4 g/min), however, for the 0.0 REDOX feed at the same weight percent solids. This may be because almost all of the MnO_2 or Mn_2O_3 goes to MnO at a REDOX 0.09. This then results in the evolution of more oxygen in the cold cap (causing foaming) which negatively impacts melt rate (Jantzen and Plodinec, 1986)(Schreiber and Hockman, 1987). As with the acid stoichiometry feeds, the melt rates were all lower when the feeds were diluted to 45 weight percent solids as compared to their 50 weight percent counterparts at a constant REDOX. For the 45 weight percent solids, the 0.0 and 0.1 REDOX melt rates were about the same and lower than the 0.2 REDOX feed. Due to short stable run time for the 0.1 REDOX feed at 45 weight percent solids, the actual melt rate may not have been accurately determined and therefore caution should be taken when using the resultant melt rate from that test.

3.5 Feed Rheology

Samples of the SME products for the 155% and 185% acid stoichiometry SMRF runs, as well as the 0.0 and 0.1 REDOX runs were taken and rheological properties were measured. Figure 3-2 shows the 155% and 185% rheogram. Figure 3-3 shows the 0.0 and 0.1 REDOX feed rheograms results and also plots the 155% acid stoichiometry feed rheogram (same as a 0.2 REDOX feed). All of the feeds plotted were at about 50 weight percent solids.

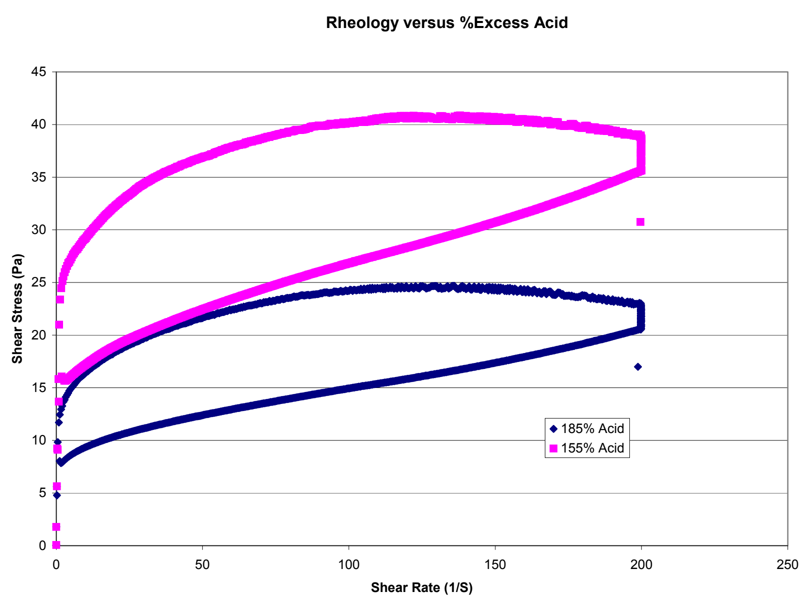


Figure 3-2. Rheogram Plots of the SMRF 155% and 185% Acid Stoichiometry Feeds

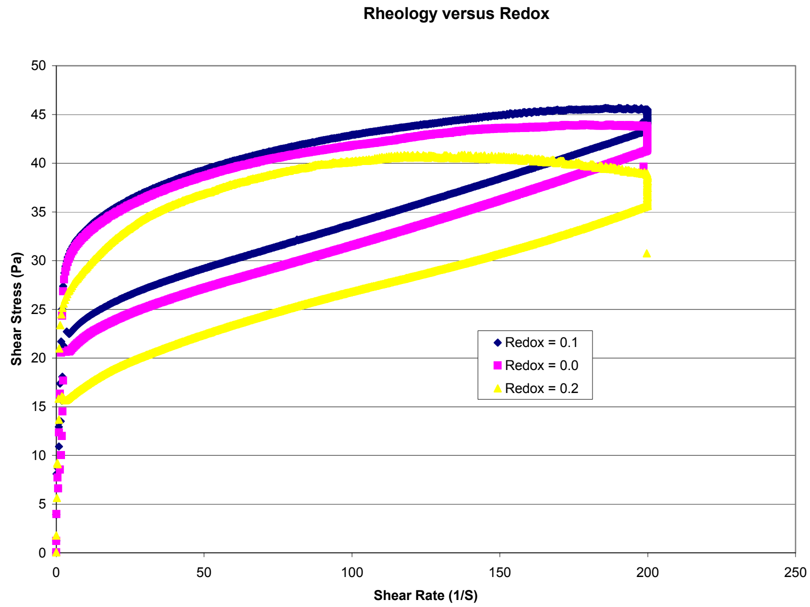


Figure 3-3. Rheogram Plots of the SMRF 0.0, 0.1, and 0.2 REDOX Feeds

Figure 3-2 shows that the 185% acid stoichiometry feed had a lower sheer stress than the 155% acid stoichiometry feed. This agrees with the SMRF feeding observations given in section 2.3.1 of this report. It was difficult to pump the 155% acid stoichiometry feed at the desired amount per feed cycle and cold cap mounding was observed as well. However, the 155% acid stoichiometry feed had a higher melt rate in spite of these problems. Figure 3-3 shows that the shear stress of the feed was not greatly impacted by REDOX. This agrees with the feedings observations given in section 2.3.2 of this report.

3.6 SMRF Top of Glass Pool Samples

After each SMRF test was completed in the evening (usually 2200 to 2300), the SMRF was idled overnight and then a top of glass pool was taken the next morning before the next test was started. As previously noted, the temperature of the top of glass measured the day after the 0.1 REDOX test ranged from 850 to 940 ºC.

Each of the top of glass samples taken was foamy. The top of glass samples taken after the 155% and 185% acid stoichiometry tests (samples SMRF-0160 and SMRF-0164 respectively) were submitted for Scanning Electron Microscopy/ Energy Dispersive Spectroscopy (SEM/EDS) analysis. In addition, the samples were analyzed by X-Ray Diffraction (XRD) to determine what crystalline phases were present. Figure 3-4 is a picture of sample SMRF-0164 which shows the foamy nature of the top of the glass after feeding has been stopped for about eight hours. The appearance of this top of glass sample was typical of the others taken.

Figure 3-4. Foamy SMRF Top of Glass Sample (SMRF-0164)

Figures 3-5 through 3-8 are SEM Micrographs of the SMRF-0160 top of glass sample. Figure 3-5 is a micrograph of a section of the sample and the next three are micrographs of higher magnifications portions of the section shown in Figure 3-5. EDS's of the various spots noted are given in Appendix C and are used to determine the elements present in the various spots analyzed. Spot 1 in Figure 3-6 is a phase composed of Fe, Si, Mn, Cr, and Ni (typical spinel components). Spot 2 appears to be glass. Spot 5 in Figure 3-7 is mostly Pd while Spot 7 is composed of Ru, Rh, Si, and Fe. In Figure 3-8, Spots 8, 9, and 10 are phases consisting of various amounts Ag, Cu and S. Figure 3-9 is an XRD plot of SMRF-0160 showing the presence of the crystalline phases trevorite ($NiFe_2O_4$) and aegerine ($NaFe^{3+}Si_2O_6$) confirming the presence of spinels in the cold cap sample.

This foamy layer can negatively impact melt rate by acting as an insulating layer between the molten glass and the cold cap. The resultant lower temperatures in the upper region of the melt pool (850 to 940 ºC as previously cited) would allow for the formation of these crystalline phases.

Figure 3-5. SEM Micrograph of Top of Glass Sample SMRF-0160

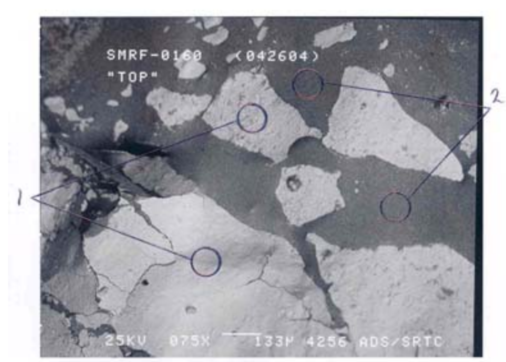


Figure 3-6. SEM Micrograph (Higher Magnification) of SMRF-0160 (Photo 4256)

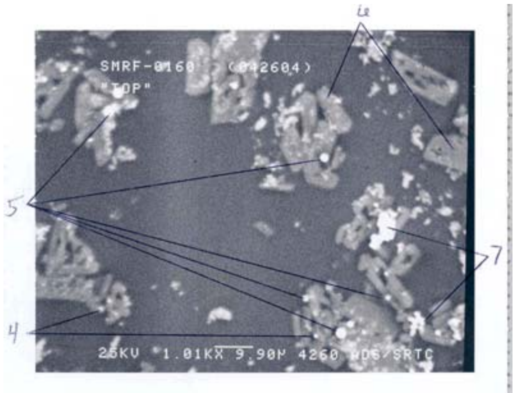


Figure 3-7. SEM Micrograph (Higher Magnification) of SMRF-0160 (Photo 4260)

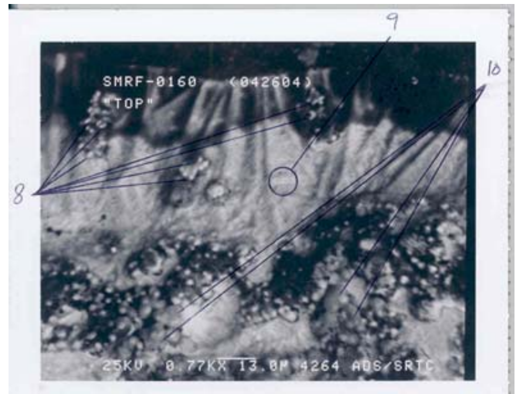


Figure 3-8. SEM Micrograph (Higher Magnification) of SMRF-0160 (Photo 4264)

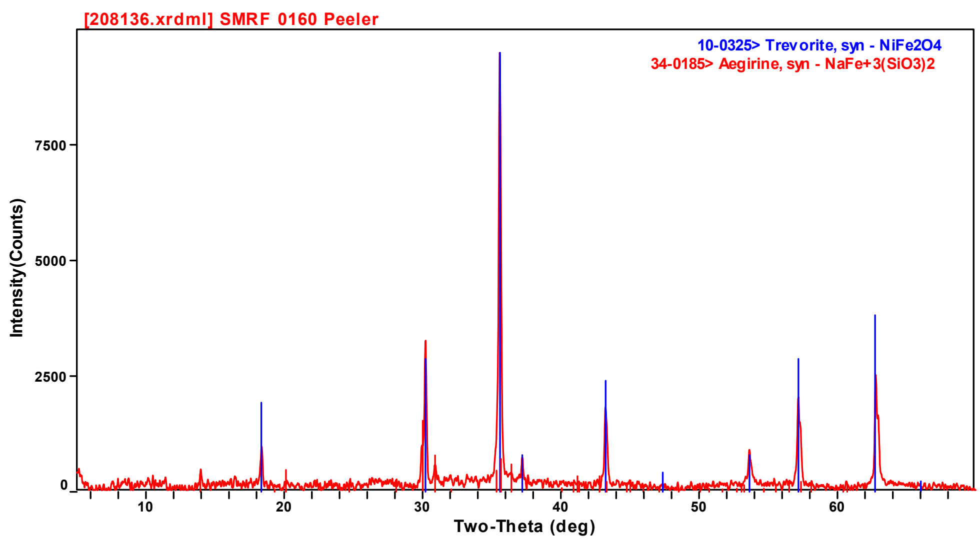


Figure 3-9. XRD of Top of Glass Sample SMRF-0160

4.0 CONCLUSIONS

A series of dry-fed tests (using the Melt Rate Furnace or MRF) and slurry fed tests (using the Slurry-Fed Melt Rate Furnace or SMRF) have been performed to investigate the effect of feed preparation acid stoichiometry or REDOX (REDuction/OXidation) on the melt rate of the SB3 – Frit 418 system. This objective was met and the following conclusion can be made based on this work:

- Melt rate results from the MRF tests contradicted the SMRF results (see SMRF acid stoichiometry test conclusion below) for the impact feed acid stoichiometry. The differences in results from the MRF and SMRF may be a parameter other than final glass product (acid stoichiometry) was investigated which may not be able to be evaluated in dry feed testing (MRF).
- Melt rate results from the MRF REDOX tests indicated no impact of REDOX (0.0, 0.1, and 0.2 REDOX feeds tested). This did not agree with the SMRF tests as discussed below. As with the MRF acid stoichiometry tests, the differences in results from the MRF and SMRF REDOX tests may be because a parameter other than final glass product was investigated which may not be able to be evaluated in dry feed testing (MRF).
- Due to the MRF results, caution should be taken when trying to use to MRF as a melt rate tool when parameters other than final glass composition are being tested.
- For the SMRF tests, the current DWPF target feed preparation acid stoichiometry of 155% gave a higher melt rate than 185%.
- MRF tests with 40% and 100% nitric acid feed (resulting in negative calculated feed REDOX's) showed high amounts of off-gassing and a much lower melt rate for the 100% nitric acid feed. Due to the off-gassing observations, these feeds were not tested in the SMRF as part of the REDOX runs.
- Feeds with targeted REDOX's of 0.0, 0.1, and 0.2 were tested in the SMRF. These tests indicate that melt rate was not negatively impacted until the targeted feed REDOX dropped below 0.1. It is postulated that this is due to the fact that the oxidation state of manganese increases at a REDOX of 0.09 and lower, thereby resulting in a foamy cold cap/glass.
- The dilution of the SMRF feed from 50 to 45 weight percent solids results in lower melt rates for all acid stoichiometry (155% and 185%) and REDOX feeds tested (0.2, 0.1, and 0.0).
- SMRF top of glass samples taken at various times after cold cap burn off were found to be foamy. The crystalline phases trevorite and aegerine were present in these samples as identified by XRD.

5.0 RECOMMENDATIONS/PATH FORWARD

- It is recommended that DWPF continue to use the targets of 155% acid stoichiometry and 0.2 REDOX for SRAT feed preparation of the SB3 – Frit 418 system.
- Continue investigating melt rate tools that can give relative differences in feeds with regards to off-gas surges and melt reactions.
- Investigate reproducibility of MRF by running multiple tests with identical feed.

6.0 REFERENCES

Jantzen, CM and MJ Plodinec. 1986. *Composition and Redox Control of Waste Glasses: Recommendation for Process Control Limit(U),* DPST-86-773, E. I. duPont deNemours & Co., Savannah River Laboratory, Aiken, South Carolina.

Lorier, TH, TM Jones, and DC Witt. 2002. *Melt Rate Testing for the DWPF: Summary of FY02 Testing (U),* WSRC-TR-2002-00545, Westinghouse Savannah River Company, Aiken, South Carolina.

Lorier, TH and ME Smith. 2004. *Melt Rate Assessment of SB3with Frit 418 – Effects of Waste Loading and Acid Addition (U),* WSRC-TR-2004-00098, Westinghouse Savannah River Company, Aiken, South Carolina.

Schreiber, HD and AL Hockman, 1987. *Redox Chemistry in Candidate Glasses for Nuclear Waste Immobilization,* Journal of the American Ceramic Society, Vol. 70, No. 8, pp. 591-594.

Smith, ME, TH Lorier, and TM Jones. 2003. *SMRF and MRF DWPF Melt Rate Testing for SB2/SB3 (Case 6b – 250 Canisters) (U)*, WSRC-TR-2003-00466, Westinghouse Savannah River Company, Aiken, South Carolina.

APPENDIX A – SRAT/SME ANALYSES

Sample ID	Lab ID	Al	B	Ba	Ca	Cr	Cu	Fe	Gd	K	Li	Mg	Mn	Na	Ni	Pb	S	Si	Zn	Zr
elemental wt%-calcined 1100C																				
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (A)	04-0251	3.46	1.68	0.043	0.709	0.061	0.062	10.5	0.024	0.075	2.37	0.899	1.34	9.78	0.325	0.032	0.104	23.4	0.109	0.178
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (B)	04-0251	3.42	1.64	0.043	0.712	0.062	0.056	10.4	0.024	0.069	2.36	0.929	1.37	9.55	0.347	0.033	0.109	23.6	0.110	0.184
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (A)	04-0252	3.39	1.61	0.042	0.688	0.062	0.051	10.1	0.024	0.070	2.33	0.915	1.36	9.78	0.332	0.032	0.113	23.3	0.108	0.178
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (B)	04-0252	3.37	1.62	0.043	0.701	0.062	0.047	10.6	0.024	0.072	2.40	0.905	1.34	9.73	0.332	0.032	0.113	23.6	0.109	0.179
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (A)	04-0253	3.42	1.63	0.041	0.659	0.060	0.049	10.5	0.023	0.074	2.39	0.909	1.35	9.66	0.335	0.030	0.124	23.7	0.106	0.171
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (B)	04-0253	3.39	1.65	0.042	0.708	0.061	0.050	10.5	0.023	0.069	2.38	0.892	1.33	9.67	0.329	0.031	0.127	23.6	0.109	0.175
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (A)	04-0254	3.36	1.62	0.042	0.667	0.062	0.055	10.7	0.024	0.070	2.39	0.861	1.29	9.63	0.322	0.032	0.122	23.7	0.108	0.186
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (B)	04-0254	3.33	1.63	0.045	0.662	0.063	0.061	10.7	0.025	0.060	2.39	0.896	1.34	9.59	0.325	0.034	0.124	23.6	0.111	0.184

oxide wt% - calcined 1100C	Lab ID	Al2O3	B2O3	BaO	CaO	Cr2O3	CuO	Fe2O3	Gd2O3	K2O	Li2O	MgO	MnO	Na2O	NiO	PbO	SO4	SiO2	ZnO	ZrO2	Totals	Li20 Normalized
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (A)	04-0251	6.54	5.41	0.048	0.993	0.089	0.078	15.0	0.027	0.089	5.10	1.49	1.73	13.2	0.413	0.035	0.313	50.1	0.135	0.241	101.0	5.04
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (B)	04-0251	6.46	5.27	0.048	0.997	0.090	0.070	14.9	0.028	0.083	5.07	1.54	1.77	12.9	0.441	0.036	0.328	50.5	0.136	0.248	100.9	5.03
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (A)	04-0252	6.41	5.18	0.047	0.963	0.090	0.064	14.4	0.027	0.084	5.01	1.52	1.75	13.2	0.422	0.034	0.338	49.9	0.134	0.240	99.8	5.02
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (B)	04-0252	6.37	5.22	0.048	0.98	0.091	0.059	15.2	0.027	0.086	5.16	1.50	1.73	13.1	0.421	0.034	0.338	50.5	0.136	0.242	101.2	5.10
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (A)	04-0253	6.46	5.26	0.046	0.923	0.088	0.061	15.0	0.026	0.088	5.14	1.51	1.74	13.0	0.426	0.032	0.371	50.7	0.132	0.231	101.3	5.07
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (B)	04-0253	6.41	5.31	0.047	0.991	0.090	0.063	15.0	0.027	0.083	5.12	1.48	1.72	13.1	0.418	0.034	0.381	50.5	0.135	0.237	101.1	5.06
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (A)	04-0254	6.35	5.21	0.048	0.93	0.090	0.069	15.3	0.027	0.084	5.14	1.43	1.66	13.0	0.409	0.035	0.365	50.7	0.134	0.251	101.3	5.07
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (B)	04-0254	6.29	5.24	0.050	0.93	0.093	0.076	15.3	0.028	0.072	5.14	1.49	1.73	12.9	0.413	0.036	0.373	50.5	0.137	0.248	101.1	5.08

Anions (mg/Kg)	Lab ID	Cl	NO2	NO3	SO4 (H2O)	SO4 acid	SO4 ICP-AES	HCO2	C2O4
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (A)	04-0251	104	<100	24100	1404	1570	1300	55400	<1000
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (B)	04-0251	98.5	<100	22800	1327	1600	1360	52400	<1000
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (A)	04-0252	104	<100	24300	1471	2200	1390	54900	<1000
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (B)	04-0252	96.1	<100	22900	1337	2210	1400	52800	<1000
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (A)	04-0253	101	<100	26400	1138	1640	1500	55400	<1000
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (B)	04-0253	96.0	<100	25800	1030	1800	1550	54400	<1000
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (A)	04-0254	96.9	<100	25100	1305	1800	1470	50700	<1000
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (B)	04-0254	92.4	<100	25000	1233	1740	1500	51000	<1000

155% Avg Li2O	185% Avg Li2O	Frit 418 Normalized Li2O (Lot L2U)
5.05	5.07	7.91

155% WL	185% WL	155 Wt% Solids	185 Wt% Solids
36.2	35.9	49.6	48.6

Weight % Solids Calculations

Sample	Lab ID	Empty Crucible wt	Crucible Wt + Wet Sample	Crucible Wt + Dry wt	Total Solids	Wet Wt	Dry Wt	Insoluble Solids	Cruc Wt+ Calcined	Wt % Calcined
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (A)	04-0251	43.989	48.7989	46.3776	49.7%	4.8099	2.389	38.4%	45.9858	41.5%
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (B)	04-0251	44.6864	50.35	47.4967	49.6%	5.6636	2.810	38.5%	47.0361	41.5%
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (A)	04-0252	45.1076	50.1844	47.6151	49.4%	5.0768	2.508	38.3%	47.1966	41.1%
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (B)	04-0252	44.6558	49.8691	47.2397	49.6%	5.2133	2.584	38.5%	46.8143	41.4%
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (A)	04-0253	45.142	51.311	48.1574	48.9%	6.1690	3.015	38.1%	47.6341	40.4%
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (B)	04-0253	45.5576	52.0854	48.7562	49.0%	6.5278	3.199	38.2%	48.2087	40.6%
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (A)	04-0254	45.1191	51.409	48.1612	48.4%	6.2899	3.042	37.3%	47.6521	40.3%
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (B)	04-0254	43.4896	50.0897	46.6736	48.2%	6.6001	3.184	37.2%	46.1373	40.1%

NOTE: All Li2O and WL calculations verified via hand calculations
WL's calculated w/ normalized Li2O values in feed and Frit 418 (Lot L2U)

Sample	Lab ID	Empty Crucible wt	Crucible Wt + Wet Sample	Crucible Wt + Dry wt	Uncorr	Soluble Solids	Density	pH
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (A)	04-0251	44.8558	45.9976	45.0651	18.3%	11.3%	1.60	6.55
SB2/3 SME Product 155% Acid Run 1:FMPR-2A (B)	04-0251	45.0381	46.1781	45.2439	18.1%	11.1%		
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (A)	04-0252	42.9286	44.0760	43.1342	17.9%	11.0%	1.60	6.36
SB2/3 SME Product 155% Acid Run 2:FMPR-4A (B)	04-0252	45.3641	46.5207	45.5717	17.9%	11.0%		
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (A)	04-0253	42.5647	43.7027	42.7635	17.5%	10.8%	1.48	4.93
SB2/3 SME Product 185% Acid Run 1:FMPR-6A (B)	04-0253	44.5798	45.7099	44.7777	17.5%	10.8%		
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (A)	04-0254	44.4219	45.5639	44.6232	17.6%	11.0%	1.53	5.33
SB2/3 SME Product 185% Acid Run 2:FMPR-8A (B)	04-0254	44.0258	45.1243	44.2192	17.6%	11.1%		

Table A-1. SMRF SB3 Frit 418 Acid Stoichiometry SRAT and SME Product Analytical Results

Sample ID	Lab ID																			
elemental wt%-calcined 1100C		Al	B	Ba	Ca	Cr	Cu	Fe	Gd	K	Li	Mg	Mn	Na	Ni	Pb	S	Si	Zn	Zr
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (A)	04-0400	8.78	<0.010	0.121	1.94	0.149	0.127	28.1	0.067	0.144	<0.010	2.68	3.90	14.9	1.08	<0.010	0.424	1.11	0.312	0.542
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (B)	04-0400	8.89	<0.010	0.125	1.90	0.149	0.128	28.6	0.070	0.125	<0.010	2.63	3.93	14.6	1.11	<0.010	0.431	1.19	0.319	0.481
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (A)	04-0401	8.77	<0.010	0.117	2.02	0.142	0.162	27.6	0.066	0.134	<0.010	2.51	3.88	14.2	1.07	0.020	0.414	2.06	0.303	0.452
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (B)	04-0401	8.55	<0.010	0.121	1.89	0.146	0.169	27.9	0.067	0.119	<0.010	2.57	3.90	14.3	1.08	0.021	0.422	2.02	0.308	0.474
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (A)	04-0402	3.30	1.50	0.047	0.675	0.062	0.058	10.2	0.026	0.069	2.31	0.953	1.39	8.74	0.378	0.036	0.105	23.3	0.112	0.197
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (B)	04-0402	3.31	1.56	0.045	0.667	0.054	0.058	10.3	0.026	0.073	2.33	0.941	1.40	8.83	0.379	0.034	0.102	23.5	0.109	0.187
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (A)	04-0403	3.39	1.65	0.045	0.793	0.075	0.056	10.1	0.025	0.063	2.33	1.03	1.45	9.66	0.375	0.036	0.129	23.5	0.111	0.177
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (B)	04-0403	3.19	1.51	0.044	0.695	0.061	0.056	9.93	0.025	0.067	2.26	1.02	1.42	9.49	0.369	0.036	0.129	22.9	0.111	0.179

oxide wt% - calcined 1100C	Lab ID	Al2O3	B2O3	BaO	CaO	Cr2O3	CuO	Fe2O3	Gd2O3	K2O	Li2O	MgO	MnO	Na2O	NiO	PbO	SO4	SiO2	ZnO	ZrO2	Totals	Li2O Normalized
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (A)	04-0400	16.6	0.00	0.136	2.72	0.218	0.159	40.2	0.077	0.173	0.00	4.45	5.03	20.1	1.37	0.000	1.27	2.38	0.387	0.732	96.0	
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (B)	04-0400	16.8	0.00	0.140	2.66	0.218	0.160	40.9	0.081	0.150	0.00	4.37	5.07	19.7	1.41	0.000	1.29	2.55	0.396	0.649	96.5	
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (A)	04-0401	16.6	0.00	0.131	2.83	0.207	0.203	39.5	0.076	0.161	0.00	4.17	5.01	19.2	1.36	0.022	1.24	4.41	0.376	0.610	96.0	
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (B)	04-0401	16.2	0.00	0.136	2.65	0.213	0.211	39.9	0.077	0.143	0.00	4.27	5.03	19.3	1.37	0.023	1.27	4.32	0.382	0.640	96.1	
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (A)	04-0402	6.24	4.83	0.053	0.945	0.091	0.073	14.6	0.030	0.083	4.97	1.58	1.79	11.8	0.480	0.039	0.315	49.9	0.139	0.266	98.2	5.06
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (B)	04-0402	6.26	5.02	0.050	0.934	0.079	0.073	14.7	0.030	0.088	5.01	1.56	1.81	11.9	0.481	0.037	0.306	50.3	0.135	0.252	99.1	5.06
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (A)	04-0403	6.41	5.31	0.050	1.11	0.110	0.070	14.4	0.029	0.076	5.01	1.71	1.87	13.0	0.476	0.039	0.387	50.3	0.138	0.239	101	4.97
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (B)	04-0403	6.03	4.86	0.049	0.97	0.089	0.070	14.2	0.029	0.080	4.86	1.69	1.83	12.8	0.469	0.039	0.387	49.0	0.138	0.242	97.9	4.97

Anions (mg/Kg)	Lab ID	NO2	NO3	SO4	HCO2
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (A)	04-0400	<100	44200	1840	48900
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (B)	04-0400	<100	44600	1860	48500
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (A)	04-0401	<100	38900	1970	46500
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (B)	04-0401	<100	37600	1990	45900
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (A)	04-0402	<100	37600	1830	36500
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (B)	04-0402	<100	35900	1730	38400
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (A)	04-0403	<100	34700	1770	44500
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (B)	04-0403	<100	35500	1750	45700

Frit 418 Normalized Li2O (Lot L2U): 7.91

Avg Norm 0 REDOX Li2O: 5.01

0 REDOX WL: 36.6

0 REDOX Weight %: 49.3

Weight % Solids Calculations

Sample	Lab ID	Empty Crucible wt	Crucible Wt + Wet Sample	Crucible Wt + Dry wt	Total Solids	Wet Wt	Dry Wt	nsoluble Solid	Cruc Wt+ Calcined	Wt % Calcined
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (A)	04-0400	40.233	46.1349	41.8284	27.0%	5.9019	1.595	13.8%	41.2363	17.0%
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (B)	04-0400	44.0251	49.9334	45.6243	27.1%	5.9083	1.599	13.8%	45.0277	17.0%
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (A)	04-0401	42.9272	48.8445	44.5392	27.2%	5.9173	1.612	14.5%	43.9700	17.6%
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (B)	04-0401	45.4997	51.5044	47.1316	27.2%	6.0047	1.632	14.2%	46.5552	17.6%
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (A)	04-0402	37.3373	42.4596	39.8724	49.5%	5.1223	2.535	37.9%	39.4240	40.7%
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (B)	04-0402	42.4838	48.3369	45.3856	49.6%	5.8531	2.902	37.9%	44.8731	40.8%
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (A)	04-0403	40.8215	45.8282	43.2808	49.1%	5.0067	2.459	37.6%	42.8510	40.5%
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (B)	04-0403	44.3862	50.2306	47.2579	49.1%	5.8444	2.872	37.6%	46.7562	40.6%

Sample	Lab ID	Empty Crucible wt	Crucible Wt + Wet Sample	Crucible Wt + Dry wt	Uncorr	Soluble Solids	Density	pH
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (A)	04-0400	43.6002	44.6861	43.7666	15.3%	13.2%	1.31	6.98
SB2/3 SRAT Product Redox = 0.0 Run 1 : FPMR-13A (B)	04-0400	43.9912	45.0998	44.1622	15.4%	13.3%		
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (A)	04-0401	43.7663	44.8741	43.9312	14.9%	12.7%	1.27	7.28
SB2/3 SRAT Product Redox = 0.0 Run 2 : FPMR-15A (B)	04-0401	43.2753	44.3808	43.4425	15.1%	13.0%		
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (A)	04-0402	44.0563	45.1877	44.2676	18.7%	11.6%	1.44	6.71
SB2/3 SME Product Redox = 0.0 Run 1 : FPMR-14A (B)	04-0402	42.3607	43.4902	42.5735	18.8%	11.7%		
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (A)	04-0403	44.1185	45.2556	44.3278	18.4%	11.5%	1.43	6.94
SB2/3 SME Product Redox = 0.0 Run 2 : FPMR-16A (B)	04-0403	44.1330	45.2660	44.3418	18.4%	11.5%		

Table A-2. SMRF SB3 Frit 418 0 REDOX SRAT and SME Product Analytical Results

Sample ID	Lab ID																					
elemental wt%-calcined 1100C		Al	B	Ba	Ca	Cr	Cu	Fe	Gd	K	Li	Mg	Mn	Na	Ni	Pb	S	Si	Zn	Zr		
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (A)	**04-0396**	9.19	<0.010	0.126	2.03	0.160	0.141	28.8	0.069	0.125	<0.010	2.56	4.07	14.4	1.11	0.011	0.438	1.14	0.321	0.478		
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (B)	**04-0396**	8.93	<0.010	0.129	1.94	0.162	0.142	28.4	0.071	0.120	<0.010	2.54	3.98	14.4	1.09	0.012	0.438	1.13	0.324	0.486		
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (A)	**04-0397**	8.72	<0.010	0.121	1.89	0.144	0.175	27.0	0.067	0.099	<0.010	2.42	3.83	13.6	1.04	0.041	0.421	3.58	0.306	0.445		
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (B)	**04-0397**	8.79	<0.010	0.114	1.96	0.143	0.157	26.9	0.064	0.118	<0.010	2.40	3.81	13.8	1.04	0.035	0.403	3.57	0.302	0.435		
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (A)	**04-0398**	3.24	1.58	0.046	0.674	0.056	0.049	10.0	0.026	0.065	2.29	0.995	1.40	8.92	0.374	0.036	0.090	22.9	0.115	0.179		
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (B)	**04-0398**	3.36	1.54	0.045	0.664	0.055	0.056	10.2	0.026	0.077	2.31	1.00	1.42	8.96	0.389	0.035	0.089	23.2	0.11	0.176		
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (A)	**04-0399**	3.31	1.54	0.046	0.670	0.056	0.061	10.2	0.026	0.074	2.31	1.00	1.43	8.85	0.379	0.036	0.108	23.4	0.121	0.180		
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (B)	**04-0399**	3.33	1.54	0.047	0.679	0.059	0.062	10.1	0.027	0.066	2.31	1.00	1.42	8.91	0.373	0.037	0.108	23.2	0.113	0.188		
																						Li2O Normalized
oxide wt% - calcined 1100C		Al2O3	B2O3	BaO	CaO	Cr2O3	CuO	Fe2O3	Gd2O3	K2O	Li2O	MgO	MnO	Na2O	NiO	PbO	SO4	SiO2	ZnO	ZrO2	Totals	
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (A)	**04-0396**	17.4	0.00	0.141	2.84	0.234	0.176	41.2	0.079	0.150	0.00	4.25	5.25	19.4	1.41	0.012	1.31	2.44	0.398	0.645	97.3	
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (B)	**04-0396**	16.9	0.00	0.144	2.72	0.237	0.178	40.6	0.082	0.144	0.00	4.22	5.13	19.4	1.38	0.013	1.31	2.42	0.402	0.656	96.0	
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (A)	**04-0397**	16.5	0.00	0.136	2.65	0.210	0.219	38.6	0.077	0.119	0.00	4.02	4.94	18.4	1.32	0.044	1.26	7.66	0.379	0.601	97.1	
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (B)	**04-0397**	16.6	0.00	0.128	2.74	0.209	0.196	38.5	0.074	0.142	0.00	3.98	4.91	18.6	1.32	0.038	1.21	7.64	0.374	0.587	97.3	
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (A)	**04-0398**	6.12	5.09	0.052	0.94	0.082	0.061	14.3	0.030	0.078	4.92	1.65	1.81	12.0	0.475	0.039	0.270	49.0	0.143	0.242	97.4	5.06
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (B)	**04-0398**	6.35	4.96	0.050	0.93	0.080	0.070	14.6	0.030	0.092	4.97	1.66	1.83	12.1	0.494	0.038	0.267	49.6	0.136	0.238	98.5	5.04
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (A)	**04-0399**	6.26	4.96	0.052	0.94	0.082	0.076	14.6	0.030	0.089	4.97	1.66	1.84	11.9	0.481	0.039	0.324	50.1	0.150	0.243	98.8	5.03
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (B)	**04-0399**	6.29	4.96	0.053	0.95	0.086	0.078	14.4	0.031	0.079	4.97	1.66	1.83	12.0	0.474	0.040	0.324	49.6	0.140	0.254	98.3	5.05

Anions (mg/Kg)		NO2	NO3	SO4	HCO2																	
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (A)	**04-0396**	<100	35900	1800	54200							**Frit 418 Normalized Li2O(Lot L2U)**					**Avg Norm. 0.1 Li2O**				**0.1 REDOX WL**	
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (B)	**04-0396**	<100	35900	1720	56300								7.91					5.04			36.2	
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (A)	**04-0397**	<100	31300	1910	50500																	
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (B)	**04-0397**	<100	31000	1920	50400																	
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (A)	**04-0398**	<100	29000	1660	45100																	
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (B)	**04-0398**	<100	28500	1720	43200											**0.1 REDOX Weight %**						
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (A)	**04-0399**	<100	27500	1750	41600											49.2						
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (B)	**04-0399**	<100	27200	1710	45500																	

Weight % Solids Calculations

		Empty	Crucible Wt +	Crucible Wt +					Cruc Wt+	Wt %
Sample	Lab ID	Crucible wt	Wet Sample	Dry wt	**Total Solids**	Wet Wt	Dry Wt	**soluble Solid**	Calcined	Calcined
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (A)	**04-0396**	44.397	50.1862	45.9521	**26.9%**	5.7892	1.555	**13.7%**	45.3831	**17.0%**
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (B)	**04-0396**	44.5777	50.5454	46.1818	**26.9%**	5.9677	1.604	**13.8%**	45.5968	**17.1%**
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (A)	**04-0397**	45.3636	50.279	46.7047	**27.3%**	4.9154	1.341	**14.7%**	46.2476	**18.0%**
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (B)	**04-0397**	44.3224	50.3036	45.9592	**27.4%**	5.9812	1.637	**14.9%**	45.4017	**18.0%**
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (A)	**04-0398**	44.0282	49.1504	46.5593	**49.4%**	5.1222	2.531	**37.9%**	46.1209	**40.9%**
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (B)	**04-0398**	42.5172	49.6028	46.02	**49.4%**	7.0856	3.503	**38.0%**	45.4163	**40.9%**
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (A)	**04-0399**	42.8576	48.0044	45.3718	**48.8%**	5.1468	2.514	**37.7%**	44.9407	**40.5%**
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (B)	**04-0399**	41.7609	49.7581	45.6716	**48.9%**	7.9972	3.911	**37.9%**	45.0054	**40.6%**

		Empty	Crucible Wt +	Crucible Wt +					
Sample	Lab ID	Crucible wt	Wet Sample	Dry wt	Uncorr	**oluble Solids**		**Density**	**pH**
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (A)	**04-0396**	43.1483	44.2071	43.3096	15.2%	**13.1%**		**1.36**	**6.55**
SB2/3 SRAT Product Redox = 0.1 Run 1 : FPMR-9A (B)	**04-0396**	43.8646	44.9692	44.0326	15.2%	**13.1%**			
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (A)	**04-0397**	44.7317	45.7777	44.8860	14.8%	**12.6%**		**1.33**	**7.09**
SB2/3 SRAT Product Redox = 0.1 Run 2 : FPMR-11A (B)	**04-0397**	44.6107	45.7093	44.7718	14.7%	**12.5%**			
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (A)	**04-0398**	43.0828	44.1955	43.2884	18.5%	**11.5%**		**1.44**	**6.73**
SB2/3 SME Product Redox = 0.1 Run 1 : FPMR-10A (B)	**04-0398**	42.4972	43.6023	42.7011	18.5%	**11.4%**			
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (A)	**04-0399**	43.9912	45.1131	44.1915	17.9%	**11.1%**		**1.41**	**6.92**
SB2/3 SME Product Redox = 0.1 Run 2 : FPMR-12A (B)	**04-0399**	42.2318	43.3556	42.4315	17.8%	**11.0%**			

Table A-3. SMRF SB3 Frit 418 0.1 REDOX SRAT and SME Product Analytical Results

APPENDIX B – SMRF GLASS SAMPLE ANALYSES

Sample ID	Lab ID																								
elemental wt%-calcined 1100C		Al	B	Ba	Ca	Cr	Cu	Fe	Gd	K	Li	Mg	Mn	Na	Ni	Pb	Pd	Rh	Ru	S	Si	Ti	Zn	Zr	
SMRF 0156 (A)	**04-0391**	3.35	1.64	0.044	1.64	0.078	0.059	10.1	0.025	0.079	2.36	0.945	1.47	10.5	0.423	0.039	<0.010	<0.010	<0.010	0.218	22.6	0.023	0.114	0.180	
SMRF 0156 (B)	**04-0391**	3.33	1.61	0.043	1.59	0.082	0.061	10.2	0.034	0.142	2.37	0.933	1.48	10.6	0.417	0.044	<0.010	<0.010	<0.010	0.218	22.6	0.025	0.111	0.180	
SMRF 0157 (A)	**04-0392**	3.19	1.67	0.042	1.67	0.072	0.061	9.64	0.026	0.048	2.41	0.877	1.59	9.38	0.344	0.030	<0.010	<0.010	<0.010	0.182	23.4	0.014	0.106	0.151	
SMRF 0157 (B)	**04-0392**	3.19	1.64	0.042	1.64	0.073	0.060	9.76	0.025	0.048	2.40	0.884	1.58	9.51	0.345	0.030	<0.010	<0.010	<0.010	0.184	23.5	0.015	0.106	0.148	
SMRF 0161 (A)	**04-0393**	2.98	1.69	0.039	1.69	0.074	0.054	8.82	0.024	0.016	2.46	0.833	1.72	9.48	0.327	0.019	<0.010	<0.010	<0.010	0.153	23.6	0.002	0.094	0.091	
SMRF 0161 (B)	**04-0393**	3.04	1.74	0.038	1.76	0.072	0.054	8.96	0.025	0.017	2.52	0.824	1.75	9.23	0.335	0.019	<0.010	<0.010	<0.010	0.154	23.6	0.002	0.096	0.086	
SMRF 0163 (A)	**04-0394**	3.41	1.60	0.045	1.60	0.068	0.055	9.62	0.026	0.075	2.39	0.929	1.39	9.64	0.353	0.039	<0.010	<0.010	<0.010	0.188	23.1	0.024	0.115	0.192	
SMRF 0163 (B)	**04-0394**	3.44	1.62	0.047	1.62	0.069	0.056	9.76	0.027	0.070	2.35	0.949	1.40	9.88	0.352	0.040	<0.010	<0.010	<0.010	0.189	23.1	0.025	0.116	0.217	
SMRF 0165 (A)	**04-0395**	3.41	1.61	0.044	1.63	0.065	0.055	9.55	0.026	0.074	2.35	0.898	1.37	9.42	0.347	0.038	<0.010	<0.010	<0.010	0.179	23.1	0.024	0.117	0.188	
SMRF 0165 (B)	**04-0395**	3.43	1.62	0.046	1.62	0.064	0.055	9.75	0.026	0.068	2.38	0.902	1.38	9.37	0.355	0.039	<0.010	<0.010	<0.010	0.177	23.1	0.025	0.114	0.186	
oxide wt% - calcined 1100C		Al2O3	B2O3	BaO	CaO	Cr2O3	CuO	Fe2O3	Gd2O3	K2O	Li2O	MgO	MnO	Na2O	NiO	PbO	PdO	RhO2	RuO2	SO4	SiO2	TiO2	ZnO	ZrO2	Totals
SMRF 0156 (A)	**04-0391**	6.33	5.28	0.049	2.30	0.114	0.074	14.4	0.029	0.095	5.07	1.57	1.90	14.2	0.537	0.042	0.000	0.000	0.000	0.654	48.4	0.038	0.141	0.243	101
SMRF 0156 (B)	**04-0391**	6.29	5.18	0.048	2.23	0.120	0.076	14.6	0.039	0.170	5.10	1.55	1.91	14.3	0.530	0.048	0.000	0.000	0.000	0.654	48.4	0.042	0.138	0.243	102
SMRF 0157 (A)	**04-0392**	6.03	5.38	0.047	2.34	0.105	0.076	13.8	0.030	0.058	5.18	1.46	2.05	12.7	0.437	0.032	0.000	0.000	0.000	0.546	50.1	0.023	0.131	0.204	101
SMRF 0157 (B)	**04-0392**	6.03	5.28	0.047	2.30	0.107	0.075	14.0	0.029	0.058	5.16	1.47	2.04	12.8	0.438	0.032	0.000	0.000	0.000	0.552	50.3	0.025	0.131	0.200	101
SMRF 0161 (A)	**04-0393**	5.63	5.44	0.044	2.37	0.108	0.068	12.6	0.028	0.019	5.29	1.38	2.22	12.8	0.415	0.021	0.000	0.000	0.000	0.459	50.5	0.003	0.117	0.123	100
SMRF 0161 (B)	**04-0393**	5.75	5.60	0.043	2.46	0.105	0.068	12.8	0.029	0.020	5.42	1.37	2.26	12.5	0.425	0.021	0.000	0.000	0.000	0.462	50.5	0.003	0.119	0.116	100.0
SMRF 0163 (A)	**04-0394**	6.44	5.15	0.050	2.24	0.099	0.069	13.8	0.030	0.090	5.14	1.54	1.79	13.0	0.448	0.042	0.000	0.000	0.000	0.564	49.4	0.040	0.143	0.259	100
SMRF 0163 (B)	**04-0394**	6.50	5.22	0.053	2.27	0.101	0.070	14.0	0.031	0.084	5.05	1.58	1.81	13.3	0.447	0.043	0.000	0.000	0.000	0.567	49.4	0.042	0.144	0.293	101
SMRF 0165 (A)	**04-0395**	6.44	5.18	0.049	2.28	0.095	0.069	13.7	0.030	0.089	5.05	1.49	1.77	12.7	0.441	0.041	0.000	0.000	0.000	0.537	49.4	0.040	0.145	0.254	100
SMRF 0165 (B)	**04-0395**	6.49	5.22	0.052	2.27	0.093	0.069	13.9	0.030	0.082	5.12	1.50	1.78	12.6	0.451	0.042	0.000	0.000	0.000	0.531	49.4	0.042	0.141	0.251	100

Sample ID	Lab ID	Fe(2+)	Fe(3+)	Fe(total)	Fe(2+)/Fe(3+)	Fe(2+)/Fe(total)
EA		0.046	0.225	0.271	0.20	0.17
Smrf-0156	**04-0391**	0.083	0.341	0.424	0.24	0.20
		0.087	0.347	0.434	0.25	0.20
Smrf-0157	**04-0392**	0.064	0.412	0.476	0.16	0.13
		0.069	0.299	0.368	0.23	0.19
Smrf-0161	**04-0393**	0.022	0.477	0.380	0.05	0.06
		0.023	0.482	0.378	0.05	0.06
Smrf-0163	**04-0394**	0.162	0.432	0.594	0.38	0.27
		0.156	0.437	0.593	0.36	0.26
Smrf-0165	**04-0395**	0.088	0.367	0.455	0.24	0.19
		0.042	0.195	0.237	0.22	0.18

SMRF-0156 WL	**SMRF-0157 WL**	**SMRF-0161 WL**	**SMRF-0163 WL**	**SMRF-0165 WL**
36.7	35.2	32.2	36.0	35.7
156 Norm Li2O	**157 Norm Li2O**	**161 Norm Li2O**	**163 Norm Li2O**	**165 Norm Li2O**
5.01	5.13	5.36	5.06	5.08

SMRF-0156 (Top of glass sample after end of 155% acid stoich feed @ 50% solids)
SMRF-0157 (8kg glass poured sample for 155% acid stoich)
SMRF-0161 (startup glass for SMRF)
SMRF-0163 (8 kg glass poured sample for 185% acid stoich)
SMRF-0165 (1.5 kg glass drain sample after 185% acid stoich)

Frit 418 Li2O value (normalized)
7.91

Note: All WL calculations verified by hand calculations, Frit 418 normalized Li2O value is 7.91 (Lot L2U from DWPF)
WL's calculated w/normalized Li2O valuses in feed and Frit 418

Table B-1. SMRF SB3 Frit 418 Acid Stoichiometry Glass Pour and Glass Drain Analytical Results

Sample ID	Lab ID																								
elemental wt%-calcined 1100C		**Al**	**B**	**Ba**	**Ca**	**Cr**	**Cu**	**Fe**	**Gd**	**K**	**Li**	**Mg**	**Mn**	**Na**	**Ni**	**Pb**	**Pd**	**Rh**	**Ru**	**S**	**Si**	**Ti**	**Zn**	**Zr**	
SMRF 0167 (A)	**04-0641**	3.58	1.55	0.054	0.856	0.090	0.057	9.97	0.029	0.127	2.34	0.927	1.42	9.63	0.425	0.041	<0.010	<0.010	<0.010	0.181	23.5	0.027	0.120	0.268	
SMRF 0167 (B)	**04-0641**	3.36	1.62	0.053	0.819	0.092	0.066	10.1	0.029	0.077	2.36	0.942	1.37	9.55	0.437	0.040	<0.010	<0.010	<0.010	0.191	22.8	0.028	0.129	0.202	
SMRF 0171 (A)	**04-0642**	3.36	1.62	0.052	0.889	0.060	0.056	9.87	0.029	0.066	2.40	0.889	1.33	9.34	0.347	0.040	<0.010	<0.010	<0.010	0.169	24.3	0.027	0.122	0.192	
SMRF 0171 (B)	**04-0642**	3.38	1.58	0.053	0.897	0.062	0.058	9.94	0.029	0.068	2.42	0.913	1.35	9.41	0.358	0.041	<0.010	<0.010	<0.010	0.175	24.3	0.027	0.121	0.200	
oxide wt% - calcined 1100C		Al2O3	B2O3	BaO	CaO	Cr2O3	CuO	Fe2O3	Gd2O3	K2O	Li2O	MgO	MnO	Na2O	NiO	PbO	PdO	RhO2	RuO2	SO4	SiO2	TiO2	ZnO	ZrO2	Totals
SMRF 0167 (A)	**04-0641**	6.77	4.99	0.060	1.20	0.131	0.071	14.3	0.033	0.152	5.03	1.54	1.83	13.0	0.540	0.044	0.000	0.000	0.000	0.543	50.3	0.045	0.149	0.362	101
SMRF 0167 (B)	**04-0641**	6.35	5.22	0.059	1.15	0.134	0.083	14.4	0.033	0.092	5.07	1.56	1.77	12.9	0.555	0.043	0.000	0.000	0.000	0.573	48.8	0.047	0.160	0.273	99.3
SMRF 0171 (A)	**04-0642**	6.35	5.22	0.058	1.24	0.088	0.070	14.1	0.033	0.079	5.16	1.48	1.72	12.6	0.441	0.043	0.000	0.000	0.000	0.507	52.0	0.045	0.151	0.259	102
SMRF 0171 (B)	**04-0642**	6.39	5.09	0.059	1.26	0.091	0.073	14.2	0.033	0.082	5.20	1.52	1.74	12.7	0.455	0.044	0.000	0.000	0.000	0.525	52.0	0.045	0.150	0.270	102

REDOX						
		Fe(2+)	**Fe(3+)**	**Fe(total)**	**Fe(2+)/Fe(3+)**	**Fe(2+)/Fe(total)**
EA		0.084	0.340	0.424	0.25	0.20
SMRF-0167	04-0641	0.016	0.522	0.538	0.03	0.03
		0.016	0.532	0.548	0.03	0.03
SMRF-0171	04-0642	0.083	0.404	0.487	0.21	0.17
		0.077	0.414	0.491	0.19	0.16

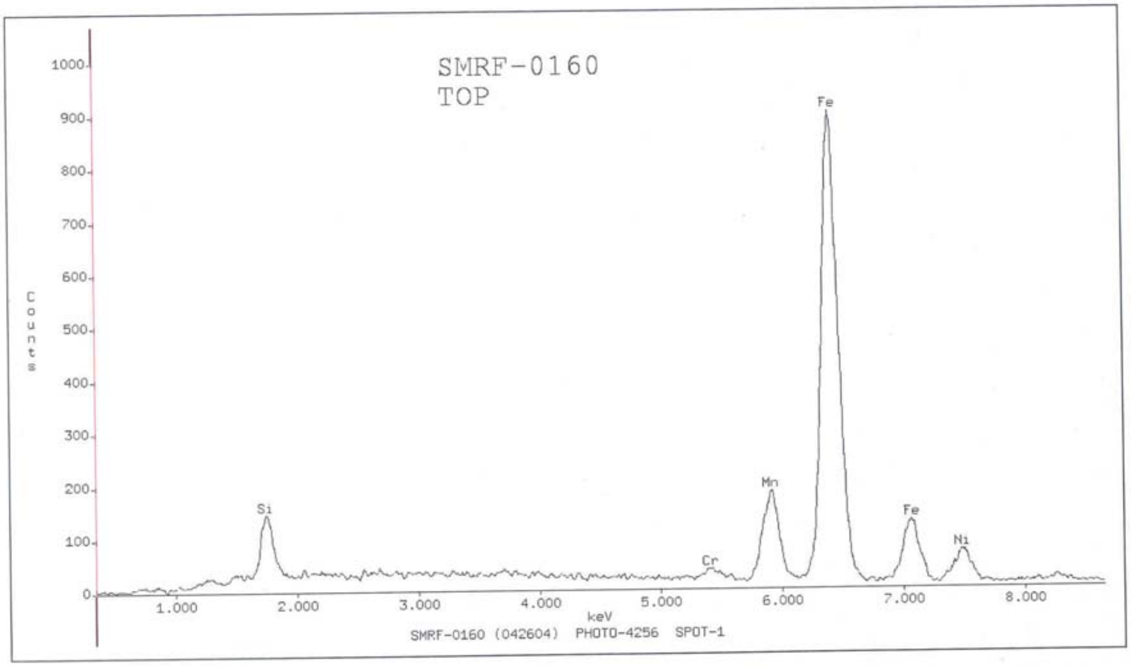
Sample Description

SMRF-0167: glass sample after 8 kg of 0 REDOX glass poured

SMRF-0171: glass sample after 10.4 kg of 0.1 REDOX glass poured

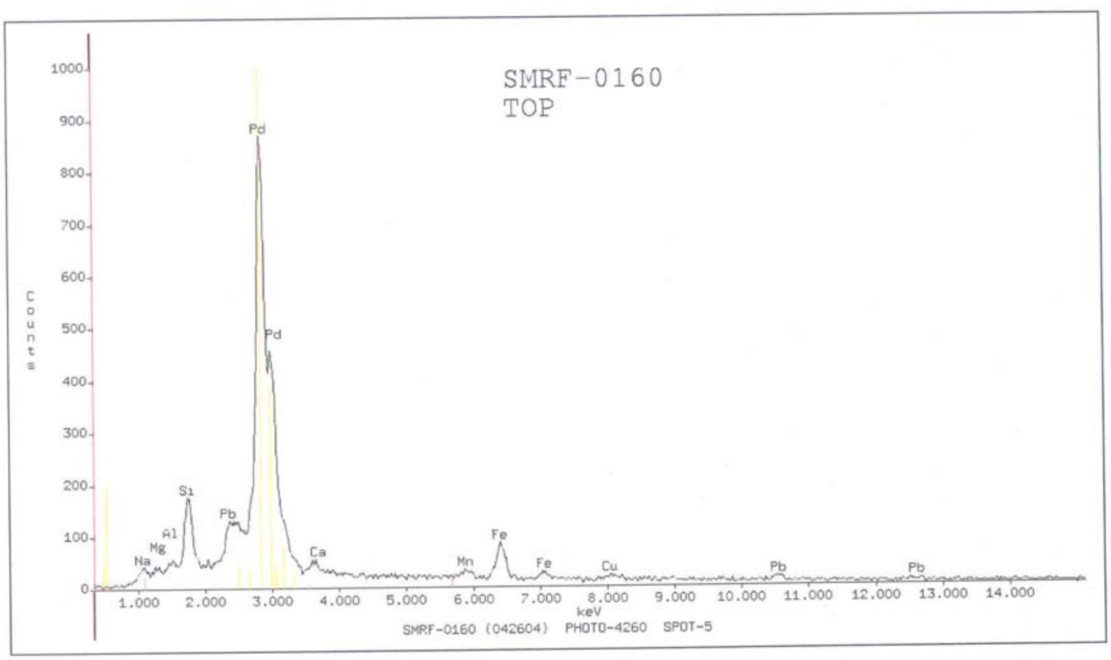
Table B-2. SMRF SB3 Frit 418 REDOX Glass Pour Analytical Results

APPENDIX C – EDS ANALYSES OF SMRF TOP OF GLASS SAMPLE SMRF-0160



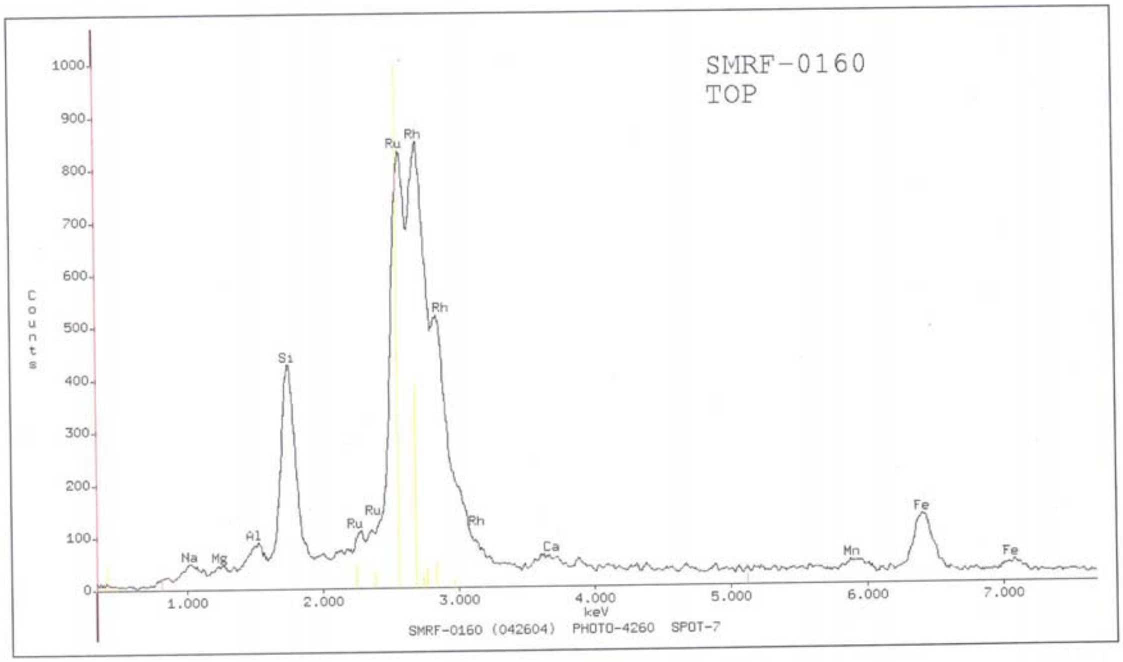
SMRF-0160 (042604) PHOTO-4256 SPOT-1

Figure C-1. EDS of SMRF Top of Glass Sample SMRF-0160 (Spot 1) Indicating Crystalline Phase Composed of Fe, Si, Mn, Cr, and Ni



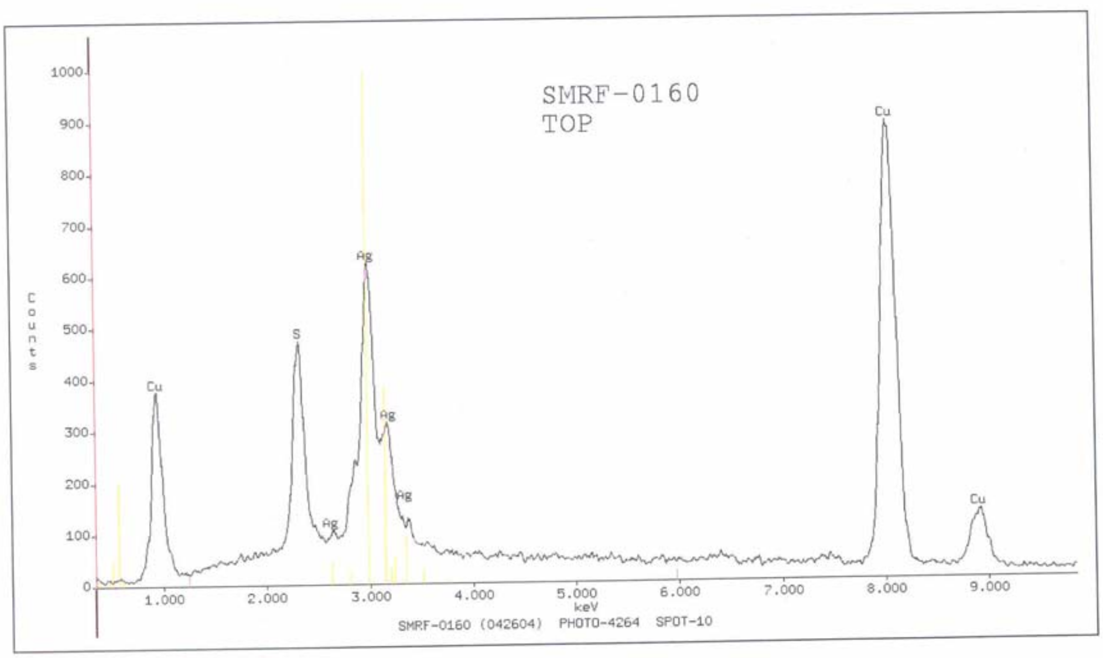
SMRF-0160 (042604) PHOTO-4260 SPOT-5

Figure C-2. EDS of SMRF Top of Glass Sample SMRF-0160 (Spot 5) Indicating Pd Rich Crystalline Phase



SMRF-0160 (042604) PHOTO-4260 SPOT-7

Figure C-3. EDS of SMRF Top of Glass Sample SMRF-0160 (Spot 7) Indicating Ru and Rh Rich Crystalline Phase



SMRF-0160 (042604) PHOTO-4264 SPOT-10

Figure C-4. EDS of SMRF Top of Glass Sample SMRF-0160 (Spot 10) Indicating Crystalline Phase with Ag, Cu, and S