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DWPF, glass

Retention:
Permanent

SLUDGE BATCH 5 SLURRY FED MELT RATE FURNACE TESTS WITH FRITS 418 AND 550

D. H. Miller
B. R. Pickenheim

DECEMBER 2008

Savannah River National Laboratory
Savannah River Nuclear Solutions
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under
Contract Number DE-AC09-08SR22470

DISCLAIMER

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LIST OF ACRONYMS

ACTL	Aiken County Technology Laboratory
DWPF	Defense Waste Processing Facility
MRF	Melt Rate Furnace
PSAL	Process Science Analytical Laboratory
REDOX	REDuction/OXidation
SB4	Sludge Batch 4
SB5	Sludge Batch 5
SME	Slurry Mix Evaporator
SMRF	Slurry Fed Melt Rate Furnace
SRAT	Sludge Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
WL	Waste Loading

1.0 EXECUTIVE SUMMARY

Based on Melt Rate Furnace (MRF) testing for the Sludge Batch 5 (SB5) projected composition and assessments of the potential frits with reasonable operating windows, the Savannah River National Laboratory (SRNL) recommended Slurry Fed Melt Rate Furnace (SMRF) testing with Frits 418 and 550. DWPF is currently using Frit 418 with SB5 based on SRNL's recommendation due to its ability to accommodate significant sodium variation in the sludge composition. However, experience with high boron containing frits in DWPF indicated a potential advantage for Frit 550 might exist. Therefore, SRNL performed SMRF testing to assess Frit 550's potential advantages.

The results of SMRF testing with SB5 simulant indicate that there is no appreciable difference in melt rate between Frit 418 and Frit 550 at a targeted 34 weight % waste loading. Both batches exhibited comparable behavior when delivered through the feed tube by the peristaltic pump. Limited observation of the cold cap during both runs showed no indication of major cold cap mounding.

MRF testing, performed after the SMRF runs due to time constraints, with the same two Slurry Mix Evaporator (SME) dried products led to the same conclusion. Although visual observations of the cross-sectioned MRF beakers indicated differences in the appearance of the two systems, the measured melt rates were both ~0.6 in/hr. Therefore, SRNL does not recommend a change from Frit 418 for the initial SB5 processing in DWPF.

Once the actual SB5 composition is known and revised projections of SB5 after the neptunium stream addition and any decants is provided, SRNL will perform an additional compositional window assessment with Frit 418. If requested, SRNL can also include other potential frits in this assessment should processing of SB5 with Frit 418 result in less than desirable melter throughput in DWPF. The frits would then be subjected to melt rate testing at SRNL to determine any potential advantages.

2.0 INTRODUCTION

The Defense Waste Processing Facility (DWPF) began processing of Sludge Batch 5 (SB5) in December 2008. In support of flowsheet development efforts for SB5, the Savannah River National Laboratory (SRNL) recommended Frit 418 for initial processing[1]. The Frit 418 recommendation was primarily based on its robustness to potential sludge composition variation. At that time, there was uncertainty in several factors in the Tank Farm and in DWPF processing that ultimately would dictate the nominal composition of SB5. These uncertainties included the mass of SB4 remaining in Tank 40 at the time of the Tank 51 transfer, additions of caustic to Tank 40 (SB4) due to anticipated decants, the degree of Al-dissolution in Tank 51, and the final wash end point. One of the primary oxides being tracked with these uncertainties was the Na_2O concentration given it has a significant impact on several process or product performance properties. Given these uncertainties and the need to order a frit to support initial operations (to avoid a feed outage), SRNL performed a series of paper study assessments which identified the ability of Frit 418 to provide relatively large operating windows for a large compositional range of SB5. That being the case, Frit 418 was recommended with the knowledge that DWPF may be giving up optimization (with respect to melt rate) for robustness to ensure Slurry Mix Evaporator (SME) acceptability evaluations would be successful.

After the initial recommendation, SRNL continued to evaluate alternative frits that might improve melt rate while maintaining acceptable operating windows. Based on Melt Rate Furnace (MRF) data, Miller et al.[2] identified Frit 550 as a leading candidate to improve melt rate while maintaining relatively large operating windows (Frit 550 is similar to the Frit 540 actually used in the initial MRF testing). Slurry-Fed Melt Rate Furnace (SMRF) tests with Frit 418 and Frit 550 were recommended to assess whether Frit 550 would lead to a higher melt rate relative to Frit 418. If so, DWPF could elect to transition from Frit 418 to Frit 550 after ordering, fabrication, and receipt of this alternative frit. The target compositions of the two frits are given in Table 1.

Table 1. Frit Target Compositions (wt%)

FRIT	B_2O_3	Li_2O	Na_2O	SiO_2
418	8	8	8	76
550	12	8	7	73

Miller et al.[3] provided a high level summary of the SMRF results to support frit procurement efforts for initial processing. This report provides a detailed discussion of the feed preparation, SMRF testing and feeding conditions, measured feed and pour rates, and observed cold cap behavior. The SB5 SMRF testing was conducted to gain insight into the feeding behavior of the SB5 system that cannot be obtained in the dry fed MRF.

This work is being performed under the auspices of the Technical Task Request HLW-DWPF-TTR-2007-0007[4] and supporting Task Technical & Quality Assurance Plan[5].

3.0 DISCUSSION

3.1 22-L SRAT/SME FEED PREPARATION DETAILS FOR SMRF RUNS

The SB5 sludge composition used in these melt rate tests was designated as "SB5-C". This sludge was designed to simulate the composition of Tank 40 after blending (30% Tank 51 / 70% Tank 40). The details regarding the preparation of this simulant have been previously reported.[6] As is common when making large batches for melter studies, no mercury or noble metals were added during the SRAT/SME process.

The Sludge Receipt and Adjustment Tank/Slurry Mix Evaporator (SRAT/SME) products were made in the Aiken County Technology Laboratory (ACTL) in 22 L vessels using the sludge composition referenced above. The feed preparation process strategy used 130% acid stoichiometry and targets of 45% total solids, 34 weight % waste loading (WL) and 0.2 REDOX defined as $Fe^{2+}/\Sigma Fe$. Further details on the run parameters are documented in the SRAT/SME R&D Directions.[7] A typical acid calculation is provided in Appendix A. Waste loading calculations using lithium values from the glass pour samples indicate that the Frit 550 run is >3% higher than the Frit 418 run. When additional frit and SRAT components are brought into the calculations, the difference is reduced to <1%; with the average being 35.6% and 34.8% respectively for Frit 550 and Frit 418. An estimate using the two SME products indicates WL values of 33.7% and 33.4% for frit runs 550 and 418. Both of these calculations yield results that are within normal variation associated with waste loading. With the limited number of samples and the sensitivity of calculations to the lithium analysis, it is believed that waste loading did not play a significant role in the results.

The change in frit composition is most evident in the increase in B with the SB5/Frit 550 composition. The final pH was much lower than typical flowsheet runs because no noble metals are used in the feed preparation. In the absence of noble metals, less formate is destroyed so the product remains more acidic. Neither pH nor offgas data was collected during these runs as is the current practice for melter feed preparation.

Table 2 gives the measured composition and physical properties of the SME Products. Two 22 L runs were performed and blended to make each SMRF feed listed in the table.

Table 2. SB5 SME Product Data

Element (wt% calcined solids)	SB5/ Frit 418	SB5/ Frit 550
Al	4.33	4.30
B	1.59	2.45
Ba	0.011	< 0.010
Ca	0.561	0.557
Cr	0.016	0.017
Cu	< 0.010	< 0.010
Fe	7.57	7.68
K	0.089	0.066
Li	2.30	2.55
Mg	0.365	0.357
Mn	1.75	1.82
Na	9.62	9.61
Ni	0.867	0.89
P	< 0.100	< 0.100
Pb	< 0.010	< 0.010
S	0.059	0.056
Si	23.9	23.6
Sr	< 0.010	< 0.010
Ti	0.045	< 0.010
Zn	0.012	< 0.010
Zr	0.098	0.011

Anion (mg/kg)	SB5/ Frit 418	SB5/ Frit 550
F	<100	<100
Cl	<100	<100
NO_2	<100	<100
NO_3	48000	48800
PO_4	<100	<100
C_2O_4	<100	<100
HCO_2	104000	106000
Solids (wt%)		
Total	45.2	45.2
Insoluble	34.9	34.6
Soluble	10.3	10.6
Calcined	36.6	36.7
Final pH	4.71	4.71
Density (g/ml)	1.34	1.34

.

3.2 SB5 SMRF RUN

Details of the SMRF configuration are documented in previous reports[8]. The operational parameters for the SMRF tests were consistent with previous testing and are outlined in the test plan[9]. The melt pool and vapor space set points were 1125°C and 750°C, respectively. The vapor space temperature controller was clamped at 87% output as in previous runs. Prior to starting the test, a sample of both SME products was run through the feed system. Both yielded acceptable flow rates based on previous pump parameters. A check of the solids content verified that both batches hit the target of 45 wt% total solids.

The SMRF was charged with 6 Kg of glass (drained from the SMRF after the July 07 SB4-Frit 418/510 run). The SMRF was heated to set point on 10/27/08. Melt pool depth was measured at 3" before the addition of 1Kg startup glass.

Feeding of SB5/Frit 418 SME product began on 10/28/08 at approximately 0700 hours. The agitator speed was 180 rpm and the feed pump motor was operated at 261 rpm. This setting yielded a feed cycle of 110-115 grams per 20 seconds. Feeding and pouring continued until

~1530 hours with only a few interruptions for feed tube plugging. These were short in duration and were corrected by reversing the material flow direction in the feed supply line for several seconds, until slurry was forced back into the feed tank. The cold cap was observed on several occasions for evidence of overfeeding, such as excessive mounding or a completely dark surface. In general, the area under the feed tube was dark, but there were areas around the edge of the cold cap that were thin and an orange glow could be observed. The cold cap level was consistently located below the over flow cap located on the pour tube. Based on round sheet data being taken at the time, the amount of glass being poured matched well with the expected quantity based on the feed rate and measured calcine solids (36.7 wt%).

Feeding of SB5/Frit 550 SME product began on 10/29/08 at approximately 0700 hours using the same agitator and pump settings. These settings yielded a feed cycle of approximately 110 grams per 20 seconds. Feeding and pouring continued until ~1530 hours with only a few stoppages for feed tube plugging. These were similar in duration to the Frit 418 run and were quickly corrected. The cold cap was observed on several occasions and no indication of overfeeding was observed. During the test run the cold cap appeared to be ~1" higher than the Frit 418 run, but never reached the top of the pour tube cap. This could be an indication of a slightly higher foaming tendency with Frit 550, but did not cause any problems in the processing. A similar pattern was observed in subsequent MRF testing and will be discussed in Section 3.7. As with the Frit 418 batch, the amount of glass being poured matched well with the expected quantity.

3.3 ANALYTICAL RESULTS

Several pour stream glass samples were taken throughout the two SMRF runs. Table 3 shows the sample ID and description of the samples that were submitted to the Process Science Analytical Laboratory (PSAL) for analysis. The results of the analyses are shown in Appendix B. As expected, the boron content steadily increased throughout the run as the Frit 550 material displaced the Frit 418 glass in the melter. The silica content also decreased slightly throughout the run, which is consistent with the lower SiO_2 content in Frit 550. Samples chosen to represent the two end products yielded results that indicated both runs had similar waste loadings as discussed in section 3.1.

Table 3. SMRF Glass Sample Identification

Sample ID	Lab ID	Date	Time	Description	Test ID
SMRF 0234	08-2290	10/28	15:35	After 7200 grams of glass poured	SB5/Frit 418
SMRF 0236	08-2291	10/29	9:45	After 1830 grams of glass poured	SB5/Frit 550
SMRF 0237	08-2292	10/29	15:45	After 6600 grams of glass poured	SB5/Frit 550
SMRF 0238	08-2293	10/30	8:20	End of drain	

3.4 REDOX

Previous studies have shown that melt rate results can be affected by the REDOX conditions during feed preparation. Completely oxidized feeds can mask the results obtained in melt rate testing. A sample of both SME batches was converted to glass using the closed crucible procedure to assess the final REDOX of the glass. The glass was then submitted to the PSAL for REDOX ($Fe^{2+}/\Sigma Fe$) determination. Table 4 summarizes the REDOX values of the glass samples and the complete data set is found in Appendix C. As seen from the data, the two SME batches showed similar REDOX results, and both were near the normal range (target values are generally from 0.15 to 0.20). Based on these results, there is no reason to suspect that REDOX conditions were a major influence on the melt rate findings.

Table 4. SMRF REDOX Results

Sample ID	Frit	*REDOX (Fe2+/∑Fe)
Blend 19/20	418	0.143
Blend 21/22	550	0.146

* Average of two measurements from each of three samples

3.5 POWER CONSUMPTION

Power consumption for the plenum and melt pool heaters were monitored for both runs. The amps are recorded every 30 minutes to verify that the furnace elements were properly functioning during the testing. Table 5 gives the total power used for the two SB5 SMRF tests as well as the power consumed in a previous SB4/Frit 418 (45% total solids) SMRF test[10].

Table 5. Power (BTU/Min) Consumptions for the SB5 SMRF Runs

Power Zone	SB5/Frit 418	SB5/Frit 550	SB4/Frit 418
Plenum	96.7	95.3	94.2
Melt Pool	46.1	54.6	44.4

Both SMRF runs had plenum power requirements that were similar and in line with previous testing. The vapor space temperature is the controlling variable in SMRF testing since it is used to initiate the feed cycle. The vapor space thermocouple is located ~ 5” below the top of the melter along with an over-temperature thermocouple. In the past, major differences in power consumption were assumed to be related to the influence of different cold cap properties and the presence of "shine" from the melt pool. It is possible that the slightly higher foam layer or the small difference in frit composition contributed to the increased melt pool power requirement for Frit 550, but the difference was not visually obvious. Melt pool temperatures remained constant at 1125 °C during both tests.

3.6 MELT RATE

Table 6 shows the melt rate calculated from feed rate, and actual melt rates over a six hour period beginning at 0900 for each frit. This time was chosen to allow the system to stabilize for ~ 2 hours after initiation of feeding. The calculated melt rate is determined by multiplying the measured feed rate by the calcine factor. The melt rate for Frit 550 was slightly lower than that for Frit 418, but not by a significant amount. The values are slightly different than those reported in the previous high level memo [3] because a different time period was used after viewing the data graphs to ensure steady state conditions.

Table 6. SMRF Melt Rates

Frit	Average Feed Rate (g/min)	Calculated Melt Rate (g/min)	Actual Melt Rate (g/min)
418	38.1	13.9	13.7
550	36.2	13.3	13.3

3.7 MRF RESULTS

Due to time constraints and frit availability, Frit 550 was not run in the MRF prior to the two SMRF runs covered in this report. A MRF run using SME product from the SMRF testing was completed on 11/31/08, along with a Frit 418 standard. The melt rate results from the MRF are shown in Table 7 below.

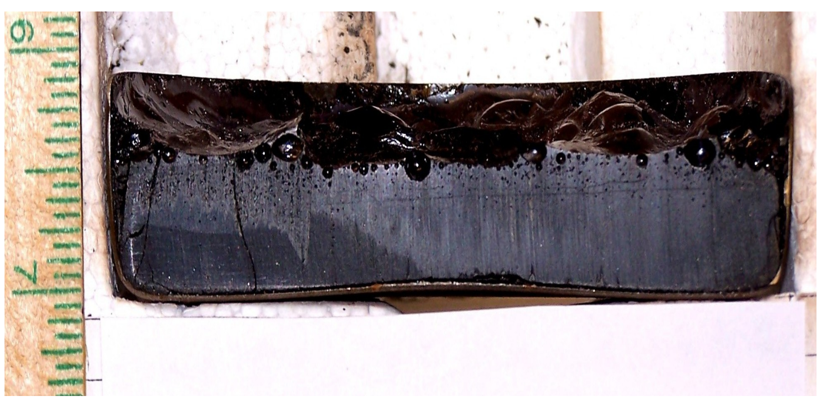
Table 7. MRF Melt Rates

Sample ID	Frit	Target Waste Loading (%)	SME Product ID	Melt Rate (In/Min)
MRF 08-077	418	34	08-SB5-19/20	0.62
MRF 08-078	550	34	08-SB5-21/22	0.58
Frit Std 1st layer	418	N/A	N/A	1.71
Frit Std 2nd layer	418	N/A	N/A	3.21

As found with the SMRF testing, the MRF results indicate little difference in melt rate between the two frits, with Frit 418 being slightly faster than Frit 550. Figures 1 and 2 show a cross section of both MRF test beakers. Although the measured melt rates (based on the amount of glass produced) were similar, there is a significant difference in the visual observations of the cross-sections. The upper crown visible in the Frit 418 beaker (Figure 1) was several inches higher in the Frit 550 beaker and did not contain as many large bubbles. The crown portion of the Frit 550 run remained in the upper portion of the beaker that was cut away prior to sectioning. The amount of glass left in the bottom of both beakers was very similar, which accounts for the reported melt rates being similar.

Figure 1. SB5 – Frit 418

Figure 2. SB5 – Frit 550



Previous SB5 MRF tests suggested that increasing the boron concentration in the frit had positive impacts on melt rate for the SB5 system. That being said, the SRMF and MRF results are somewhat surprising given the B_2O_3 concentrations of Frit 550 and Frit 418 of 12 wt % and 8 wt %, respectively. However, Frit 550 does have 1 wt % less Na_2O than Frit 418, which may have offset the potential or assumed advantage of the higher B_2O_3 concentration.

4.0 SUMMARY AND PATH FORWARD

The results of SMRF testing with SB5 indicate that there is no appreciable difference in melt rate between Frit 418 and Frit 550. The average pour rates for the Frit 418 and Frit 550 systems were 13.7 and 13.3 grams of glass per minute, respectively. Both batches exhibited comparable behavior in the feed system and processed smoothly without a large number of interruptions. Limited observation of the cold cap during both runs showed similar patterns with no indication of major cold cap mounding. Subsequent MRF testing with the same two SME products led to the same conclusion. Although there were differences in the appearance of the MRF beakers, the measured melt rates were both ~0.6 in/hr.

Coupling the melt rate information with recent Measurement Acceptability Region (MAR) assessments for SB5 pre- and post-nitrite and Np additions[11] SRNL recommends that DWPF utilize Frit 418 to process SB5 without transitioning to Frit 550. Given the potential differences in waste loading indicated by lithium analyses, additional MRF testing could be conducted to better define the impact of waste loading on melt rate in the current SB5/Frit 550 system. Once an actual SB5 composition is obtained from the Tank 40 3 L sample, revised projections of SB5 after the neptunium stream addition and any decants should be assessed. As part of the neptunium qualification efforts, SRNL will perform an additional MAR assessment with Frit 418. If requested, SRNL could also include other potential frits in this assessment should processing of SB5 with Frit 418 result in less than desirable melter throughput in DWPF. The frits could then be subjected to melt rate testing at SRNL to determine any potential advantages.

5.0 REFERENCE

[1] K. M. Fox, **Recommended Frit Composition for Initial Sludge Batch 5 Processing at the Defense Waste Processing Facility, WSRC-STI-2008-00338,** June 2008, Savannah River National Laboratory, Aiken, South Carolina.

[2] D. H. Miller, K. M. Fox, B. R. Pickenheim, M. E. Stone, **Melt Rate Furnace Testing for Sludge Batch 5 Frit Selection**, SRNS-STI-2008-00092, September 2008, Savannah River National Laboratory, Aiken, South Carolina.

[3] D. H. Miller, T. M. Jones, B. R. Pickenheim, **Sludge Batch 5 Slurry-Fed Melt Rate Furnace (SMRF) Results for Frit 418 and Frit 550,** SRNL-L3100-2008-00102, November 2008, Savannah River National Laboratory, Aiken, South Carolina.

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[6] D. P. Lambert, M. E. Stone, B. R. Pickenheim, D. R. Best, D. C. Koopman, **Sludge Batch 5 Simulant Flowsheet Studies**, SRNS-STI-2008-00024, October 2008, Savannah River National Laboratory, Aiken, South Carolina.

[7] B. R. Pickenheim, **22-L SRAT Runs for Melt Rate Testing: SB5-19 – SB5-22**, SRNL-L3100-2008-00075, October 2008, Savannah River National Laboratory, Aiken, South Carolina.

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[10] M.E. Smith, T. M. Jones, D. H. Miller, **Sludge Batch 4 Baseline Melt Rate Furnace and Slurry-Fed Melt Rate Furnace Tests with Frits 418 and 510**, WSRC STI-2007-00450, Savannah River National Laboratory, Aiken, South Carolina.

[11] K. M. Fox, T. B. Edwards, D. K. Peeler, **The Impact of Np and Sodium Nitrite Additions on the Projected Operating Windows for Sludge Batch 5,** SRNL-L3100-2008-00103, Savannah River National Laboratory, Aiken, South Carolina.

APPENDIX A. Acid Calculation

SRNL SRAT Acid, Trim Chemical, Dewater and Redox Calc Revised:	*6/1/2007*	
Run Description:		
Run #	**SB5-19**	
Sludge Feed Batch #	**SB5-C carboy 1**	
SRAT Vessel Volume, L	**22**	
Calculation Notes:		
1. To calculate an acid mix to achieve a REDOX target, click on macro.	RedOx Target Macro	**Redox at target**
2. To Calculate the oxalate in the trimmed sludge, click on macro.	Oxalate Target	**Trimmed Sludge Oxalate at target**
Warnings		
No Mercury Warning, No Coal Warning, No Oxalate Warning		
Table 1 -- Sludge Analyses for Acid Calculations, Batch #	**SB5-C carboy 1**	
Fresh Sludge Mass without trim chemicals	16,758.9	g slurry
Fresh Sludge Weight % Total Solids	12.50	wt%
Fresh Sludge Weight % Calcined Solids	9.51	wt%
Fresh Sludge Weight % Insoluble Solids	7.85	wt%
Fresh Sludge Density	1.090	kg / L slurry
Fresh Sludge Nitrite	6,175	mg/kg slurry
Fresh Sludge Nitrate	3,940	mg/kg slurry
Fresh Sludge Oxalate	0	mg/kg slurry
Fresh Sludge Formate	0	mg/kg slurry
Fresh Sludge Coal/Carbon source	0.000	wt% dry basis
Fresh Sludge Manganese (% of Calcined Solids)	5.050	wt % calcined basis
Fresh Sludge Slurry TIC (treated as Carbonate)	1,338	mg/kg slurry
Fresh Sludge Hydroxide (Base Equivalents) pH = 7	0.632	Equiv Moles Base/L slurry
Fresh Sludge Mercury (% of Total Solids in untrimmed sludge)	0.0000	wt% dry basis
Fresh Sludge Supernate manganese	0	mg/L supernate
Fresh Sludge Supernate density	1.024	kg / L supernate
Table 2 -- SRAT Processing Assumptions, Run #	**SB5-19**	
Conversion of Nitrite to Nitrate in SRAT Cycle	20.00	gmol NO_3^-/100 gmol NO_2^-
Destruction of Nitrite in SRAT and SME cycle	100.00	% of starting nitrite destroyed
Destruction of Formic acid charged in SRAT	15.00	% formate converted to CO_2 etc.
Destruction of oxalate charged	50.00	% of total oxalate destroyed
Percent Acid in Excess Stoichiometric Ratio	130.00	%
SRAT Product Target Solids	25.00	%
Nitric Acid Molarity	10.340	Molar
Formic Acid Molarity	23.600	Molar
DWPF Nitric Acid addition Rate	2.0	gallons per minute
DWPF Formic Acid addition Rate	2.0	gallons per minute
REDOX Target	0.200	$Fe^{+2} / \Sigma Fe$
REDOX Equation (7 for Mn^{+7}, otherwise assumes Mn^{+4})	7	Enter 7 for newest redox equation
Trimmed Sludge Target Ag metal content	0.00000	total wt% dry basis after trim
Trimmed Sludge Target wt% Hg dry basis	0.00000	total wt% dry basis after trim
Trimmed Sludge Target Pd metal content	0.00000	total wt% dry basis after trim
Trimmed Sludge Target Rh metal content	0.00000	total wt% dry basis after trim
Trimmed Sludge Target Ru metal content	0.00000	total wt% dry basis after trim
Trimmed Sludge Target Wt% Coal/carbon source dry basis	0.00	total wt% dry basis after trim
Trimmed Sludge Target oxalate after trim (wt % not mg/kg)	0.000	total wt% dry basis after trim
Water to dilute fresh sludge and/or rinse trim chemicals	0.000	g
Total Water added to flush both the Nitric and Formic Acid Lines	50.0	g
Sample Mass of Trimmed sludge (SRAT Receipt sample, if any)	0.0	g
Mass of SRAT cycle samples	0.000	g
Wt% Active Agent In Antifoam Solution	10	%
Basis Antifoam Addition for SRAT (generally 100 mg antifoam/kg slurry)	100.00	mg/kg slurry
Number of basis antifoam additions added during SRAT cycle	7.00	
Table 3 -- SME Processing Assumptions, Run #	**SB5-19**	
Enter 1 for Redox Balance with SME Cycle or 0 for Redox Balance with no SME Cycle	1.00	
Frit type	418.00	
Destruction of Formic acid in SME	5.00	% Formate converted to CO_2 etc.
Destruction of Nitrate in SME	0.00	% Nitrate destroyed in SME
Assumed SME density	1.39	kg / L
Basis Antifoam Addition for SME cycle	100.00	mg/kg slurry
Number of basis antifoam additions added during SME cycle	1.000	
Sludge Oxide Contribution in SME (Waste Loading)	34.000	%
Frit Slurry Formic Acid Ratio	0.0	g 90 wt% FA/100 g Frit
Target SME Solids total Wt%	45.0	wt%
Number of frit additions in SME Cycle	2.000	
# DWPF Canister decons simulated	0.0	

Calculations Below:		
Acid and Glass Calculation Base Values		
Fresh Sludge nitrite	2.249	gmol
Fresh Sludge Mn minus soluble Mn	1.465	gmol
Fresh Sludge carbonate	1.867	gmol
Fresh Sludge hydroxide	9.717	gmol
Fresh Sludge mercury	0.000	gmol
Fresh Sludge oxalate	0.000	gmol
Fresh Sludge grams of calcined oxides	1593.771	g
Trim Chemicals Calculations	0.0000	
Fresh Sludge Calcine Factor (1100°C), g oxide/g dry solids (calculated)	0.7608	g/g
Total solids before trim addition	2,094.8625	g
Total solids before trim less HgO, NaOxalate, coal)	2,094.86	g
Predicted total solids at target levels	2,094.8625	g
Predicted total mass at target levels	16,758.9000	g
Target Ag metal content in trimmed sludge	0.000000	total wt% dry basis
$AgNO_3$ to add (CF=0.682)	0.00000	g
Ag2O calcined solids	0.00000	g
Water added with Ag	0.00000	g
Target wt% Hg dry basis	0.000	total wt% dry basis
Total HgO in fresh Sludge	0.000	g
Total HgO in trimmed Sludge	0.000	g
HgO to add	0.000	g
HgO calcined solids	0.00000	g
Water added with Hg	0.00000	g
Calculated total wt% Hg dry basis	0.0000	wt% dry basis
Target Pd metal content in trimmed sludge	0.0000	total wt% dry basis
Wt % Pd in reagent solution	15.2700	wt% in solution
$Pd(NO_3)_2*H_2O$ solution to add (CF=1.150 g metal oxide/g metal)	0.000	g of solution
Pd(NO3)2 to add	0.00000	g
PdO calcined solids	0.00000	g
Water added with Pd	0.000	g
Target Rh metal content in trimmed sludge	0.0000	total wt% dry basis
Wt% Rh in reagent solution	4.93	wt% in solution
$Rh(NO_3)_3*2H_2O$ (CF=1.311g metal oxide/g metal)	0.000	g of solution
Rh(NO3)3 to add	0.00000	g
Rh2O3 calcined solids	0.00000	g
Water added with Rh	0.000	g
Target Ru metal content in trimmed sludge	0.0000	total wt% dry basis
Wt% Ru in $RuCl_3$ reagent solids	41.74	wt% in solids
$RuCl_3$ to add (CF=1.0)	0.000	g solid
Target wt% Coal/carbon source in trimmed sludge, dry basis	0.00	total wt% dry basis
Total Coal in fresh Sludge	0.000	g
Total Coal in trimmed Sludge	0.000	g
Mass of Coal to add (CF =.08)	0.00	g
Calculated wt% coal after trim additions	0.00	wt%
Oxides added with coal		
Target sodium oxalate in trimmed sludge per gm total solids	0.00	total wt% dry basis
Total Sodium Oxalate in fresh Sludge	0.000	g
Total Sodium Oxalate in trimmed Sludge	0.000	g
Sodium oxalate to add (CF=0.463)	0.0000000	g
Calculated oxalate conc. after trim chemical additions	0.00	total wt% dry basis
Na2O calcined solids from sodium oxalate	0.00000	
Total mass of trim chemicals added	0.0	g
Calcined oxides added in trim chemicals	0.00	g
Total solids after trim addition	2,094.86	g
Match of actual to predicted total solids mass	100.00%	
Total Calcine solids after trim	1,593.77	g
Water added to dilute and/or rinse trim chemicals	0.0	g
Mass of trimmed sludge	16,758.90	g
Calculated wt% total solids in trimmed sludge	12.5	wt%
Sample mass of trimmed sludge	0.00	g
Mass of trimmed sludge reacted	16,758.90	g
Sample removal ratio at start of SRAT	1.000	
Calcined solids at start of SRAT	1,593.8	g

STOICHIOMETRIC ACID CALCULATION		
Stoichiometric Acid Ratios Used		
Acid requirement per mole of Nitrite	0.75	mole H+/ mole NO2-
Acid requirement per mole of Mn	1.20	mole H+/ mole Mn
Acid requirement per mole of Carbonate	2.00	mole H+/ mole CO3=
Acid requirement per mole of Hydroxide	1.00	mole H+/ mole OH-
Acid requirement per mole of Hg	1.00	mole H+/ mole Hg++
Acid requirement per mole of Oxalate	0.00	mole H+/ mole C2O4=
Fresh feed NO_2^-	1.6871	gmol
Fresh feed Mn	1.75803	gmol
Fresh feed Carbonate	3.7339	gmol
Fresh feed OH^-	9.7171	gmol
Hg from trim	0.000000	gmol
Hg from fresh sludge	0.00000	gmol
Total Stoichiometric Acid required	16.8961	gmol
Percent Acid in Excess Stoichiometric Ratio	130.000	%
Actual acid to add to SRAT	21.9649	gmol
Acid required in moles per liter of starting sludge (less receipt samples)	1.4286	gmol/L
REDOX CALCULATION (SME PRODUCT REDOX PREDICTION)		
REDOX Target	0.200	Fe+2 / Fe
Predicted REDOX	0.200	
Ratio of formic acid to total acid	0.8425	moles formic acid / mole total acid
Delta between predicted REDOX and target REDOX	-0.000002	
Activation of SME cycle corrections? (1=SME corrections performed):	1	
Nitric acid density, 20 °C	1.308	g/mL
Formic acid density, 20 °C	1.2047	g/mL
Nitric acid, wt %	49.80	wt %
Formic acid, wt %	90.16	wt %
Formic acid amount	18.506	gmol
Nitric acid amount	3.459	gmol
Total Manganese in fresh feed	1.465	gmol
Manganese removed with SRAT product sample	0.000	gmol
Projected Melter Feed Manganese, total moles	1.465	gmol
Formate moles with fresh sludge	0.000	gmol
Formate moles added with formic acid	18.506	gmol
Formate moles destroyed in SRAT (% of acid charged)	2.776	gmol
Formate moles removed with SRAT product sample	0.000	gmol
Formate moles reacted in SME (% of acid charged)	0.787	gmol
Formate Moles after SME	14.944	gmol
Frit slurry formate (when SME cycle frit additions are made with formic acid)	0.000	gmol
Projected Melter Feed Formate, total moles	14.944	gmol
Nitrate moles from fresh sludge	1.065	gmol
Nitrate moles from nitric acid	3.459	gmol
Nitrate from conversion of nitrite to nitrate in SRAT and SME	0.450	gmol
Nitrate from minor trim chemicals	0.00000	gmol
Nitrate removed with SRAT product sample	0.00000	gmol
Nitrate destroyed in the SME	0.00000	gmol
Projected Melter Feed Nitrate, total moles (Sum of inputs - destroyed)	4.974	gmol
Oxalate in fresh feed	0.000	gmol
Oxalate from trim	0.000	gmol
Oxalate destroyed during reaction	0.000	gmol
Oxalate removed with SRAT product sample	0.000	gmol
Projected Melter Feed Oxalate, total moles	0.000	gmol
Carbon from Coal in fresh feed	0.000	gmol
Carbon from trim coal	0.000	gmol
Carbon removed in SRAT product Sample	0.000	gmol
Projected Melter Feed Carbon from coal, total moles	0.000	gmol
Projected Melter Feed Nitrite, total moles	0.0000	gmol
Assumed SME density	1.390	g/ml
Projected final SME mass	12.876	kg
Manganese concentration in final melter feed	0.114	gmol/kg melter feed slurry
Formate concentration in final melter feed	1.161	gmol/kg melter feed slurry
Oxalate concentration in final melter feed	0.000	gmol/kg melter feed slurry
Carbon from coal concentration in final melter feed	0.000	gmol/kg melter feed slurry
Nitrate concentration in final melter feed	0.386	gmol/kg melter feed slurry
Nitrite concentration in final melter feed	0.000	gmol/kg melter feed slurry

BENCH SCALE CALCULATIONS		
Bench Scale Operational Setting		
Scaled formic acid feed rate based on nominal 23.551 M	5.1139	ml/min
Scaled nitric acid feed rate based on nominal 10.395 M	5.1517	ml/min
Prototypical formic acid feed time	153.3	min
Prototypical nitric acid feed time	64.9	min
Formic acid volume required	784.156	ml
Nitric acid volume required	334.511	ml
Wt% active agent in antifoam solution	10	%
Target concentration for overall SRAT cycle	700	ppm
Total SRAT antifoam charge for 1:10 dilution	117.31	g
100 ppm SRAT antifoam charge at 1:10	16.76	g
Dewatering Calc for Target Wt. % Total Solids in SRAT Product		
Final SRAT Product Total Solids (UNDER TOOLS USE SOLVER)	25.00	%
Water in Trimmed (and sampled) Sludge	14,664.04	g
Water added with antifoam	222.89	g
Water added with formic acid	92.93	g
Water added with nitric acid	219.73	g
Water added in acid flushing	50.00	g
Water made during base equiv neutralization	175.06	g
Water made in TIC destruction	33.63	g
Water made in SRAT nitrite destruction	13.51	g
Water made in Mercury Reduction	0.00	
Revised water mass in slurry	15,471.79	g
Solids in Trimmed (and sampled) Sludge	2,094.86	g
Mass 1:20 antifoam added	11.73	g
Mass of pure formic acid (HCOOH) added	851.75	g
Mass of pure nitric acid (HNO3) added	217.95	g
Solids lost during base equiv neutralization	175.06	g
Solids lost in TIC destruction	115.80	g
Solids lost in SRAT nitrite destruction	58.51	g
Solids lost in SRAT nitrite destruction	76.50	g
Solids lost in SRAT formate destruction (formic acid)	127.76	g
Solids lost in Mercury Stripping	0.00	
Revised solids mass in slurry	2,699.17	g
Target final water mass in slurry to hit total solids target	8,097.52	g
Total water to remove	7,374.27	g
Calculated total water to remove to return to starting volume	1,613.76	g
net (used in Macro iteration)	-5760.52	g
Mass of carbonate lost as CO_2	82.16	g
Mass of nitrite lost as NO	36.00	g
Formate converted to CO_2	127.76	g
Formate converted to CO2 in SRAT	127.76	g
Final sludge mass in SRAT after acid addition and dewater (neglecting samples)	10796.69	g
Mass of SRAT cycle samples (excluding SRAT Receipt)	0.00	g
Mass of treated sludge going into SME cycle	10796.69	g
SME sample ratio	1.0000	
Calcined Solids going to SME	1593.77	g

DWPF SCALE TO BENCH SCALE		
DWPF Scale SRAT cycle		
density estimate =	1.090	
Volume based scale factor 6000 gal starting SRAT	1477.2	
SRAT air purge	230	scfm
SRAT boil-up rate	5000	lbs/hr
SRAT total boil-up (reflux)	60,000	lbs
Indicated SRAT refluxing time	720	min
Bench Scale SRAT cycle		
99.5% of scaled air purge	4386.8	sccm
Helium purge rate at 0.5 vol%	21.9	sccm
Scaled boil-up rate	25.59	g/min
Required dewatering time at above rate	288.2	min
DWPF Scale SME cycle		
Water flush volume after frit slurry addition	0.0	gal
SME air purge	74.0	scfm
SME boil-up rate	5000	lbs/hr
Bench Scale SME cycle		
SME scale factor (ADJUSTED FOR SRAT SAMPLES)	1477.2	
99.5% scaled SME air purge	1411.4	sccm
Helium purge rate at 0.5 vol%	7.06	sccm
Solids remaining at start of SME	2699.2	g
SRAT product Calcine Factor (calculated)	0.590	g oxide/g dry SRAT Product
Sludge calcined solids - based on SRAT product	1593.77	g
Sludge oxide contribution in SME	34.00	%
Frit oxide contribution	66.00	%
Frit slurry wt % solids	50.00	wt%
Frit slurry formic acid ratio	0.00	g 90 wt% FA/100 g Frit
Added water simulating decontamination of canisters	0.0	g
SME cycle antifoam addition at 1:10	10.80	g
Frit solids (total)	3093.8	g
90 wt% formic acid (corrections necessary for other concentrations)	0.00	g
Water in frit slurry	3093.8	g
Scaled transfer water	0.00	g
Total frit slurry water	3093.8	g
Total mass of frit slurry	6187.6	g
Number of equal SME frit slurry additions	2	
Each SME frit addition	1546.9	g
Each SME 90-wt% formic acid addition	0.00	g
Each SME water addition	1546.9	g
Scaled SME boil-up rate	25.59	g/min
Approximate time to remove water:	60.5	min
Final solids content in SME	5794.0	g
Target SME solids total wt%	45.0	%
Mass of water to boil off for final SME concentration	1036.4	g
Scaled boil-up rate	25.59	g/min
Approximate time to reach solids target concentration.	40.5	min

APPENDIX B. SMRF Glass Analysis (wt% Oxide)

Sample ID	Al_2O_3	B_2O_3	BaO	CaO	CeO_2	Cr_2O_3	CuO	Fe_2O_3	K_2O	Li_2O	MgO
SMRF 0234 (A)	9.56	5.31	0.025	0.937	0.047	0.050	0.030	11.3	0.184	4.75	0.911
SMRF 0234 (B)	9.60	5.28	0.025	0.939	0.047	0.053	0.023	11.3	0.185	4.71	0.908
SMRF 0236 (A)	8.83	5.02	0.015	0.875	0.02	0.039	0.03	11.1	0.125	4.69	0.739
SMRF 0236 (B)	8.94	4.96	0.015	0.889	0.02	0.034	0.03	11.1	0.120	4.71	0.754
SMRF 0237(A)	8.49	6.70	<0.011	0.869	0.012	0.047	0.028	10.9	0.097	5.05	0.702
SMRF 0237 (B)	8.51	6.44	<0.011	0.858	0.012	0.047	0.026	10.9	0.103	4.92	0.689
SMRF 0238 (A)	8.16	7.28	<0.011	0.812	<.012	0.140	0.020	11.0	0.094	5.10	0.634
SMRF 0238 (B)	8.22	7.34	<0.011	0.825	<.012	0.145	0.023	11.1	0.092	5.14	0.637
Sample ID	**MnO_2**	**Na_2O**	**NiO**	**P_2O_5**	**PbO**	**SO_4**	**SiO_2**	**TiO_2**	**ZnO**	**ZrO_2**	**Total**
SMRF 0234 (A)	2.88	12.8	0.841	<.229	<.011	0.363	50.5	0.097	0.030	0.128	101
SMRF 0234 (B)	2.88	12.8	0.847	<.229	<.011	0.360	50.7	0.100	0.025	0.132	101
SMRF 0236 (A)	2.83	13.1	1.02	<.229	<.011	0.258	50.5	0.085	0.024	0.126	99.4
SMRF 0236 (B)	2.97	13.2	1.05	<.229	<.011	0.258	50.7	0.089	0.021	0.128	100
SMRF 0237(A)	2.97	13.5	1.14	<.229	<.011	0.303	50.9	0.055	0	0.069	102
SMRF 0237 (B)	2.99	13.5	1.13	<.229	<.011	0.300	51.1	0.053	0	0.070	102
SMRF 0238 (A)	2.88	13.2	1.31	<.229	<.011	0.210	49.2	0.035	0	0.027	100
SMRF 0238 (B)	2.91	13.2	1.36	<.229	<.011	0.210	49.4	0.025	0	0.024	101

APPENDIX C. SMRF REDOX Results

					Fe(2+)	Fe(2+)
Sample	**Lab ID**	**Fe(2+)**	**Fe(3+)**	**Fe(total)**	**Fe(3+)**	**Fe(total)**
EA		0.050	0.187	0.237	0.267	0.211
19/20 BLEND-1 (A)	**08-2297**	0.025	0.150	0.175	0.167	0.143
19/20 BLEND-1 (B)	**08-2297**	0.025	0.151	0.176	0.166	0.142
19/20 BLEND-2 (A)	**08-2298**	0.034	0.208	0.242	0.163	0.140
19/20 BLEND-2 (B)	**08-2298**	0.034	0.209	0.243	0.163	0.140
19/20 BLEND-3 (A)	**08-2299**	0.030	0.175	0.205	0.171	0.146
19/20 BLEND-3 (B)	**08-2299**	0.030	0.175	0.205	0.171	0.146
21/22 BLEND-1 (A)	**08-2300**	0.023	0.127	0.150	0.181	0.153
21/22 BLEND-1 (B)	**08-2300**	0.024	0.126	0.150	0.190	0.160
21/22 BLEND-2 (A)	**08-2301**	0.032	0.181	0.213	0.177	0.150
21/22 BLEND-2 (B)	**08-2301**	0.031	0.182	0.213	0.170	0.146
21/22 BLEND-3 (A)	**08-2302**	0.029	0.192	0.221	0.151	0.131
21/22 BLEND-3 (B)	**08-2302**	0.030	0.193	0.223	0.155	0.135

Distribution:

J. C. Griffin, 773-A
S. L. Marra, 773-A
A. B. Barnes, 999-W
D. A. Crowley, 773-43A
S. D. Fink, 773-A
C. W. Gardner, 773-A
B. J. Giddings, 786-5A
C. C. Herman, 999-W
F. M. Pennebaker, 773-42A
J. E. Occhipinti, 704-S
D. C. Sherburne, 704-S
R. T. McNew, 704-27S
J. F. Iaukea, 704-30S
J. W. Ray, 704-S
H. B. Shah, 766-H
J. M. Gillam, 766-H
B. A. Hamm, 766-H
D. D. Larsen, 766-H
C. J. Bannochie, 773-42A
A. Y. Billings, 999-W
A. S. Choi, 773-42A
K. M. Fox, 999-W
D. J. McCabe, 773-42A
D. H. Miller, 999-W
J. D. Newell, 999-W
D. K. Peeler, 999-W
B. R. Pickenheim, 999-W
F. C. Raszewski, 999-W
M. E. Stone, 999-W
J. P. Vaughan, 773-41A
P. L. Bovan, 704-27S
M. A. Broome, 704-29S
A. J. Cross, 704-71S
J. M. Bricker, 704-27S
T. L. Fellinger, 704-26S
E. W. Holtzscheiter, 704-15S
M. T. Keefer, 766-H
M. E. Smith, 704-30S