SLUDGE BATCH 4 BASELINE MELT RATE FURNACE AND SLURRY-FED MELT RATE FURNACE TESTS WITH FRITS 418 AND 510 (U)

M. E. Smith T. M. Jones D. H. Miller

September 2007

Process Science and Engineering Section Savannah River National Laboratory Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DEAC09-96SR18500



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WSRC-STI-2007-00450

EXECUTIVE SUMMARY

Several Slurry-Fed Melt Rate Furnace (SMRF) tests with earlier projections of the Sludge Batch 4 (SB4) composition have been performed.^{1,2} The first SB4 SMRF test used Frits 418 and 320, however it was found after the test that the REDuction/OXidation (REDOX) correlation at that time did not have the proper oxidation state for manganese.¹ Because the manganese level in the SB4 sludge was higher than previous sludge batches tested, the impact of the higher manganese oxidation state was greater. The glasses were highly oxidized and very foamy, and therefore the results were inconclusive. After resolving this REDOX issue, Frits 418, 425, and 503 were tested in the SMRF with the updated baseline SB4 projection.²

Based on dry-fed Melt Rate Furnace (MRF) tests and the above mentioned SMRF tests, two previous frit recommendations were made by the Savannah River National Laboratory (SRNL) for processing of SB4 in the Defense Waste Processing Facility (DWPF). The first was Frit 503 based on the June 2006 composition projections.³ The recommendation was changed to Frit 418 as a result of the October 2006 composition projections (after the Tank 40 decant was implemented as part of the preparation plan).[#] However, the start of SB4 processing was delayed due to the control room consolidation outage and the repair of the valve box in the Tank 51 to Tank 40 transfer line. These delays resulted in changes to the projected SB4 composition.⁴

Due to the slight change in composition and based on preliminary dry-fed MRF testing, SRNL believed that Frit 510 would increase throughput in processing SB4 in DWPF. Frit 418, which was used in processing Sludge Batch 3 (SB3), was a viable candidate and available in DWPF. Therefore, it was used during the initial SB4 processing. Due to the potential for higher melt rates with Frit 510, SMRF tests with the latest SB4 composition (1298 canisters) and Frits 510 and 418 were performed at a targeted waste loading (WL) of 35%. The "1298 canisters" describes the number of equivalent canisters that would be produced from the beginning of the current contract period before SB3 is blended with SB4.

The melt rate for the SMRF SB4/Frit 510 test was 14.6 grams/minute. Due to cold cap mounding problems with the SMRF SB4/Frit 418 feed at 50 weight % solids that prevented a melt rate determination, this feed was diluted to 45 weight % solids. The melt rate for this diluted feed was 8.9 grams/minute. A correction factor of 1.2 for estimating the melt rate at 50 weight % solids from 45 weight % solids test results (based on previous SMRF testing⁵) was then used to estimate a melt rate of 10.7 grams/minute for SB4/Frit 418 at 50 weight % solids. Therefore, the use of Frit 510 versus Frit 418 with SB4 resulted in a higher melt rate (14.6 versus an estimated 10.7 grams/minute). For reference, a previous SMRF test with SB3/Frit 418 feed at 35% waste loading and 50 weight % solids resulted in a melt rate of 14.1 grams/minute.⁵ Therefore, depending on the actual feed rheology, the use of Frit 510 with SB4 could result in similar melt rates as experienced with SB3/Frit 418 feed in the DWPF.

[#]Frit 418 was identified as a primary candidate for the October 2006 SB4 projections. In addition, the higher B_2O_3 based frits (Frits 505, 510, and 511) were also identified as candidate frits. This information was presented to the Liquid Waste Organization and the DWPF via a presentation at the DWPF Plan of the Month meeting (see WSRC-NB-2006-00017, pages 106-118 for more detail).

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LIST OF ACRONYMS/ABBREVIATIONS

ACTL	Aiken County Technology Laboratory
ARP	Actinide Removal Process
DWPF	Defense Waste Processing Facility
ID	Identification
LMR	Linear Melt Rate
MRF	Melt Rate Furnace
MST	Monosodium Titanate
REDOX	REDuction/OXidation
SB3	Sludge Batch 3
SB4	Sludge Batch 4
SME	Slurry Mix Evaporator
SMRF	Slurry-Fed Melt Rate Furnace
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
WL	Waste Loading
WSRC	Washington Savannah River Company

WSRC-STI-2007-00450

1.0 INTRODUCTION

Several Slurry-Fed Melt Rate Furnace (SMRF) tests with earlier projections of the Sludge Batch 4 (SB4) composition have been performed.^{1,2} The first SB4 SMRF test used Frits 418 and 320, however it was found that the REDuction/OXidation (REDOX) correlation at that time did not have the proper oxidation state for manganese.¹ Because the manganese level in the SB4 sludge was higher than previous sludge batches tested, the impact of the higher manganese oxidation state was greater. The glasses were highly oxidized and very foamy and therefore the results were inconclusive. After resolving this REDOX issue, Frits 418, 425, and 503 were tested in the SMRF with an updated baseline projection of the SB4 composition.²

Based on dry-fed Melt Rate Furnace (MRF) tests and the above mentioned SMRF tests, two previous frit recommendations were made by the Savannah River National Laboratory (SRNL) for processing of SB4 in the Defense Waste Processing Facility (DWPF). The first was Frit 503 based on the June 2006 composition projections.³ The recommendation was changed to Frit 418 from the October 2006 composition projections (after the Tank 40 decant was implemented as part of the preparation plan). However, the start of SB4 processing was delayed due to the control room consolidation outage and the repair of the valve box in the Tank 51 to Tank 40 transfer line. These delays resulted in changes to the projected SB4 composition.⁴

Due to the slight change in composition and based on preliminary dry-fed MRF testing, SRNL believed that Frit 510 would increase throughput in processing SB4 in DWPF. Frit 418, which was used to process SB3, was a viable candidate and available in DWPF. Therefore it was used during the initial SB4 processing. Due to the potential for higher melt rates with Frit 510, SMRF tests with the latest SB4 composition (1298 canisters) and Frits 510 and 418 were performed at a targeted waste loading (WL) of 35%. The "1298 canisters" describes the number of equivalent canisters that would be produced from the beginning of the current contract period before SB3 is blended with SB4.

The work was performed per Task Technical and Quality Assurance Plan WSRC-RP-2006-00713.⁶ Tests were performed with non-radioactive, simulated SB4 material. Due to the small-scale of the test equipment and the design of the equipment, as well as the use of non-radioactive simulant feed, the behavior of the actual radioactive SB4 feed in the DWPF melter cannot be fully proven. However, previous SRNL simulant melt rate results have shown trends that are consistent with those of actual melt rates obtained at the DWPF.

2.0 EXPERIMENTAL

2.1 22-L SRAT/SME Feed Preparation Details for SMRF Runs

The SB4 sludge composition used in these melt rate tests was designated as "1298 canisters". As previously stated, "1298 canisters" describes the number of equivalent canisters that would be produced from the beginning of the current contract period before SB3 is blended with SB4. This targeted sludge did not include auxiliary waste streams such as the Actinide Removal Process (ARP) stream, which contains monosodium titanate (MST), entrained sludge, and various soluble sodium compounds as a result of filter cleaning and stream adjustment for transfer.

The Sludge Receipt and Adjustment Tank/Slurry Mix Evaporator (SRAT/SME) products were made in the Aiken County Technology Laboratory (ACTL) 22-L vessels using the above described sludge composition. The feed preparation process strategy used 135% acid stoichiometry and targets of 35% waste loading (WL) and 0.2 REDOX defined as $Fe^{2+}/\Sigma Fe$.

Table 2-1 gives the analysis of SB4 simulant used for these tests as well as the composition of the typical SB3 simulant. The main differences between the SB4 and SB3 sludge compositions are the higher levels of aluminum and the lower level of iron in the SB4 sludge. The higher aluminum and lower iron concentrations would tend to lower melt rate. The SB4 sludge identification (ID) used for these tests was 1298-1.

		TYPICAL SB3 ⁷
ELEMENT	SB4 SIMULANT	SIMULANT
Al	16.0	9.57
Ba	0.05	0.14
Ca	2.47	2.37
Cr	0.11	0.15
Fe	21.80	28.35
K	0.17	0.12
Mg	1.98	2.15
Mn	5.24	4.07
Na	12.7	14.05
Ni	1.43	1.06
Pb	0	0.01
Si	1.44	1.04
Zn	0.04	0.32
Zr	0.06	0.49

Table 2-1. SB4 Sludge Simulant and Typical SB3 Simulant Sludge Compositions (Elemental Weight % Calcined Solids)

Two SRAT/SME runs were performed to make each SMRF feed listed in Table 2-2. The SB4 SRAT/SME run ID numbers and the run plan numbers for each feed are given in Table 2-2. Compositions of the SME products for the SMRF runs are given in Appendix A. Prior to performing the SMRF tests, both SMRF feeds were pumped with the SMRF feed delivery system to ensure that they could be fed at the required 100 grams/20 second feed cycle. Both feeds met this requirement.

SRAT/SME RUN ID	FEED DESCRIPTION	*SRNL RUN PLAN #
SB4-73 and SB4-74	SB4/Frit 418 (35% waste loading)	SRNL-PSE-2007-00142
SB4-75 and SB4-76	SB4/Frit 510 (35% waste loading)	SRNL-PSE-2007-00147

Table 2-2.	Descriptions of SRAT/SME Runs for SB4 SMRF Tests	
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* Additional feed preparation details for these runs are given in SRNL-PSE-2007-00197

2.2 Test Frit Compositions

Frits 418, 503, 509, 510, and 511 were tested in the MRF with this same SB4 composition at 35% waste loading. Based on the tests results and other considerations, Frits 418 and 510 were selected for testing in the SMRF. Frit 418 was used to process SB3 at DWPF as well as for initial processing of SB4 and was considered the baseline frit for these tests. Table 2-3 gives the compositions of these frits.

FRIT	B_2O_3	Li ₂ O	Na ₂ O	SiO ₂
418	8	8	8	76
510	14	8	8	70
503	14	8	4	74
509	14	8	7	71
511	14	8	9	69

 Table 2-3. Frits Compositions Used in Latest SB4 MRF and SMRF Tests (SMRF Frits in "Bold")

2.3 SMRF Testing Details

Details of the SMRF configuration and operation are documented elsewhere.⁸ The operational parameters for the SMRF tests were consistent with previous testing. More specifically, the melt pool and vapor space setpoints were 1125°C and 750°C, respectively. The duration for each feed cycle after the vapor space had reached the feed initiation setpoint of 750°C was 20 seconds. The measured current for the melt pool and vapor space heaters were both about 20 amps for the two tests.

2.4 MRF Testing Details

The dry-fed MRF has a cylindrical inner chamber that is approximately 0.5 cubic feet in size, with heating coils winding around the chamber walls. The diameter of the chamber is \sim 7", and an insulating sleeve and 1200 mL stainless steel beaker (6" deep) were inserted from the top for each test. The tests were conducted with the 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 a beaker containing sufficient dried, sieved material to produce 525 grams of glass was inserted. After 50 minutes, the beaker was removed from the furnace and allowed to cool to room temperature. This residence time in the furnace was established as a standard test time for melt rate comparison for this dry-fed furnace.⁹ For these MRF tests, relative melt rate was determined by measuring the height of the glass layer in the bottom of each sectioned beaker at 0.25" intervals. A new technique using digital radiography to view the glass layer height was not used for these MRF tests due to equipment problems. The average height of the glass layer and residence time in the furnace was used to yield a relative linear melt rate (LMR) number (inches/hour). General observations of the beakers can also be used to describe differences between runs.

3.0 RESULTS AND DISCUSSION

As previously discussed, several SB4 MRF runs were performed with a set of candidate frits. Based on the results of these runs, two SMRF runs were performed with SB4 simulated sludge with Frits 418 and 510 at 35% WL. The SMRF runs were recorded in notebook WSRC-NB-2004-00123 and performed per Run Plan SRNL-PSE-2007-00114.

3.1 SB4 MRF Runs

Table 3-1 summarizes the results of the SB4 MRF runs performed in 2007. All melt rates are given in LMR. For comparison, the melt rate previously measured for SB3/Frit 418 (35% WL) was 0.51 inches/hr.¹⁰ However, a shift in the melt rate of the standard used does not allow comparison with these results and the previous SB3/Frit 418 MRF result. The frits that indicated a possible positive impact on SB4 melt rate were Frits 510 and 511.

FRIT ID	WL	*LMR (IN/HR)
418	35	0.69
503	35	0.65
509	35	0.69
510	35	0.71
511	35	0.77
510	35	0.71
510	38	0.66
510	41	0.53

Table 3-1. SB4 MRF Test Summary and Results

* Based on historical MRF data, the estimated precision in LMR is about +/- 0.02 inches/hour

SRNL made the test frits for the MRF runs because the amount of test frit needed for each MRF run was small (less than 100 grams). The frits for the SMRF tests (418 and 510), on the other hand, were taken from existing lots at DWPF. Based on the promising SB4/Frit 511 MRF result, it would appear that Frit 511 would be the logical frit to be recommended for SB4. However, the nominal stage assessment for Frit 512, when coupled with the baseline SB4 composition, indicated a projected operating window of only 25-38% WL (being low viscosity limited). This is a 3 point decrease in the upper percent WL limit for Frit 512 relative to the projected operating window from Frit 510's assessment. Frit 511 is very similar to Frit 512 (Frit 512 contains only 1% more Na₂O and 1% less SiO₂). Due to its relatively poor projected operational window, no SB4 "1298 canister" MRF tests were performed with Frit 512. Because of this compositional similarity between Frits 511 and 512, the use of Frit 511 presents concerns of negative impacts on property predictions due to compositional variation at the upper end of DWPF's projected WL operating window. Therefore, Frit 511 was not considered for SB4 SMRF testing. Additional details concerning this decision are documented elsewhere.⁴

3.2 SB4/Frit 510 SMRF Run

The SMRF was charged with 6.1 kg of glass (drained from the SMRF after the SB4/Frit 503 (35% WL) 5/26/06 test) and heated up to operating temperatures on 7/9/07. Feeding of the SB4/Frit 510 feed was started at 0710 hours on 7/10/07. The feed pump speed was 250 rpm to deliver about 100 grams of feed per each 20 second feed cycle. Feeding and pouring were continued until 1330 hours without any major feed problems or signs of overfeeding of the melter. Some cold cap mounding was observed and the cold

cap mound under the feed tube was manually knocked down at 1330. At 1400 the pour rate was higher than what would be expected with the feed rate and therefore feeding was stopped to allow the cold cap to burn off. Feeding was restarted at 1537. At 1720 feeding was stopped because the cold cap was very dark and a mound was up near the tip of the feed tube. The average melt rate from 1000 to 1330 hours (steady state portion of test) was 14.6 grams/minute. A total of 7262 grams of glass was poured during this test.

3.3 SB4/Frit 418 SMRF Run

The SB4/Frit 418 SMRF test was performed on 7/11/07 and 7/12/07. Feeding was started at 0710 hours on 7/11/07. A feed pump speed of 250 rpm was used to deliver about 100 grams of feed per each 20 second feed cycle. Feeding and pouring were continued until 1150 hours when an inspection of the cold cap showed a complete bridging of the cold cap and the cold cap was very thick. Therefore feeding was stopped at 1155 hours but the pouring was continued. At 1300 hours the cold cap had burned off and the glass height was at the overflow level. Feeding was restarted at 1300 hours but stopped at 1400 hours due to mounding of the cold cap (see Figure 3-1). The left side of the melt pool was uncovered, but the mounding indicated that the feed was too thick. In addition, it was noted that the feed rate of 40 grams/minute was too high relative to the resultant pour rate of only 6-8 grams/minute (40 grams/minute should result in a pour rate of about 16.5 grams/minute). A total of 3800 grams of glass was poured during this part of the test. Due to excessive mounding and lack of cold cap coverage, an accurate melt rate could not be determined for SB4/Frit 418 at 50 weight % solids. A decision was made to dilute the feed from 49.5 to 45 weight % solids to improve the rheology of the feed to allow for better cold cap coverage. Therefore 101 grams of water was added per 1 kg of feed. This decision was made even though the SB4/Frit 510 feed was only tested at 50 weight % solids. Previous SMRF tests with SB3/Frit 418 had determined a correction factor of 1.2 to estimate a melt rate at 50 weight % solids from tests using 45 weight % solids feed.⁵ Part of the rationale for the dilution decision was that there was not enough feed left to allow for any more mounding problems.



Figure 3-1. SMRF Cold Cap at 1400 on 7/11/07 during SB4/Frit 418 Test

Feeding was restarted at 1438 hours with the diluted feed. However, at 1600 hours a power outage stopped the test as the pour and drain induction heaters could not be restarted (possibly due to power supply tuning problems). It was decided to restart the test the next day. For information, 810 grams of glass was poured after the dilution of the feed so that a total of 4610 grams was poured on 7/11/07.

The test was resumed on 7/12/07 at 0710 with the diluted feed. At 1230 hours, the entire melt surface was covered with a cold cap, but the cold cap was orange with no major dark areas, thus indicating a thin cold cap. Just 15 minutes later the cold cap was observed to be turning slightly darker than that observed at 1230 hours. At 1255 hours, the test had to be stopped because there was not enough feed left to continue the test. The average melt rate from 0800 to 1230 hours (steady state portion of test) was 8.9 grams/minute. With the previously mentioned correction factor of 1.2, an estimated melt rate of about 10.7 grams/minute would have been achieved at 50 weight % solids if the feed had flowed better on the melt pool/cold cap. A total of 3332 grams of glass was poured. At 1330 hours the drain induction heater was started and draining began at 1415 hours. Draining was completed at 1538 hours and about 5000 grams of glass was drained.

3.4 Discussion of Results

Glass pour samples were taken at the end of each SMRF test. In addition, a glass drain sample was taken at the end of the SB4/Frit 418 test drain. The compositions of these three glass samples are given in Attachment A (Table A-4). Table 3-2 summarizes the REDOX values of the three glass samples. The Fe²⁺/ Σ Fe target was 0.20. SME product analyses projected a value >0.15.¹¹ The actual measured values of the glass samples were slightly higher than the SME product projections, but close to the target of 0.20.

SAMPLE ID	DESCRIPTION	GRAMS POURED/ DRAINED	*REDOX (F e^{2+}/Σ Fe)
SMRF-0230	End of SB4/Frit 510 pouring	7262	0.200
SMRF-0231	End of SB4/Frit 418 pouring	4610	0.216
SMRF-0232	End of SB4/Frit 418 draining	5000	0.225

 Table 3-2. REDOX Values of the SB4 SMRF Test Glass Samples

*Average of two measurements from one sample

Another set of important parameters that was reviewed was the power consumption (BTU/min) of the plenum and melt pool heaters during these tests. Table 3-3 gives these values for the two SB4 SMRF tests as well as the power consumed in a previous SB3/Frit 418 (45% solids) SMRF test (actual power consumptions for the SB3/Frit 418 tests documented in later SB4 melt rate report²). The only difference is the lower SB4/Frit 418 plenum power consumption for the 50 weight % solids run. This indicates that the cold cap was not fully covered, and therefore, less plenum power was required. When the feed was diluted to 45 weight % solids, the cold cap flowed better and its coverage was greater, resulting in higher plenum power consumption as the contribution of heat from the melt pool to the plenum was less.

Table 3-3.	Power	(BTU/Min)	Consumptions	for the	SB4	SMRF 1	Runs
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	SB4/FRIT 418	SB4/FRIT 510	SB3/FRIT 418
Plenum Power	*85.2 / **94.2	*96.3	**97.6
Melt Pool Power	*45.8 / **44.4	*46.2	**45.4

* 50 wt% solids run, ** 45 wt% solids run

A summary of the SB4 SMRF melt rate results is given in Table 3-4. The melt rates for the SB4/Frit 510 feed at 50 weight % solids and the estimate for 50 weight % solids SB4/Frit 418 feed were 14.6 grams/minute and 10.7 grams/minute, respectively, at 35% waste loading. For reference, a previous SMRF test with SB3/Frit 418 feed at 35% waste loading and 50 weight % solids resulted in a melt rate of 14.1 grams/minute.⁵ Therefore, depending on the actual feed rheology, the use of Frit 510 with SB4 could result in similar melt rates as experienced with SB3/Frit 418 feed.

	SB4/FRIT 510	SB4/FRIT 418
	(50 WT%)	(45/50 WT%)
Melt Rate (grams/minute)	14.6	8.9 / *10.7

Table 3-4. SB4 SMRF Tests Melt Rate (Grams/Minute) Results at 35% WL

* This is the estimated melt rate for SB4/Frit 418 at 50 weight % solids using a correction factor of 1.2 with the actual SB4/Frit 418 melt rate at 45 weight % solids

4.0 CONCLUSIONS

The following conclusions can be made with regard to the latest baseline SB4 composition (designated "1298 canisters") MRF and SMRF melt rate tests with Frits 418 and 510:

- MRF tests indicated that Frits 509, 510, and 511 resulted in higher melt rates with SB4 feed than Frit 418. These frits have higher B₂O₃ and similar Na₂O concentrations as compared with Frit 418.
- Based on the promising SB4/Frit 511 MRF result, it would appear that Frit 511 would be the logical frit to be recommended for SB4. However, the nominal stage assessment for Frit 512, when coupled with the baseline SB4 composition, indicated a projected operating window of only 25-38% WL (being low viscosity limited). This is a 3 point decrease in the upper percent WL limit for Frit 512 relative to the projected operating window from Frit 510's assessment. Frit 511 is very similar to Frit 512 (Frit 512 contains only 1% more Na₂O and 1% less SiO₂). Due to its relatively poor projected operational window, no SB4 "1298 canister" MRF tests were performed with Frit 512. Because of this compositional similarity between Frits 511 and 512, the use of Frit 511 presents concerns of negative impacts on property predictions due to compositional variation at the upper end of DWPF's projected WL operating window. Therefore, Frit 511 was not considered for SB4 SMRF testing. Additional details concerning this decision are documented elsewhere.⁴
- The melt rate for the SMRF SB4/Frit 510 (35% waste loading and 50 weight % solids) test was 14.6 grams/minute. Due to cold cap mounding problems with the SMRF SB4/Frit 418 feed at 50 weight % solids that prevented a melt rate determination, this feed was diluted to 45 weight % solids. The melt rate for this diluted feed was 8.9 grams/minute. A correction factor of 1.2 was applied to allow an estimation of the melt rate at 50 weight % solids based on the 45 weight % SMRF feed tests. Therefore, the estimated melt rate for SB4/Frit 418 (35% waste loading and 50 weight % solids) is 10.7 grams/minute.⁵
- The use of Frit 510 in place of Frit 418 with SB4 resulted in a higher SMRF melt rate.
- For reference, a previous SMRF test with SB3/Frit 418 feed at 35% waste loading and 50 weight % solids resulted in a melt rate of 14.1 grams/minute.⁵ Therefore, depending on the actual sludge rheology, the use of Frit 510 with SB4 could result in similar melt rates as experienced with SB3/Frit 418 feed in the DWPF.

5.0 RECOMMENDATIONS/PATH FORWARD

• Based on the latest results of the SB4 MRF and SMRF testing, SRNL supports its previous recommendation to use Frit 510 in processing SB4 to increase waste throughput.

6.0 REFERENCES

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ATTACHMENT A

COMPOSITION ANALYSES OF THE SME PRODUCTS AND GLASS SAMPLES FOR THE SMRF SB4 TESTS

<u>Zn</u>

<u>Zr</u>

Table A-1. Composition of SME Product SB4-73 for SB4/Frit 418 SMRF Tests

Process Science Analytical Laboratory Customer: Mike Stone Date: 7/12/07 Sample ID: 07-SB4-1381 SB4-73 Lab ID: 07-2322 Sample Preparation: Li2BO4/LiNO3 and Na2O2/NaOH fusions

Sample ID Lab ID elemental wt%-calcined <u>AI</u> B Ba <u>Ca</u> Ce <u>Cr</u> Fe K Li Mg Mn Na Ni <u>S</u> Si <u>Ti</u> 07-SB4-1381 (A) 07-2322 5.54 1.32 0.023 1.00 0.068 0.043 7.65 0.104 2.25 0.57 1.78 8.96 0.398 0.1 23.7 0.044 0.023 0.106 07-SB4-1381 (B) 07-2322 0.024 0.070 0.042 7.49 0.112 2.25 0.58 1.76 9.10 0.394 0.1 23.9 0.044 0.019 0.103 5.52 1.30 1.11 oxide wt% - calcined AI2O3 B2O3 BaO CaO CeO2 Cr2O3 Fe2O3 K2O Li20 MgO MnO2 Na2O NiO SO4 SiO2 TiO2 ZnO ZrO2 Totals 07-SB4-1381 (A) 07-2322 10.5 4.25 0.026 1.40 0.084 0.063 10.9 0.125 4.84 0.95 2.81 12.1 0.51 0.30 50.7 0.073 0.029 0.143 99.8 07-SB4-1381 (B) 07-2322 10.4 4.19 0.027 1.55 0.086 0.061 10.7 0.134 4.84 0.96 2.78 12.3 0.50 0.30 51.1 0.073 0.024 0.139 100.3 anions (mg/Kg) F NO2 NO3 HCO2 SO4 PO4 CI 07-SB4-1381 (A) 07-2322 539 171 <100 <100 22800 52100 <100 07-SB4-1381 (B) 07-2322 22700 52000 538 <100 <100 <100 170

Weight % Solids Calculations

	Empty	Crucible Wt -C	rucible Wt	+		l	nsoluble	Cruc Wt+	Wt %
Sample	Crucible wt	Wet Sample	Dry wt	Tot Solids	Wet Wt	Dry Wt	Solids	Calcined	Calcined
07-SB4-1381 (A) 07-2322	45.4854	50.9935	48.2580	50.3%	5.5081	2.773	40.3%	47.7814	41.7%
07-SB4-1381 (B) 07-2322	44.3198	49.8290	47.0943	50.4%	5.5092	2.775	40.3%	46.6190	41.7%

Table A-2. Composition of SME Product SB4-74 for SB4/Frit 418 SMRF Tests

Process Science Analytical Laboratory Customer: Mike Stone Date: 7/12/07 Sample ID: 07-SB4-1383 SB4-74 Lab ID: 07-2324 Sample Preparation: Li2BO4/LiNO3 and Na2O2/NaOH fusions

Sample ID Lab ID <u>elemental wt%-calcined</u> 07-SB4-1383 (A) 07-2324 07-SB4-1383 (B) 07-2324	<u>B</u> 1.30 1.34	<u>Ba</u> 0.023 0.023	<u>Ca</u> 1.19 1.04	<u>Cd</u> <0.010 <0.010		<u>Cr</u> 0.041 0.042	<u>Fe</u> 7.68 7.81	 	<u>Mg</u> 0.540 0.546			<u>Ni</u> 0.37 0.39	<u>S</u> 0.1 0.104		<u>Ti</u> 0.045 0.046		
<u>oxide wt% - calcined</u> 07-SB4-1383 (A) 07-2324 07-SB4-1383 (B) 07-2324	<u>B2O3</u> 4.19 4.31	<u>BaO</u> 0.026 0.026	<u>CaO</u> 1.67 1.46	0.00	<u>CeO2</u> 0.084 0.085	<u>Cr2O3</u> 0.060 0.061	<u>Fe2O3</u> 11.0 11.2	 4.88	<u>MgO</u> 0.896 0.906	2.80	12.2	<u>NiO</u> 0.47 0.50	0.30	50.7	0.075	0.021	<u>ZrO2</u> <u>Totals</u> 0.143 100.5 0.143 99.6
<u>anions (mq/Kg)</u> 07-SB4-1383 (A) 07-2324 07-SB4-1383 (B) 07-2324	<u>NO2</u> <100 <100	<u>NO3</u> 22000 22200	<u>HCO2</u> 50800 50800	<u>SO4</u> 532 568	<u>PO4</u> <100 <100	<mark>CI</mark> 137 142											

Weight % Solids Calculations

	Empty	Crucible Wt	Crucible W	/t +		Ir	nsoluble	Cruc Wt+	Wt %
Sample	Crucible wt	Wet Sample	Dry wt	Tot Solids	Wet Wt	Dry Wt	Solids	Calcined	Calcined
07-SB4-1383 (A)	07-2324 44.9133	50.6040	47.7197	49.3%	5.6907	2.806	39.6%	47.2323	40.8%
07-SB4-1383 (B)	07-2324 43.7512	49.2119	46.4420	49.3%	5.4607	2.691	39.6%	45.9818	40.8%

Table A-3. Composition of SME Products SB4-75 and SB4-76 for SB4/Frit 510 SMRF Tests

Process Science Ar Customer: Mike Sto Date: 9/04/07 Sample ID: 07-SB4 Lab ID: 07-3008, 07 Sample Preparation	one -1402, 07-S 7-3009	B4-1404	I Na2O2/Na		07-SB4-14 s	902 (SB4- ⁻	75)		07-SB4-1	404 (SB	4-76)									
Sample ID <u>elemental wt%-calc</u> 07-SB4-1402 (A) 07-SB4-1402 (B) 07-SB4-1404 (A) 07-SB4-1404 (B)	Lab ID <u>ined</u> 07-3008 07-3008 07-3009 07-3009	<u>Al</u> 5.25 5.26 5.34 5.34	<u>B</u> 2.59 2.62 2.62 2.60	<u>Ba</u> 0.045 0.046 0.045 0.045	<u>Ca</u> 0.854 0.844 0.838 0.830	<u>Cd</u> <0.010 <0.010 <0.010 <0.010	0.039 0.039	<u>Fe</u> 7.63 7.60 7.37 7.46	<u>K</u> 0.181 0.180 0.195 0.207	<u>Li</u> 2.16 2.17 2.17 2.16	<u>Mq</u> 0.566 0.565 0.576 0.596	<u>Mn</u> 1.74 1.76 1.78 1.76	<u>Na</u> 9.19 9.00 9.22 9.29	<u>Ni</u> 0.40 0.41 0.40 0.41	<u>S</u> 0.11 0.10 0.10 0.10	<u>Si</u> 22.2 22.4 22.3 22.0	<u>Ti</u> 0.115 0.115 0.139 0.141	<u>Zn</u> 0.032 0.024 0.024 0.025	<u>Zr</u> 0.068 0.067 0.065 0.067	
<u>oxide wt% - calcine</u> 07-SB4-1402 (A) 07-SB4-1402 (B) 07-SB4-1404 (A) 07-SB4-1404 (B)	<u>d</u> 07-3008 07-3008 07-3009 07-3009	<u>AI2O3</u> 9.92 9.94 10.1 10.1	<u>B2O3</u> 8.34 8.44 8.44 8.37	<u>BaO</u> 0.050 0.052 0.050 0.050	<u>CaO</u> 1.20 1.18 1.17 1.16	<u>CdO</u> 0.00 0.00 0.00 0.00	<u>Cr2O3</u> 0.057 0.057 0.057 0.064	Fe2O3 10.9 10.9 10.5 10.7	<u>K2O</u> 0.217 0.216 0.234 0.248	<u>Li20</u> 4.64 4.67 4.67 4.64	<u>MgO</u> 0.940 0.938 0.956 0.989	<u>MnO2</u> 2.75 2.78 2.81 2.78	<u>Na2O</u> 12.4 12.2 12.4 12.5	<u>NiO</u> 0.51 0.52 0.51 0.52	<u>SO4</u> 0.32 0.31 0.31 0.31	<u>SiO2</u> 47.5 47.9 47.7 47.1	<u>TiO2</u> 0.192 0.192 0.232 0.235	<u>ZnO</u> 0.040 0.030 0.030 0.031	<u>ZrO2</u> 0.092 0.090 0.088 0.090	<u>Totals</u> 100 100 100 100
<u>anions (mg/L)</u> 07-SB4-1402 (A) 07-SB4-1402 (B) 07-SB4-1404 (A) 07-SB4-1404 (B)	07-3008 07-3008 07-3009 07-3009	<u>F</u> <100 <100 <100 <100	<u>NO2</u> <100 <100 <100 <100 Weight %	<u>NO3</u> 20800 20200 18300 18500 Solids Cal	HCO2 54400 53700 49600 48500	<u>SO4</u> 1060 1050 1180 1130	<u>P04</u> <100 <100 <100 <100	<u>CI</u> 207 193 165 160	<u>C2O4</u> <100 <100 <100 <100											

				weight %	Solids Ca	iculations					
			Empty	Crucible Wt	Crucible W	/t +		Ir	nsoluble	Wt %	
_	Sample	С	rucible wt	Wet Sample	Dry wt	Tot Solids	Wet Wt	Dry Wt	Solids	Calcined	Calcined
_	07-SB4-1402 (A)	07-3008	43.4945	49.0971	46.357	51.1%	5.6026	2.863	40.6%	45.8693	42.4%
	07-SB4-1402 (B)	07-3008	42.3819	47.9026	45.2020	51.1%	5.5207	2.820	40.5%	44.7181	42.3%
	07-SB4-1404 (A)	07-3009	42.9540	48.6131	45.7434	49.3%	5.6591	2.789	39.0%	45.2718	41.0%
	07-SB4-1404 (B)	07-3009	44.0719	49.6030	46.7997	49.3%	5.5311	2.728	39.2%	46.3450	41.1%

Table A-4. Analyses of SB4/Frit 418 and SB/Frit 510 SMRF Test Glass Pour Samples

Sample ID	Lab ID																									
elemental wt%		AL	B	Ba	Ca	Cr	Cu	Fe	Gd	K	Li	Mg	Mn	Na	Ni	Р	Pb	Pd	Rh	Ru	S	Si	Ti	Zn	Zr	
SMRF-0230 (A)	07-2640	5.30	2.64	0.051	0.729	0.049	0.021	7.34	<0.010	0.171	2.29	0.584	1.66	9.0	0.414	<0.100	0.014	< 0.010	<0.010	0.024	0.147	22.7	0.089	0.039	0.073	
SMRF-0230 (B)	07-2640	5.31	2.60	0.052	0.727	0.050	0.022	7.13	<0.010	0.158	2.31	0.603	1.61	9.0	0.451	<0.100	0.015	<0.010	<0.010	0.022	0.150	22.7	0.093	0.041	0.073	
SMRF-0231 (A)	07-2641	5.49	1.79	0.032	0.807	0.042	0.022	7.60	<0.010	0.122	2.22	0.609	1.71	9.2	0.404	<0.100	<0.010	<0.010	<0.010	0.020	0.134	23.3	0.074	0.024	0.096	
SMRF-0231 (B)	07-2641	5.58	1.78	0.033	0.809	0.043	0.020	7.77	<0.010	0.116	2.24	0.627	1.70	9.1	0.431	<0.100	<0.010	<0.010	<0.010	0.022	0.137	23.6	0.076	0.025	0.099	
SMRF-0232 (A)	07-2642	5.52	1.74	0.033	0.813	0.055	0.024	7.81	<0.010	0.115	2.23	0.635	1.77	9.1	0.452	<0.100	<0.010	<0.010	<0.010	0.020	0.138	23.4	0.070	0.026	0.103	1
SMRF-0232 (B)	07-2642	5.57	1.73	0.033	0.825	0.053	0.026	7.70	<0.010	0.127	2.25	0.618	1.74	9.2	0.410	<0.100	<0.010	<0.010	<0.010	0.021	0.139	23.6	0.068	0.025	0.101	1
oxide wt%		<u>Al2O3</u>	<u>B2O3</u>	<u>BaO</u>	<u>CaO</u>	<u>Cr2O3</u>	<u>CuO</u>	Fe2O3	<u>Gd2O3</u>	<u>K20</u>	<u>Li20</u>	MgO	MnO2	<u>Na2O</u>	<u>NiO</u>	P205	PbO	PdO	RhO2	<u>RuO2</u>	<u>SO4</u>	<u>SiO2</u>	<u>TiO2</u>	ZnO	<u>ZrO2</u>	Totals
SMRF-0230 (A)	07-2640	10.0	8.50	0.057	1.02	0.072	0.026	10.5	0.0	0.205	4.92	0.969	2.62	12.1	0.526	0.00	0.015	0.000	0.000	0.032	0.441	48.6	0.149	0.048	0.099	101
SMRF-0230 (B)	07-2640	10.0	8.37	0.058	1.02	0.073	0.028	10.2	0.0	0.190	4.97	1.00	2.54	12.1	0.573	0.00	0.016	0.000	0.000	0.029	0.450	48.6	0.155	0.051	0.099	100
SMRF-0231 (A)	07-2641	10.4	5.76	0.036	1.13	0.061	0.028	10.9	0.0	0.146	4.77	1.01	2.70	12.4	0.513	0.00	0.000	0.000	0.000	0.026	0.402	49.9	0.124	0.030	0.130	100
SMRF-0231 (B)	07-2641	10.5	5.73	0.037	1.13	0.063	0.025	11.1	0.0	0.139	4.82	1.04	2.69	12.3	0.547	0.00	0.000	0.000	0.000	0.029	0.411	50.5	0.127	0.031	0.134	101
SMRF-0232 (A)	07-2642	10.4	5.60	0.037	1.14	0.080	0.030	11.2	0.0	0.138	4.79	1.05	2.80	12.3	0.574	0.00	0.000	0.000	0.000	0.026	0.414	50.1	0.117	0.032	0.139	101
SMRF-0232 (B)	07-2642	10.5	5.57	0.037	1.16	0.077	0.033	11.0	0.0	0.152	4.84	1.03	2.75	12.4	0.521	0.00	0.000	0.000	0.000	0.028	0.417	50.5	0.114	0.031	0.136	101
					Fe(2+)	Fe(2+)																				
Sample	Lab ID	Fe(2+)	Fe(3+)	Fe(total)	Fe(3+)	Fe(total)																				
EA		0.051	0.204	0.255	0.250	0.200																				
<u> </u>		0.001	0.204	0.200	0.200	0.200																				
SMRF-0230 (A)	07-2640	0.089	0.355	0.444	0.251	0.200																				
SMRF-0230 (B)	07-2640		0.355	0.444	0.251	0.200																				
SMRF-0231 (A)	07-2641	0.090	0.327	0.417	0.275	0.216																				
SMRF-0231 (B)	07-2641	0.091	0.330	0.421	0.276	0.216																				
SMRF-0232 (A)	07-2642	0.110	0.377	0.487	0.292	0.226																				
SMRF-0232 (B)	07-2642	0.109	0.379	0.488	0.288	0.223																				

Notes: SMRF-0230 is last pour glass sample (7/10/07 @ 1740 hours) for SB4/Frit 510 test SMRF-0231 is last pour glass sample (7/12/07 @ 1255 hours) for SB4/Frit 418 test SMRF-0232 is drain glass sample after SB4/Frit 418 test This page intentionally left blank.

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