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THE IMPACT OF THE SOURCE OF ALKALI ON SLUDGE BATCH 3 MELT RATE (U)

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

April 2005

Immobilization Technology 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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REVIEWS AND APPROVALS

AUTHORS:

M. E. Smith, Immobilization Technology Section

nilla

D. H. Miller, Immobilization Technology Section

mithin n Jones

T. M. Jones, Immøbilization Technology Section

TECHNICAL REVIEWER:

D. K. Peeler, Immobilization Technology Section

APPROVERS

En Haltenhalt	6-01-05
E. W. Holtzscheiter, Manager, Immobilization Technology Section	Date
Shara & Mana	5/27/05
S. L. Marra, Manager, Glass Formulation & Process Development	Date
las find	6-10-05
J. E. Occhipinti, Manager, DWPF Process Engineering	Date

5/23/05 Date

24/05 Date

<u> 5/24/05</u> Date

5-24-05

Date

EXECUTIVE SUMMARY

Previous Savannah River National Laboratory (SRNL) melt rate tests in support of the Defense Waste Processing Facility (DWPF) have indicated that improvements in melt rate can be achieved through an increase in the total alkali of the melter feed. Higher alkali can be attained by the use of an "underwashed" sludge, a high alkali frit, or a combination of the two. Although the general trend between melt rate and total alkali (in particular Na₂O content) has been demonstrated, the question of "does the source of alkali (SOA) matter?" still exists. Therefore the purpose of this set of tests was to determine if the source of alkali (frit versus sludge) can impact melt rate.

The general test concept was to transition from a Na₂O-rich frit to a Na₂O-deficient frit while compensating the Na₂O content in the sludge to maintain the same overall Na₂O content in the melter feed. Specifically, the strategy was to vary the amount of alkali in frits and in the sludge batch 3 (SB3) sludge simulant (midpoint or baseline feed was SB3/Frit 418 at 35 % waste loading) so that the resultant feeds had the same final glass composition when vitrified.

A set of SOA feeds using frits ranging from 0 to 16 weight % Na_2O (in 4% increments) was first tested in the Melt Rate Furnace (MRF) to determine if indeed there was an impact. The dry-fed MRF tests indicated that if the alkali is too depleted from either the sludge (16% Na_2O feed) or the frit (the 0% Na_2O feed), then melt rate was negatively impacted when compared to the baseline SB3/Frit 418 feed currently being processed at DWPF. The MRF melt rates for the 4 and 12% SOA feeds were similar to the baseline SB3/Frit 418 (8% SOA) feed.

Due to this finding, a smaller subset of SOA feeds that could be processed in the DWPF (4 and 12% SOA feeds) was then tested in the Slurry-fed Melt Rate Furnace (SMRF). The results from a previous SMRF test with SB3/Frit 418 (Smith et al. 2004) were used as the SMRF melt rate of the baseline feed. The SOA SMRF test results agreed with those of the MRF tests for these two feeds as the melt rates were similar to the baseline SB3/Frit 418 feed. In other words, the source of alkali was close enough to the baseline feed as to not negatively impact melt rate. Based on these results, there appears to be an acceptable range of the source of alkali that results in the highest melt rate for a particular sludge batch. If, however, the alkali is too depleted from either the sludge or the frit, then melt rate will be lower. Although SB3 simulant sludge and Frit 418 were used for these tests, it was not the intent of these tests to determine an optimum source of alkali range for SB3. Rather, the findings of these tests should be used to help in the decision process for future sludge batch washing and/or blending strategies. The results, however, do confirm that the current processing of SB3 is being performed in the proper source of alkali range.

Because all of this testing was performed on small-scale equipment with slurried, non-radioactive simulant, the exact impact of the source of alkali with SB3 in the DWPF melter could not be fully evaluated.

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CPC	Chemical Process Cell
DSC	Differential Scanning Calorimetry
DWPF	Defense Waste Processing Facility
HTXRD	High Temperature X-Ray Diffraction
LMR	Linear Melt Rate
MRF	Melt Rate Furnace
REDOX	REDuction/OXidation
SB2	Sludge Batch 2
SB3	Sludge Batch 3
SEM/EDS	Scanning Electron Microscopy/Energy-Dispersive Spectroscopy
SME	Slurry Mix Evaporator
SMRF	Slurry-Fed Melt Rate Furnace
SOA	Source of Alkali
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
TGA	Thermogravimetric Analysis
WSRC	Westinghouse Savannah River Company
WTF	Waste Throughput Factor

LIST OF ACRONYMS

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WSRC-TR-2005-00177

1.0 INTRODUCTION

Previous melt rate tests have indicated that improvements in melt rate can be achieved through an increase in the total alkali of the melter feed. Higher alkali can be attained by the use of an "underwashed" sludge, a high alkali frit, or a combination of the two. Although the general trend between melt rate and total alkali (in particular Na₂O content) has been demonstrated, the question of "does the source of alkali (SOA) matter?" still exists. Therefore the purpose of this set of tests was to determine if the source of alkali (frit versus sludge) can impact melt rate. This was done with the understanding that the source of alkali may impact acid stoichiometry requirements and hence rheology of the feeds as well as the tendency of the feed to cause problematic off-gas surges in the melter.

The general test concept was to transition from a Na₂O-rich frit to a Na₂O-deficient frit while compensating the Na₂O content in the sludge to maintain the exact overall Na₂O content in the melter feed. If the source of alkali does impact melt rate and ultimately overall waste throughput, this series of tests may allow SRNL to provide guidance on the washing and/or blending strategies for future sludge batches. The approach for this series of tests was to vary the amount of alkali in frits and in the SB3 sludge simulant (midpoint or baseline feed was SB3/Frit 418 at 35% waste loading) so that the resultant feeds had the same final glass composition when vitrified. The feeds for these SOA tests are described based on the percent Na₂O that is in the frit for that feed. For example, SOA test feed that used a frit with 4 weight % Na₂O was called 4% SOA feed. Therefore the baseline SB3/Frit 418 feed is called 8% SOA feed as Frit 418 has 8% Na₂O.

The waste loadings of these feeds were varied to keep the final glass composition constant. The goal was to also maintain the acid stoichiometry and the REDuction/OXidation (REDOX) constant at the current DWPF targets for each. Therefore an acid stoichiometry of 155% was selected for the range of sludge compositions to be tested. The target REDOX was 0.2 but as long as the final REDOX was within the range of 0.1 to 0.33, no impact on melt rate was expected based on previous testing (Smith et al. 2004). A set of SOA feeds using frits ranging from 0 to 16% Na₂O (in 4% increments) was first tested in the MRF to determine if indeed there was an impact. Because an impact was observed in the MRF tests, a smaller subset of SOA feeds (4 and 12% SOA feeds) was then tested in the SMRF. The results from a previous SMRF test with SB3/Frit 418 (Smith et al. 2004) were used as the SMRF melt rate of the baseline feed.

This task was initiated by DWPF Engineering via Task Technical Request (TTR) HLW-DWPF-2004-0030 to SRNL. The work was performed per Task Technical and Quality Assurance Plan WSRC-RP-2004-00713.

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 slurried, non-radioactive 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 Details

The SOA tests were conducted using the current SB3 sludge simulant and process strategy (155% acid stoichiometry, 0.2 REDOX, Frit 418, and 35% waste loading) as the baseline (Smith et al. 2004). Frit 418 contains 8% Na₂O as well other oxides given in Table 2-1. Frit compositions were targeted at approximately 0, 4, 12, and 16% Na₂O with all other test frit species then being renormalized from the baseline Frit 418. The amount of sodium in the sludge as well as the waste loading was adjusted to maintain a constant glass composition for each of the feeds tested.

A comparison of the target compositions of the SOA frits is given below in Table 2-1. The frits (except Frit 418) are named SOA frits with the amount of Na_2O in the frit given in the description. For example, the SOA frit with a target of 4% Na_2O is called 4% SOA frit. In addition, the resultant feed made with each SOA frit is named based on the amount of Na_2O in the frit used. For example, the SOA feed using the 4% SOA frit is called 4% SOA feed. Appendix A gives the actual compositions of all of the SOA frits made for the MRF and SMRF SOA tests.

	0% SOA	4% SOA	Frit 418	12% SOA	16% SOA
Oxide	Frit	Frit	(8% SOA)	Frit	Frit
B_2O_3	8.69	8.35	8	7.66	7.32
Li ₂ O	8.69	8.35	8	7.66	7.32
Na ₂ O	0.05	4.02	8	11.95	15.80
SiO ₂	82.57	79.29	76	72.73	69.55
Total	100	100	100	100	100

 Table 2-1.
 Target SOA Frit Compositions (Weight %)

Sludge was produced for each test by blending the existing SB2 and SB3 simulants and then adding trim species as required to meet composition requirements. Sodium is present in the sludge as a number of species, including carbonate, fluoride, hydroxide, nitrate, nitrite, phosphate, and sulfate. In order to maintain a constant glass composition, the fluoride, phosphate, and sulfate were held constant during the tests. The amount of carbonate, hydroxide, nitrate, and nitrite was adjusted in proportion to meet the required sodium concentrations in the sludge. Appendix B gives the targeted sludge compositions for the SOA tests.

Waste loading adjustment was required since removal of sodium from the frit requires less frit in the glass to maintain constant boron oxide, lithia, and silica concentrations because the concentration of these species in the frit increases as sodium is removed. Therefore, waste loading was decreased as the sodium concentration of the sludge was decreased. The reduction in Na₂O content in the sludge corresponds to additional washing. Likewise, waste loading was increased as the sludge was decreased and less sodium was added to the frit. Table 2-2 gives the target waste loadings for the various SOA feeds.

Table 2-2.	Target Waste	Loadings for	the SOA	Feed MRF	and SMRF	Tests
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SOA Feed	Target WL
0% SOA	40.2
4% SOA	37.7
SB3/Frit 418 (8%SOA)	35.0
12% SOA	32.1
16% SOA	29.0

The sludge was processed through lab-scale simulations of the Chemical Process Cell (CPC) process to provide feed for the melt rate program. Five Sludge Receipt and Adjustment Tank (SRAT) cycles were performed to make the 0%, 4%, 8%, 12% and 16% SOA sludges in 4-L vessels. This provided five 2.5-L batches of slurry to be dried as feed for the MRF SOA tests. Four SRAT/Slurry Mix Evaporator (SME) cycles were performed in 22-L vessels to provide four 14-L batches of slurry feed for the SMRF tests. These feeds consisted of two 14-L batches each of 4 and 12% SOA feeds. After review of all of the feed analyses, all SOA SRAT (for the MRF tests) and SRAT/SME (for the SMRF tests) products were deemed acceptable for use in the tests.

One of the main SOA test premises is that all of the feeds and hence final glass compositions for the various SOA feeds were basically the same. For MRF tests, the SRAT product is analyzed and the proper amount of frit is added but this feed is usually not analyzed. To ensure that indeed the final glasses compositions were the same for all of the MRF SOA tests, samples of each of the MRF glasses produced were taken and analyzed to confirm this premise. For the SMRF tests, feed samples for the two SOA feeds were compared with the previous tested SB3/Frit 418 SMRF feed that was used to determine the 8% SOA feed melt rate (Smith et al. 2004). Appendix C gives the analyses of all of these samples. Indeed, all of the MRF SOA glasses and the SMRF SOA feeds were very similar in composition.

The run plans for the five 4-L SRAT runs for MRF tests are given in SRNL inter-office memorandums SRT-GPD-2004-00084, SRT-GPD-2004-00085, SRT-GPD-2004-00086, SRT-GPD-2004-00087, and SRT-GPD-2004-00088. The run plan for the 22-L SRAT/SME runs for the SMRF tests are given in SRNL inter-office memorandums SRT-GPD-2005-00012 and SRT-GPD-2005-00013. Details of the 4-L SRAT runs for the MRF SOA tests and the 22-L SRAT/SME runs for the SMRF SOA tests are documented by Lambert and Stone (2005).

2.2 MRF SOA 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 ~7", and an insulating sleeve and a 1200 mL stainless steel beaker (6" deep) were inserted from the top. 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 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 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). After cooling down, the beakers are then sectioned.

The relative melt rate is determined by measuring the height of the glass layer in the bottom of each sectioned beaker at 0.25" intervals. The average height and duration in the furnace is used to yield a relative linear melt rate number (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 SOA Testing Details

The SMRF was heated up on February 14, 2005 with 6.4 kg of a prefabricated SB3 Frit 418 glass targeting 35% waste loading. The run plan used for the SMRF tests was SRT-GPD-2005-00005 ("Run

Plan for the Source of Alkali Runs in the Slurry Fed Melter Rate Furnace"). The SRNL notebook used was WSRC-NB-2004-00123. SMRF SOA testing of SOA 4% and SOA 12% feeds were conducted the following two days.

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 feed initiation setpoint of 750°C was 20 seconds. As in past SMRF runs, the measured current for the melt pool and vapor space heaters were both about 20 amps for the two tests.

Before the testing was started, it was noted that the 12% SOA feed was very thick. The thickness of the feed was probably because the total acid requirement was low for this overwashed sludge. Therefore the feeding of this 12% SOA feed with the SMRF feed system was tested before the SMRF was heated up. It was determined that this feed at 50 weight % solids could not be pumped in the targeted amount (90-100 grams) per 20 second feed cycle. Some of this feed was diluted to 45 weight % solids and feeding at the proper rate was accomplished. Therefore a decision was made to test the 12% SOA feed at 45 weight % solids only and to then test the 4% SOA feed at 50 weight % solids and then at 45 weight % solids. This was so that direct melt rate comparisons could be made for the same feeds at the same weight percent solids. Measured rheological properties of the 4 and 12% SOA SMRF feeds before and after dilution are given below in Table 2-3. Dilution of the 12% SOA feed to 45 weight % solids lowered the yield stress to about the same value as the 4% SOA feed at 50 weight % solids. A more detailed discussion about the rheological properties of the SOA feed at 50 weight % solids. A more detailed discussion about the rheological properties of the SOA feed at 50 weight % solids.

	Consistency	Yield Stress
SOA Feed (Targeted Wt% Solids)	(cP)	(Pa)
4% SOA 22-L SME Run 1 (50)	56.0	27.5
4% SOA 22-L SME Run 2 (50)	94.0	32.7
*4% SOA Feed (45)	39.1	8.4
12% SOA 22-L SME Run 1 (50)	193.0	71.4
12% SOA 22-L SME Run 2 (50)	93.9	64.1
*12% SOA Feed (45)	53.9	34.0

Table 2-3. Rheological Properties of the SMRF SOA Feeds Before and After Dilution

*Note: These feeds are the mixture of SME Runs 1 and 2 (SMRF SOA feed)

The feed dilution decision was also based on the fact that the reference (or baseline) SB3/Frit 418 feed had been tested at both 50 and 45 weight % solids as documented by Smith, et al. (2004). The SMRF SOA targeted test conditions were the same as this previously run baseline SB3/Frit 418 test. This allowed the best possible comparison of the three feeds. The melt rate results of this SB3/Frit 418 SMRF test (155% acid stoichiometry, 0.2 REDOX, 35% waste loading) were used as the baseline melt rates (8% SOA feed at 45 and 50 weight % solids) for the SOA tests.

3.0 RESULTS AND DISCUSSION

3.1 MRF SOA Results

The results of the MRF SOA tests (melt rate and waste throughput factor or WTF) are shown in Table 3-1. The melt rates cited are linear melt rates (LMR) with the units being inches/hour of melt time. Waste throughput factor (WTF) is calculated as follows:

WTF = LMR x WL (waste loading)

Figure 3-1 is a plot of the impact of SOA on melt rate and waste throughput factor.

Table 3-1. Melt Rates (LMR) and Waste Throughput Factors (WTF) for the MRF SOA Tests

SOA MRF Feed	LMR (in/hr)	WTF (Target WL)
0% SOA	0.39	15.7 (40.2)
4% SOA	0.55	20.7 (37.7)
SB3/Frit 418 (8%SOA)	0.59	20.7 (35.0)
12% SOA	0.55	17.7 (32.1)
16% SOA	0.38	11.0 (29.0)



Figure 3-1. Impact of SOA on Melt Rate (LMR - in/hr) and Waste Throughput Factor

The highest linear melt rate was achieved with the 8% SOA feed (SB3/Frit 418 baseline feed). The 4% and 12% SOA feeds, however, had linear melt rates that were almost as high (both at 0.55 in/hr vs. 0.59 in/hr for the 8% SOA feed). Indeed, these three melt rates could be considered as basically equivalent. A significant drop off in melt rate (approximately 30%) was observed for the 0% and 16% SOA feeds. The data indicates that there is an optimum range in the source of alkali that gives the highest melt rate. If the alkali is too depleted from either the frit (underwashed sludge) or from the sludge (overwashed sludge), then melt rate and waste throughput will be negatively impacted. The one difference in the

LMR and WTF curves is that the WTF peak is shifted slightly to lower SOA feeds (closer to 4% SOA feed). Part of this shift may be because there is more sodium in the waste. Appendix D has pictures of the some of the SOA MRF test beakers after they were cross-sectioned. It was observed that the 0% SOA MRF test resulted in expansion of the unmolten feed to the point that it lifted the M-board cover that is placed on top of each MRF beaker about one inch. The beakers shown in Appendix D were cut in such a way that this feed expansion was removed before the post 0% SOA MRF test picture was taken.

The reasons for the decrease in melt rate with the 0 and 16% SOA feeds could not be determined with these tests. However, theories can be made as to the causes in the drop in melt rate for these two feeds. For the 0 % SOA feed, there are several possible drivers. First, the frit is the most refractory of all the frits used. Therefore melting (or liquid phase formation) may be only occurring near the hot glass section of the cold cap. In addition, the sludge was the least washed (requiring higher amounts of acid addition and also more anions being released as off-gas during the melting process) of those tested and targeted the highest waste loading (40.2%) to maintain the same final glass composition. Both of these factors would cause the amount of off-gas to be higher, thereby potentially reducing heat transfer efficiencies which would negatively impact melt rate. With the 16% SOA feed (i.e. the most washed), the refractoriness (lowest amount of sodium) of the sludge may have resulted in minimal liquid state formation. In addition, the 16% SOA frit would have been the most fluid (for a given temperature), but it could be melting or softening in a temperature range that would trap off-gas from reactions occurring in the cold cap. This could result in foam formation which would inhibit heat transfer in the cold cap.

Finally, there has been some question about the overall repeatability of the MRF melt rate results. Including the 8% MRF SOA test results documented in this report, there have been four different MRF runs that used SB3 SRAT product with Frit 418 at 35% WL and similarly targeted acid stoichiometry and REDOX. All four of these tests had linear melt rates in the range of 0.58 to 0.60 inches/hour. This data indicates that indeed the MRF can give fairly repeatable melt rate results.

3.2 SMRF SOA Results

Details of the SMRF SOA runs are given in Section 2.3. The 4% SOA (at 50 and 45 weight % solids) and 12% SOA (at 45 weight % solids) feeds were tested. As discussed previously, the 12% SOA feed at 50 weight % solids was not tested as it could not be pumped at the target rate of 90-100 grams per 20 second feed cycle. The results of these tests were compared to the SMRF melt rates of a previously run 8% SOA feed (SB3/Frit 418 feed at 35 % waste loading) as documented by Smith et al. (2004). It should be noted that the feeds cited in Table 3-3 of this reference report were not named correctly. The names for the 185/50 feed (185 % acid stoichiometry/50 weight % solids) and the 155/45 feed (155 % acid stoichiometry/45 weight % solids) were erroneously switched. This error does not change the overall findings of that report, but is discussed here to ensure that there is no confusion with the SMRF melt rates of those tests as cited in this document. Table 3-2 summarizes the results of the SMRF SOA tests. Melt rate values are given in grams/minute. Tables 3-3 summarizes the vapor space and melt pool heater power usages for the tests. The data shows that the power usages were similar for these tests.

	4% SOA Feed	*8% SOA Feed	12% SOA Feed
50 weight % solids	13.8	14.1	
45 weight % solids	14.4	**11.5/13.1	13.6

Fable 3-2. Melt Rates	(g/min) for th	e SMRF	SOA Tests
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*baseline feed – SB3/Frit 418 at 35% waste loading, 155% acid stoichiometry, 0.2 REDOX (Smith, et al. 2004) **test only lasted 2 hours and there were several feed stoppages. The pour rate for the last 17 minutes of the test was up to 13.1 g/min and therefore is cited as a possible higher melt rate for this test.

	4% SOA (Vapor/Melt)	8% SOA (Vapor/Melt)	12% SOA (Vapor/Melt)
50 weight % solids	93.3 / 47.6	95.6 / 48.4	
45 weight % solids	96.8 / 44.3	97.3 / 45.5	95.6 / 49.5

Table 3-3. SMRF Vapor Space and Melt Pool Heater Power (BTU/min) Usages for the SOA Tests

The results of the tests are somewhat difficult to compare. One thing to note is that the SMRF top was taken off and then replaced after a series of sight glass tests were performed for a proposed larger melter tool. The first SMRF tests run after this top head replacement was this series of SOA tests. The impact of this top head removal and replacement (same top head) is unknown, although it should be minimal.

The melt rates for the 4% SOA and 8% SOA feeds (at 50 weight % solids) were similar. This agrees with MRF SOA tests. If the 12% SOA feed could have been tested at 50 weight % solids as well, a more definitive conclusion may have been possible.

The results for the feeds at 45 weight % solids are somewhat more difficult to use for making any conclusions on the source of alkali. The slight increase in melt rate for the 4% SOA feed when it was diluted from 50 to 45 weight % solids could be explained in that melt rates were so close that they could be considered as equal. As given in the notes below Table 3-2, the results of the 8% SOA test may not be correct due to the shortness of the test and the problems that occurred during the test. If the melt rate for the last 17 minutes of the test is used (13.1 g/min), then the results indicate that the melt rates for all three feeds are similar. This would again agree with the MRF SOA test results.

After considering all of the SMRF data as well as the various caveats discussed above, an overall conclusion from the SMRF SOA tests is that for the range of SOA feeds tested (4-12%), the source of alkali had little impact on melt rate. The 0 and 16% SOA feeds tested by the dry fed MRF were not run in the SMRF due to their low MRF melt rates, the processing problems observed during 0% SOA feed MRF testing, and the impractical amount of washing that would be required for the 16% SOA feed sludge.

4.0 CONCLUSIONS

A series of dry-fed (using the MRF) and slurry-fed tests (using the SMRF) have been performed to investigate the impact of the source of alkali (frit versus sludge) on melt rate on the DWPF SB3/Frit 418 feed system. Due to some problems in testing such as the inability to feed the 12% SOA feed (at 50 weight % solids) in the SMRF at the required rate per feed cycle, some direct comparisons of melt rates in the SMRF tests could not be made. The overall objective of the test was met, however, and the following conclusions can be made based on this work:

- The source of alkali (frit versus sludge) can impact melt rate as shown by the MRF SOA tests. From the MRF SOA tests findings, there appears to be a region for the source of alkali that results in the highest melt rates. If the alkali is too depleted from either the frit or the sludge, then melt rate is negatively impacted.
- The SMRF SOA tests agreed with the MRF tests in that the 4, 8, and 12 % SOA feeds gave similar melt rates. The 0 and 16% SOA feeds were not tested due to the much lower melt rates observed for both of these feeds in the MRF SOA tests, the processing problems observed during 0% SOA feed MRF testing, and the impractical amount of washing that would be required for the 16% SOA feed sludge.
- For the SB3/Frit 418 feed system, the current washing strategy is in the region of highest melt rate and waste throughput.
- Melt rates for DWPF sludge batches can be negatively impacted if the sludge is overwashed or underwashed.

5.0 RECOMMENDATIONS/PATH FORWARD

Based on the results of the SB3 SOA tests, the following recommendations are given:

- Thermal analysis techniques such as high temperature X-ray diffraction (HTXRD), quantitative X-ray diffraction, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), and scanning electron microscopy with energy-dispersive spectroscopy (SEM/EDS) should be performed on SB3 SOA feeds to access the impact of the source of alkali on the reaction pathways and kinetics during the batch to glass conversion process. The HTXRD work has already been started.
- The source of alkali should be considered when determining the sludge washing strategies and frits for future DWPF sludge batches to ensure the highest possible equivalent canister production rates.

6.0 REFERENCES

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APPENDIX A – ANALYSES OF THE SOA FRITS MADE FOR THE MRF AND SMRF TESTS

Sample ID	Lab ID	В	Cr	Fe	Li	Na	Ni	Si	
0% Na Frit (A)	04-2151*	2.83	0.016	0.049	4.16	0.090	<0.010	38.6	
0% Na Frit (B)	04-2151*	2.72	0.017	0.044	4.20	0.050	<0.010	38.6	
4% Na Frit (A)	04-2176*	2.66	0.003	0.048	3.79	3.17	<0.010	37.6	
4% Na Frit (B)	04-2176*	2.68	0.003	0.043	3.80	3.12	<0.010	38.1	
12% Na Frit (A)	04-2151*	2.45	0.018	0.055	3.66	8.98	<0.010	33.9	
12% Na Frit (B)	04-2151*	2.45	0.017	0.046	3.67	8.74	<0.010	33.7	
16% Na Frit (A)	04-2151*	2.39	0.017	0.040	3.54	11.6	<0.010	32.7	
16% Na Frit (B)	04-2151*	2.32	0.017	0.043	3.52	11.5	<0.010	31.8	
		B2O3	Cr2O3	Fe2O3	Li2O	Na2O	NiO	SiO2	Total
0% Na Frit (A)	04-2151*	9.11	0.023	0.070	8.94	0.122	0.000	82.6	101
0% Na Frit (B)	04-2151*	8.76	0.025	0.063	9.03	0.068	0.000	82.6	101
0% Na2O Frit	Average	8.94	0.02	0.07	8.99	0.09	0.00	82.60	
	Target	8.69	0.00	0.00	8.69	0.05	0.00	82.60	
4% Na Frit (A)	04-2176*	8.57	0.004	0.069	8.15	4.28	0.000	80.5	102
4% Na Frit (B)	04-2176*	8.63	0.004	0.061	8.17	4.21	0.000	81.5	103
4% Na2O Frit	Average	8.60	0.00	0.07	8.16	4.25	0.00	81.00	
	Target	8.35	0.00	0.00	8.35	4.02	0.00	79.29	
12% Na Frit (A)	04-2151*	7.89	0.026	0.079	7.87	12.1	0.000	72.5	101
12% Na Frit (B)	04-2151*	7.89	0.025	0.066	7.89	11.8	0.000	72.1	100
12% Na2O Frit	Average	7.89	0.03	0.07	7.88	11.96	0.00	72.33	
	Target	7.66	0.00	0.00	7.66	11.95	0.00	72.73	
16% Na Frit (A)	04-2151*	7.70	0.025	0.057	7.61	15.7	0.000	70.0	101
16% Na Frit (B)	04-2151*	7.47	0.025	0.061	7.57	15.5	0.000	68.1	98.7
16% Na2O Frit	Average	7.58	0.02	0.06	7.59	15.59	0.00	69.02	
	Target	7.32	0.00	0.00	7.32	15.80	0.00	69.55	

TABLE A-1. Analyses of the SOA Frits Used for the MRF SOA Tests

SRTC Mobile Laboratory Customer: David Peeler Date: 1/28/05 Samples: 12% Na Frit 1, 2, 3 - 4% Na Frit 1, 2, 3 Lab ID: 05-0079 through 05-0084 Units: elemental and oxide wt% Sample Preparation: LiBO2 and Na2O2 Preps Comments: Samples run in duplicate

 12% Na Li2O Norm
 4% Na Li2O Norm

 7.25
 7.71

Sample ID	Lab ID	В	Cr	Fe	Li	Na	Ni	Si	
12% Na Frit 1 (A)	05-0079	2.42	0.012	0.058	3.34	9.02	<0.010	34.2	
12% Na Frit 1 (B)	05-0079	2.43	0.013	0.055	3.41	8.76	<0.010	34.2	
12% Na Frit 2 (A)	05-0080	2.39	0.014	0.068	3.29	8.61	<0.010	33.2	
12% Na Frit 2 (B)	05-0080	2.40	0.014	0.064	3.30	8.70	<0.010	33.9	
12% Na Frit 3 (A)	05-0081	2.40	0.014	0.048	3.39	8.84	<0.010	33.8	
12% Na Frit 3 (B)	05-0081	2.34	0.013	0.059	3.31	8.80	<0.010	33.1	
4% Na Frit 1 (A)	05-0082	2.62	0.015	0.055	3.67	3.08	<0.010	37.3	
4% Na Frit 1 (B)	05-0082	2.62	0.015	0.052	3.66	2.93	<0.010	37.2	
4% Na Frit 1 (A)	05-0083	2.85	0.015	0.083	3.66	2.96	<0.010	37.7	
4% Na Frit 1 (B)	05-0083	2.77	0.014	0.090	3.59	2.94	<0.010	37.4	
4% Na Frit 1 (A)	05-0084	2.76	0.015	0.052	3.51	3.02	<0.010	36.6	
4% Na Frit 1 (B)	05-0084	2.78	0.014	0.051	3.52	2.93	<0.010	37.4	
		B2O3	Cr2O3	Fe2O3	Li2O	Na2O	NiO	SiO2	Total
12% Na Frit 1 (A)	05-0079	7.79	0.018	0.083	7.18	12.2	0.000	73.2	100
12% Na Frit 1 (B)	05-0079	7.82	0.019	0.079	7.33	11.8	0.000	73.2	100
12% Na Frit 2 (A)	05-0080	7.70	0.020	0.097	7.07	11.6	0.000	71.0	97.6
12% Na Frit 2 (B)	05-0080	7.73	0.020	0.092	7.10	11.7	0.000	72.5	99.2
12% Na Frit 3 (A)	05-0081	7.73	0.020	0.069	7.29	11.9	0.000	72.3	99.4
12% Na Frit 3 (B)	05-0081	7.53	0.019	0.084	7.12	11.9	0.000	70.8	97.5
Average		7.72	0.02	0.08	7.18	11.86	0.00	72.19	99.06
Target		7.66	0.00	0.00	7.66	11.95	0.00	72.73	
4% Na Frit 1 (A)	05-0082	8.44	0.022	0.079	7.89	4.16	0.000	79.8	100
4% Na Frit 1 (B)	05-0082	8.44	0.022	0.074	7.87	3.96	0.000	79.6	100
4% Na Frit 1 (A)	05-0083	9.18	0.022	0.119	7.87	4.00	0.000	80.7	102
4% Na Frit 1 (B)	05-0083	8.92	0.020	0.129	7.72	3.97	0.000	80.0	101
4% Na Frit 1 (A)	05-0084	8.89	0.022	0.074	7.55	4.08	0.000	78.3	98.9
4% Na Frit 1 (B)	05-0084	8.95	0.020	0.073	7.57	3.96	0.000	80.0	101
Average		8.80	0.02	0.09	7.74	4.02	0.00	79.75	100.43
Target		8.35	0.00	0.00	8.35	4.02	0.00	79.29	

TABLE A-2. Analyses of the 4% and 12% SOA Frits Used for the SMRF SOA Tests

APPENDIX B - TARGET COMPOSITIONS (OXIDE BASIS) FOR THE SOA SLUDGES

	0% SOA	4% SOA	8 % SOA	12% SOA	16% SOA
OXIDE	SLUDGE	SLUDGE	SLUDGE	SLUDGE	SLUDGE
AI2O3	14.82	15.79	17.01	18.56	20.55
Ag2O	0.02	0.02	0.02	0.02	0.02
BaO	0.12	0.13	0.14	0.16	0.17
CaO	3.19	3.40	3.67	4.00	4.43
Cr2O3	0.20	0.22	0.23	0.25	0.28
CuO	0.17	0.18	0.19	0.21	0.23
Fe2O3	36.07	38.44	41.40	45.17	50.01
Gd2O3	0.07	0.07	0.08	0.08	0.09
K2O	0.04	0.04	0.04	0.05	0.05
MgO	3.71	3.95	4.26	4.64	5.14
MnO	5.65	6.02	6.48	7.07	7.83
Na2O	30.84	26.30	20.63	13.41	4.13
NiO	1.11	1.18	1.27	1.39	1.54
P2O5	0.19	0.21	0.22	0.24	0.27
PbO	0.06	0.06	0.07	0.07	0.08
RhO2	0.01	0.01	0.01	0.02	0.02
RuO2	0.06	0.06	0.07	0.07	0.08
SiO2	2.03	2.17	2.33	2.55	2.82
ZnO	0.33	0.35	0.37	0.41	0.45
ZrO2	0.35	0.37	0.40	0.43	0.48
SO4	0.96	1.03	1.10	1.20	1.33

Table B-1. Target Compositions (Oxide Basis) of SOA Sludges

APPENDIX C – ANALYSES OF THE SOA MRF GLASSES AND SOA SMRF FEEDS

SRTC Mobile Laboratory																														
Customer: Don Miller																											—			
Date: 1/14/05																														
Sample ID: SMRF 0181 - 0185																												L		
Lab ID: 05-0048, 04-0049, 05-0050, 0	05-005	51, 05-0052																												
Sample Preparation: LiBO2 and Na2O2/NaOH fusions		s																								<u> </u>	<u> </u>			
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							0.10	0.00	0.000			0.1000	1/00	1 000	1.10.0												<u> </u> '			
oxide wt% - calcined 1100C		<u>Ag20</u>	<u>AI203</u>	<u>B203</u>	BaO	CaO	CaO	<u>CeO2</u>	<u>Cr203</u>	Cuo	Fe203	<u>Gd2O3</u>	<u>K20</u>	La203	<u>Li20</u>	MgO	MnO	MoO3	<u>Na2O</u>	NiO	P205	PbO	<u>SO4</u>	<u>Sb2O3</u>	SiO2	<u>SnO</u>	<u>TiO2</u>	ZnO	<u>ZrO2</u>	Totals
181 Avg - 0%		0.000	6.133	5.152	0.036	0.969	0.000	0.000	0.096	0.075	14.515	0.024	0.061	0.000	5.526	1.645	1.883	0.000	12.899	0.420	0.074	0.000	0.477	0.000	49.969	0.020	0.017	0.151	0.240	100.38
182 Avg - 4%		0.000	6.143	5.297	0.036	0.935	0.000	0.000	0.091	0.066	14.343	0.024	0.059	0.000	5.923	1.575	1.864	0.000	12.980	0.404	0.071	0.038	0.477	0.000	51.574	0.020	0.017	0.144	0.247	102.33
183 Avg - 8%		0.000	6.303	4.846	0.038	0.977	0.000	0.000	0.085	0.072	13.749	0.025	0.073	0.000	5.633	1.622	1.845	0.000	12.920	0.377	0.073	0.039	0.479	0.000	51.253	0.019	0.038	0.144	0.252	100.86
184 Avg- 12%		0.000	6.152	5.361	0.036	0.911	0.000	0.000	0.082	0.074	14.271	0.025	0.060	0.000	5.751	1.562	1.864	0.000	12.845	0.383	0.071	0.039	0.453	0.000	51.895	0.018	0.018	0.143	0.258	102.27
185 Avg - 16%		0.000	6.020	5.474	0.035	0.880	0.000	0.000	0.093	0.083	14.271	0.026	0.067	0.000	5.880	1.726	1.812	0.000	13.244	0.418	0.070	0.037	0.524	0.000	52.751	0.022	0.017	0.142	0.237	103.83
																											—			
oxide wt% - calcined 1100C																														
		MnO	MoO3	Na2O	NiO	P205	PbO	<u>SO4</u>	Sb2O3	SiO2	SnO	TiO2	ZnO	ZrO2	Totals												1 '	1		
181 Avg - 0%		1.883	0.000	12.899	0.420	0.074	0.000	0.477	0.000	49.969	0.020	0.017	0.151	0.240	100.4															
182 Avg - 4%		1.864	0.000	12.980	0.404	0.071	0.038	0.477	0.000	51.574	0.020	0.017	0.144	0.247	102.3															
183 Avg - 8%		1.845	0.000	12.920	0.377	0.073	0.039	0.479	0.000	51.253	0.019	0.038	0.144	0.252	100.9															
184 Avg- 12%		1.864	0.000	12.845	0.383	0.071	0.039	0.453	0.000	51.895	0.018	0.018	0.143	0.258	102.3															
185 Avg - 16%		1.812	0.000	13.244	0.418	0.070	0.037	0.524	0.000	52.751	0.022	0.017	0.142	0.237	103.8															

Table C-1. MRF SOA Glass Analytical Results

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	-																											
Commits ID	Lab ID																	-										
Sample ID	LabiD	A.,		-	D-	0.	0.1	0.	0	F	0.1	~		M.,	M.,		N-	Na			Dh	-	01	01	T !	7	7.	
elemental wt%-calcined 1100C		Ag	AI	B	Ва	Ca	Cd	Cr	Cu	Fe	Gd	<u>K</u>		Mo	Mg	Mn	Na	Nd	NI	P	Pb	<u>s</u>	Sb	51	<u> 11</u>	Zn	<u>Zr</u>	
FPMR-0089 (A)	05-0111	0.020	3.39	1.58	0.046	0.672	<0.010	0.054	0.057	10.1	0.02	0.046	2.38	<0.020	0.854	1.39	9.40	<0.010	0.402	0.028	0.034	0.083	<0.050	23.6	0.010	0.116	0.189	
FPMR-0089 (B)	05-0111	0.011	3.43	1.57	0.046	0.665	<0.010	0.054	0.057	10.1	0.02	0.046	2.40	<0.020	0.843	1.40	9.30	<0.010	0.397	0.027	0.033	0.081	< 0.050	23.3	0.010	0.115	0.188	
FPMR-0101 (A)	05-0112	0.013	3.55	1.55	0.047	0.695	<0.010	0.056	0.081	10.3	0.02	0.050	2.34	<0.020	0.861	1.45	9.36	<0.010	0.407	0.039	0.034	0.114	<0.050	22.8	0.010	0.117	0.191	
FPMR-0101 (B)	05-0112	0.012	3.54	1.53	0.047	0.698	<0.010	0.056	0.078	10.2	0.02	0.048	2.35	<0.020	0.874	1.43	9.51	<0.010	0.399	0.028	0.034	0.118	<0.050	22.9	0.010	0.127	0.193	
FPMR-0111 (A)	05-0113	<0.010	3.47	1.62	0.045	0.663	<0.010	0.053	0.063	10.2	0.02	0.048	2.44	<0.020	0.826	1.42	9.15	<0.010	0.396	0.036	0.032	0.085	< 0.050	23.8	0.010	0.111	0.185	
FPMR-0111 (B)	05-0113	<0.010	3.46	1.59	0.044	0.651	<0.010	0.053	0.063	9.94	0.02	0.046	2.44	<0.020	0.819	1.37	9.07	<0.010	0.388	0.029	0.031	0.084	< 0.050	23.8	0.010	0.110	0.184	
FPMR-0119 (A)	05-0114	<0.010	3.49	1.58	0.044	0.700	<0.010	0.053	0.059	10.5	0.02	0.052	2.42	<0.020	0.861	1.40	9.53	<0.010	0.460	0.023	0.031	0.097	< 0.050	23.5	0.010	0.110	0.181	
FPMR-0119 (B)	05-0114	<0.010	3.47	1.58	0.044	0.698	<0.010	0.053	0.059	9.65	0.02	0.050	2.41	<0.020	0.864	1.38	9.29	<0.010	0.369	0.025	0.031	0.098	< 0.050	23.2	0.010	0.111	0.183	
oxide wt% - calcined 1100C		Aa2O	Al2O3	B2O3	BaO	CaO	CdO	Cr2O3	CuO	Fe2O3	Gd2O3	K20	Li2O	MoO3	MaQ	MnO	Na2O	Nd2O3	NiO	P2O5	PbO	SO4	Sb2O3	SiO2	TiO2	ZnO	ZrO2	Totals
EPMR-0089 (A)	05-0111	0.021	6.41	5.09	0.052	0.941	0.00	0.079	0.071	14.4	0.020	0.055	5.12	0.00	1.42	1.79	12.7	0.00	0.511	0.064	0.037	0.249	0.00	50.5	0.017	0.144	0.255	100.0
EPMR-0089 (B)	05-0111	0.012	6.48	5.06	0.052	0.931	0.00	0.079	0.071	14.4	0.020	0.055	5.16	0.00	1.40	1.81	12.6	0.00	0.504	0.062	0.036	0.243	0.00	49.9	0.017	0.143	0.254	99.2
EPMR-0101 (A)	05-0112	0.014	6.71	4.99	0.053	0.973	0.00	0.082	0.101	14.7	0.018	0.060	5.03	0.00	1.43	1.87	12.6	0.00	0.517	0.089	0.037	0.342	0.00	48.8	0.017	0.145	0.258	98.9
EPMR-0101 (B)	05-0112	0.013	6.69	4.93	0.053	0.977	0.00	0.082	0.098	14.6	0.018	0.058	5.05	0.00	1.45	1.84	12.8	0.00	0.507	0.064	0.037	0.354	0.00	49.0	0.017	0.157	0.261	99.1
EPMR-0111 (A)	05-0113	0.00	6.56	5.22	0.050	0.928	0.00	0.077	0.079	14.6	0.020	0.058	5.25	0.00	1.37	1.83	12.0	0.00	0.503	0.082	0.035	0.255	0.00	50.9	0.017	0.138	0.250	101
EPMR-0111 (B)	05-0113	0.00	6.54	5.12	0.049	0.920	0.00	0.077	0.079	14.2	0.018	0.055	5.25	0.00	1.36	1.00	12.4	0.00	0.493	0.066	0.033	0.252	0.00	50.9	0.017	0.136	0.248	99.9
EPMP-0119 (A)	05-0113	0.00	6.60	5.09	0.049	0.911	0.00	0.077	0.073	15.0	0.017	0.055	5.20	0.00	1.30	1.77	12.2	0.00	0.435	0.053	0.033	0.201	0.00	50.3	0.017	0.130	0.240	101
EDMR 0110 (R)	05-0114	0.00	0.00	5.00	0.043	0.300	0.00	0.077	0.074	12.0	0.017	0.002	5.20	0.00	1.43	1.01	12.5	0.00	0.460	0.055	0.033	0.201	0.00	40.6	0.017	0.130	0.244	09.5
CD2/Erit 419 (1EE Apid)(2EWI tot)(Norm)	03-0114	0.00	6.40	5.03	0.043	0.076	0.00	0.000	0.067	14.0	0.017	0.000	5.10	0.00	1.45	1.70	12.0	0.00	0.403	0.051	0.035	0.234	0.00	40.0	0.017	0.130	0.241	30.5
3B3/FIL 418 (155 ACIU)(55WLIGI)(NOIII)			0.40	5.25	0.047	0.976		0.069	0.067	14.0	0.027	0.065	5.05		1.50	1.73	13.0	1	0.421	1	0.034	0.327		49.9		0.134	0.241	
Aniono (malka)	-	F	CI	NO2	NO2	804	804	4002	6204																			
EDMD 0090 (A)	05 0111	-100	100	100	25400	100	1540	62500	-1000																			
FPMR-0089 (A)	05-0111	<100	<100	<100	35400	<100	1540	62500	<1000																			
FPMR-0089 (B)	05-0111	<100	<100	<100	34700	<100	1540	61500	<1000																			
FPMR-0101 (A)	05-0112	<100	101	<100	33000	<100	1450	58700	<1000																			
FPMR-0101 (B)	05-0112	<100	100	<100	34000	<100	1450	59200	<1000																			
FPMR-0111 (A)	05-0113	<100	102	1/3	19900	<100	1580	42500	<1000																			
FPMR-0111 (B)	05-0113	<100	103	175	19900	<100	1600	43700	<1000																			
FPMR-0119 (A)	05-0114	<100	107	<100	20900	<100	1680	43900	<1000																			
FPMR-0119 (B)	05-0114	<100	104	<100	20600	<100	1680	43100	<1000																			
			Weight %	Solids Ca	lculations																							
		Empty	Crucible Wt +	Crucible Wt +				Insoluble	Cruc Wt+	Wt %																		
Sample		Crucible wt	Wet Sample	Dry wt	Total Solids	Wet Wt	Dry Wt	Solids	Calcined	Calcined																		
FPMR-0089 (A)	05-0111	45,4021	51.2023	48.275	49.5%	5.8002	2.873	36.1%	47.7229	40.0%																		
FPMR-0089 (B)	05-0111	44 3471	50.0895	47,1796	49.3%	5 7424	2 833	35.8%	46.6299	39.8%																		
EPMR-0101 (A)	05-0112	40 747	45 7918	43 2039	48.7%	5.0448	2 457	36.3%	42 7433	39.6%						1	1	1		1								
EPMR-0101 (B)	05-0112	42.3477	47,5402	44 8699	48.6%	5.1925	2.522	36.1%	44,3930	39.4%						1	1	1		1								
EPMR-0111 (A)	05-0113	41.6252	46,2554	43 9442	50.1%	4,6302	2.319	41.8%	43,6262	43.2%						1	1	1		1								
EPMR-0111 (B)	05-0113	42,6096	47.502	45.0531	49.9%	4.8924	2 444	41.6%	44 7166	43.1%							1	1										
EPMR-0119 (A)	05-0114	43 2249	48.0139	45 579	49.2%	4 7890	2 354	40.7%	45 2446	42.2%						1	1	1										
EPMP-0119 (B)	05-0114	42.4514	47 1427	44 7565	40.1%	4.6013	2 305	40.7%	44.4277	42.1%								-										
11 Mile-0113 (D)	03-0114	42.4514	47.1427	44.7303	43.176	4.0313	2.505	40.7 /6	44.4211	42.170																		
	-	Emplu	Coucible M/L	Crusible Mit 1	1	Soluble		ł								 	+	1		1								
Sample	-	Coucible	Mot Complex	Crocible WI +	Uncorr	Solide		1	Doneity	n H								1										<u> </u>
EPMP-0089 (A)	05-0111	42 1767	44 3370	43 4214	21.05%	13.5%		1	1.42	7 20								1										<u> </u>
EDMD 0090 (R)	05-0111	44.0100	44.33/9	43.4211	21.03%	13.3%		+	1.44	1.29						l	<u> </u>	+										
FPWR-0089 (B)	05-0111	44.0168	45.1812	44.203/	21.07%	13.5%			4.00	7.07						I	+	-										
FPWR-0101 (A)	05-0112	44.8456	45.9967	45.0703	19.52%	12.4%			1.38	1.87						I	+											
FPMR-0101 (B)	05-0112	45.4051	46.5553	45.6301	19.56%	12.5%		l								I	I	+	ļ	<u> </u>		I						I
FPMR-0111 (A)	05-0113	42.9256	44.0340	43.0829	14.19%	8.26%		I	1.34	1.12																		L
FPMR-0111 (B)	05-0113	44.2966	45.4044	44.4543	14.24%	8.31%										I	I	-										
FPMR-0119 (A)	05-0114	44.2966	45.4044	44.4543	14.24%	8.44%			1.35	7.42																		
FPMR-0119 (B)	05-0114	44.2966	45.4044	44.4543	14.24%	8.44%																						

Table C-2. SMRF SOA Feed Analytical Results (NOTE: Samples FPMR-0089/0101 and 0111/0119 are from 4% and 12% SOA feeds respectively)

APPENDIX D – CROSS-SECTIONS OF MRF SOA TEST BEAKERS



Figure D-1. Cross-Section of 0% MRF SOA Beaker



Figure D-2. Cross-Section of 8% MRF SOA Beaker



Figure D-3. Cross-Section of 12% MRF SOA Beaker

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Distribution:

J. E. Marra, 773-A E. W. Holtzscheiter, 773-A D. A. Crowley, 999-W S. L. Marra, 999-W T. B. Calloway, 999-W N. E. Bibler, 773-A C. M. Jantzen, 773-A J. R. Harbour, 773-42A C. A. Langton, 773-43A G. C. Wicks, 773-A T. H. Lorier, 999-W M. E. Smith, 773-42A D. H. Miller, 786-1A T. M. Jones, 999-W D. K. Peeler, 999-W C. C. Herman, 773-42A M. E. Stone, 999-W M. A. Baich, 999-W T. L. Fellinger, 773-A A. S. Choi, 773-42A M. S. Miller, 704-S A. B. Barnes, 704-30S J. E. Occhipinti, 704-S R. M. Hoeppel, 704-27S J. F. Iaukea, 704-30S D. C. Iverson, 704-30S R. J. O'Driscoll, 704-30S J. W. Ray, 704-S J. F. Sproull, 704-30S F. A. Washburn, 704-27S