WSRC-TR-2001-00126

MELT RATE IMPROVEMENT FOR MB3:
Feed Preparation

M. E. Stone
D. P. Lambert

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT NO. DE-AC09-96SR18500

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-96SR18500 with the U.S. Department of Energy.

DISCLAIMER

WSRC-TR-2001-00126

Keywords: DWPF
Vitrification,
Melt Rate
Feed
Preparation

Retention: **Permanent**

MELT RATE IMPROVEMENT FOR MB3:
Feed Preparation

M. E. Stone
D. P. Lambert

Publication Date: April 1, 2001

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

SAFETY • RESPONSIBILITY • SECURITY
SRS
SAVANNAH RIVER SITE

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT NO. DE-AC09-96SR18500

APPROVALS

M. E. Stone, Author Immobilization Technology Section	Date
D. P. Lambert, Author Immobilization Technology Section	Date
D. C. Witt, Technical Reviewer Immobilization Technology Section	Date
S. L. Marra, Manager Glass Form & Process Development Group - DWPF	Date
E. W. Holtzscheiter, Manager Immobilization Technology Section	Date

TABLE OF CONTENTS

1.0 INTRODUCTION

The Defense Waste Processing Facility (DWPF) would like to increase its canister production rate. A study[1] was completed in FY00 to look for processing and frit changes that would improve the melting rate of the glass in the DWPF melter specifically for Macrobatch 2 (Sludge batch 1B). This study concluded a change from Frit 200 to Frit 165 is expected to increase the melt rate in DWPF without decreasing waste loading[1].

DWPF requested that SRTC complete a new melt rate study[2] for Macrobatch 3 (Sludge batch 2), the next sludge batch that will be processed by DWPF. This task is more extensive than the Macrobatch 2 study[1]. The Macrobatch 2 study looked at existing frits only while the Macrobatch 3 study was directed to develop new frits and evaluate processing changes to improve melt rate[3].

The proposed DWPF flowsheet for nominal Macrobatch 3 sludge[4] has been tested extensively using simulated sludge. The chemical processing recommended is similar to Macrobatch 2 in that it utilizes a combination of nitric acid and formic acid during the melter feed preparation to produce a glass with a predicted $Fe^{+2}/\Sigma Fe$ ratio of approximately 0.20. Frit 200 was utilized as the glass former. The Macrobatch 3 melt rate study focuses on two changes to improve melt rate: (1) changes in frit composition, and (2) changes to the feed preparation process to alter the redox of the melter feed. These two factors were investigated for Macrobatch 3 (Sludge batch 2) utilizing crucible studies and a specially designed "melt rate" furnace. Other potential factors that could increase melt rate that are being investigated by separate programs include: mechanical mixing via stirring or the use of bubblers, changing the power skewing to redistribute the power input to the melter, and elimination of heat loss (e.g. air inleakage).

2.0 SUMMARY

This report describes the non-radioactive preparation of Macrobatch 3 simulated sludge and Macrobatch 3 Sludge Receipt and Adjustment Tank (SRAT) products necessary for the FY01 melt rate testing. The SRAT products were combined with various frits, dried and size-reduced to produce the dried melter feeds that were used in the crucible and melt rate furnace testing. The results of the crucible and melt rate furnace testing will be summarized in separate reports.

One three-liter and four fifteen-liter batches of nominal Macrobatch 3 SRAT product were produced to support this testing. In addition, three-liter batches representing three processing options were prepared; (1) reducing SRAT product, (2) underwashed SRAT product, and (3) overwashed SRAT product. The underwashed SRAT product was of particular importance to DWPF because tank space and evaporator problems make it more likely that the next sludge batch will be underwashed.

3.0 DISCUSSION

All the testing was completed with melter feed prepared from Macrobatch 3 sludge simulant. The feed preparation process converted the Tank 8 and Tank 40 sludge simulant into feed for the melt rate furnace and crucible testing. The feed preparation process was composed of three steps; 1) Combining the Tank 8 and Tank 40 sludge and adding trim chemicals to produce Tank 8/40 blended sludge simulant (Macrobatch 3 sludge), 2) Processing the sludge simulant through a Sludge Receipt and Adjustment Tank (SRAT) process, and 3) Combining the SRAT product with frit and removing water by drying in an oven. Note that no DWPF Slurry Mix Evaporator (SME) cycles were performed to prepare the melter feeds.

3.1. Sludge Combination and Trimming

The Macrobatch 3 sludge composition for nominally washed and underwashed sludge was predicted by Hank Elder[5]. This recipe was the basis for the sludge preparation. Macrobatch 3 is approximately 50% Tank 40 sludge and 50% Tank 8 sludge by weight. The two sludge simulants were prepared at the Fred Facility at the University of South Carolina[6].

Three significant changes were made in preparing the SRAT products. First, no uranium was added to the sludge since there is no nonradioactive form of uranium that can be added and there is no known nonradioactive element that is a suitable replacement in the chemical processing and in the melter operations. Macrobatch 3 is high in uranium (8.6 wt% U in calcined sludge solids) so this is a significant omission. Additional frit was added as a replacement for the uranium, therefore, the sludge content in glass was unchanged. Second, no mercury was added to the sludge since the melt rate furnace is not designed to handle the mercury vapors and the mercury is not part of the glass matrix (mercury is removed in DWPF chemical processing and any residual mercury in the melter feed is volatile and removed in the melter offgas). The mercury in MB3 is low (0.3 wt% Hg in sludge solids). Third, no insoluble sodium was added in the sludge preparation process. To produce a SRAT product with the correct sodium concentration, the additional sodium was added to the sludge by the addition of sodium nitrate and sodium formate.

The Macrobatch 3 (Tank 8/40) sludge simulant was prepared by combining equal amounts (by weight) of Tank 8 and Tank 40 sludge simulant. The combined sludge was low in manganese and nickel so manganese (IV) oxide (85%), nickel chloride hexahydrate, and noble metals were then added to trim the sludge to the required compositions. Sodium formate and sodium nitrate were added to the sludge to adjust the sodium concentration to desired levels. The ratio of sodium formate and sodium nitrate was calculated to maintain a targeted redox of 0.2 $Fe^{+2}/\Sigma Fe$.

3.2. SRAT Process

A series of SRAT cycles were completed with simulated sludge to produce the SRAT products necessary for the planned crucible and melt rate tests. The SRAT cycles were fairly representative of the recommended processing for Macrobatch 3[4]. The SRAT cycles were completed over two days to give the same twelve hours of boiling as is the practice in a DWPF SRAT cycle and eliminate the need for overtime in the making of the SRAT product. This processing deviation had no impact on the chemical content of the SRAT product and did not impact the objective of this testing. The runs were completed using the SRAT cycle procedure[7] and a run plan specific for each SRAT cycle. The data from the runs were recorded in a lab notebook[8].

The baseline SRAT process was conducted in 3-liter batches and/or 15-liter batches. A schematic of the 15-liter batch setup is included in Figures 1, 2 and 3. The baseline SRAT cycle consisted of adding nitric acid and formic acid to acidify the sludge, concentrating the batch to the original volume, and then refluxing the batch for 8 hours. After the reflux, the SRAT product was concentrated to reduce the time required to dry the material after combining with frit. The nitric acid / formic acid ratio was adjusted to target a glass redox of 0.2 $Fe^{+2}/\Sigma Fe$. A sample of each batch of SRAT product was analyzed to determine the calcine factor (amount of solids remaining after the sample is heated to 900°C). The calcine factor was used to determine the amount of frit to add to produce melter feed.

Four different types of SRAT product were made: baseline (or nominal), reducing (formic acid only), underwashed, and overwashed. The reducing SRAT product was produced with formic acid only (no nitric acid) during the SRAT cycle. The underwashed SRAT product was produced by adding the soluble sodium salts removed during sludge washing to the simulated sludge to represent one less wash during sludge pretreatment. The overwashed sludge had an additional wash performed on the simulated sludge to reduce the sodium content by half. The amount of nitric acid and formic acid added was calculated to target a redox of 0.2 $Fe^{+2}/\Sigma Fe$ for the nominal, underwashed and overwashed SRAT product. The composition of the nominal SRAT product is shown in Table 1. The run plan for this nominal SRAT product preparation is included in Appendix A-1.

Table 1. Composition of Baseline Macrobatch 3 SRAT Product (nominal)

Solids	***Analysis***	***Units***
Total solids	18.85	Wt %
Soluble solids	6.305	Wt %
Insoluble solids	12.55	Wt %
Calcine solids	14.45	Wt %

Elements	***Wt% Elemental (Calcined Basis)***	***Wt % Oxide (Calcined Basis)***
Al	9.870	18.649
B	0.000	0.000
Ba	0.267	0.298
Ca	3.210	4.491
Cr	0.253	0.370
Cu	0.161	0.202
Fe	36.000	51.469
K	0.128	0.154
Li	0.000	0.000
Mg	0.147	0.243
Mn	2.635	3.402
Na	10.450	14.087
Ni	1.030	1.311
P	0.048	0.000
Pb	0.231	0.249
Si	1.395	2.984
Sr	0.116	0.137
Ti	0.005	0.008
Zn	0.315	0.391
Zr	0.619	0.836
Sum of Oxides		99.281

A total of one 3-liter and four 15-liter batches were completed to produce approximately 60 liters of nominal Macrobatch 3 SRAT product. In addition, one 3-liter SRAT cycle was completed for each of the three processing options; formic acid only, underwashed sludge and overwashed sludge. This was enough material for the crucible studies and one melt rate furnace run for each of the processing options. The preparation of each of these processing changes is described below.

Preparation of Reducing SRAT Product

Adding only formic acid (no nitric acid) during the SRAT cycle produced the reducing SRAT product. The same nominal Macrobatch 3 sludge was used as with the nominal SRAT product. The reducing SRAT product contained the same moles of formic acid as were added as either

[#] Based on analysis of sludge from experiments performed by David Koopman.

formic acid or nitric acid in the nominal SRAT product. The hydrogen generation rate was not measured during this SRAT product preparation. The run plan for producing reducing SRAT product is included in Appendix A-2.

Preparation of Overwashed SRAT Product

The overwashed sludge was prepared from 2800 g of nominal Macrobatch 3 sludge by (1) decanting 500 ml of supernate, (2) adding 1075 g of water, (3) decanting 1075 ml of supernate, and adding 500 g of DI water to the sludge. This is approximately equivalent to two additional washes of the sludge. The run plan for the overwashed sludge preparation is included in Appendix B-1. The sludge was analyzed as necessary to calculate the nitric and formic acid additions necessary for the SRAT cycle. The calculations and run plan for producing the overwashed sludge is included in Appendix A-3. The hydrogen generation rate was not measured during this SRAT product preparation.

Preparation of Underwashed SRAT Product

The underwashed sludge was prepared from 2800 g of nominal Macrobatch 3 sludge by adding additional sodium nitrate, sodium nitrite and sodium hydroxide to the sludge. This is approximately equivalent to one less wash cycle in preparing the sludge. The advantage of less washing is that the sludge preparation will be quicker and less spent wash water will be generated. Less spent wash water would be a significant advantage to the high level waste process because of the recent evaporator problems[9]. The run plan for the underwashed sludge preparation is included in Appendix B-2. The sludge was analyzed as necessary to calculate the nitric and formic acid additions necessary for the SRAT cycle. The calculations and run plan for producing the underwashed sludge are included in Appendix A-4. The hydrogen generation rate was not measured during this SRAT product preparation.

3.3.Melter Feed Preparation and Drying

For each crucible or melt rate furnace test, the required amount of SRAT product slurry was combined with the required amount of frit based on a waste loading of 23.2%. This is equivalent to 26 wt% sludge oxide loading in glass (26 wt% sludge oxide loading contains 2.8 wt% U_3O_8 in glass – 26-2.8 wt% = 23.2 wt % uranium free sludge oxide loading in glass). This gives the correct solids concentration of iron, sodium and the other sludge elements in the glass. Of particular concern was the sodium concentration since many of the frits were developed to maximize the alkali content in an attempt to improve melt rate. If more sludge was added to replace the uranium, the concentration of sodium would have been higher in the glass than planned. This could lead to the development of frits that were lower in sodium than desired because of the high sodium content in the sludge. Since uranium was not added in these nonradioactive experiments, frit was added on a weight basis to replace the uranium. Thus a 25 wt% sludge mixture with uranium would lead to a 23.2 wt% sludge mixture without the added uranium oxide.

The frit was added to the SRAT product as a dry powder (DWPF frit size range <80 mesh, 177µm, >200 mesh, 74µm). No Slurry Mix Evaporator (SME) cycles were performed since the frit was added as a dry powder. After batching, the melter feed was dried to the desired batch weight in an oven, then placed in a desiccator until ready to be vitrified.

During the first ten melt rate furnace tests, the melter feed slurry was dried under vacuum at 70°C to reduce the amount of formic acid lost during the drying process. The melter feed was dried to a final batch weight of 794 grams. At this batch weight, the feed for some runs had the consistency of peanut butter while other runs had the consistency of wet clay. The batch height varied from 2.5" to 3.5" during the ten runs.

As a result of the difference in batch height and problems with reproducibility of the initial runs, the feed was dried in an atmospheric convection oven at 105°C for the next six runs to a batch weight of 575 g. The feed was then ground with a mortar and pestle and passed through a #10 mesh screen (<2 mm). The resulting feed had a uniform appearance and batch height prior to firing. The batch weight for all remaining runs was 545 ±5 grams.

The amount of dried SRAT product and frit to be combined for each of the crucible runs was calculated to produce 45 g of glass. The amount of dried SRAT product and frit to be combined for each of the melt rate furnace runs was calculated to produce 500 g of glass. The results are summarized in Table 2 for the nominal batches and in Table 3 for the alternative processing.

Table 2 -- Melt Rate Furnace SRAT Product Calculation – Nominal

Crucible Runs (glass basis)	45grams
Melt Rate Furnace Runs (glass basis)	500grams
Loading (oxide basis)	23.2 %

	MB3 Baseline Feed			
Processing	Nominal			
Batch size	3-liter Batch	15-liter Batch 1	15-liter Batch 2	30-liter Batch 3/4
Date	10/11/00	11/1/00	11/20/00	1/12/01
Calcine Factor, %	15.1	15.44	15.76	15.38
Amount Dried Solids Produced, kg	1.11	13.914	14.0	28.0
Melter Feed for Crucible Studies				
Amount of frit, g	34.56	34.56	34.56	34.56
Amount of SRAT Product, g	69.14	67.62	66.24	67.88
Melter Feed for Melt Rate Furnace Studies				
Amount of frit, g	384.00	384.00	384.00	384.00
Amount of SRAT Product, g	768.21	751.30	736.04	754.23

[#] Based on analysis of sludge from experiments performed by David Koopman.

Table 3-- Melt Rate Furnace SRAT Product Calculation – Processing Alternatives

Crucible Runs (glass basis)	45grams
Melt Rate Furnace Runs (glass basis)	500grams
Loading (oxide basis)	23.2 %

	Alternative Feeds		
Processing	Formic Only	Overwashed	Underwashed
Batch size	3-liter Batch	3-liter Batch	3-liter Batch
Date	12/15/00	12/15/00	12/15/00
Calcine Factor, %	15.46	14.49	16.04
Amount Dried Solids Produced, kg	2.385	2.386	2.442
Melter Feed for Crucible Studies			
Amount of frit, g	34.56	34.56	34.56
Amount of SRAT Product, g	67.53	72.05	65.09
Melter Feed for Melt Rate Furnace Studies			
Amount of frit, g	384.00	384.00	384.00
Amount of SRAT Product, g	750.32	800.55	723.19

Figure 1 -- 22-Liter SRAT Vessel

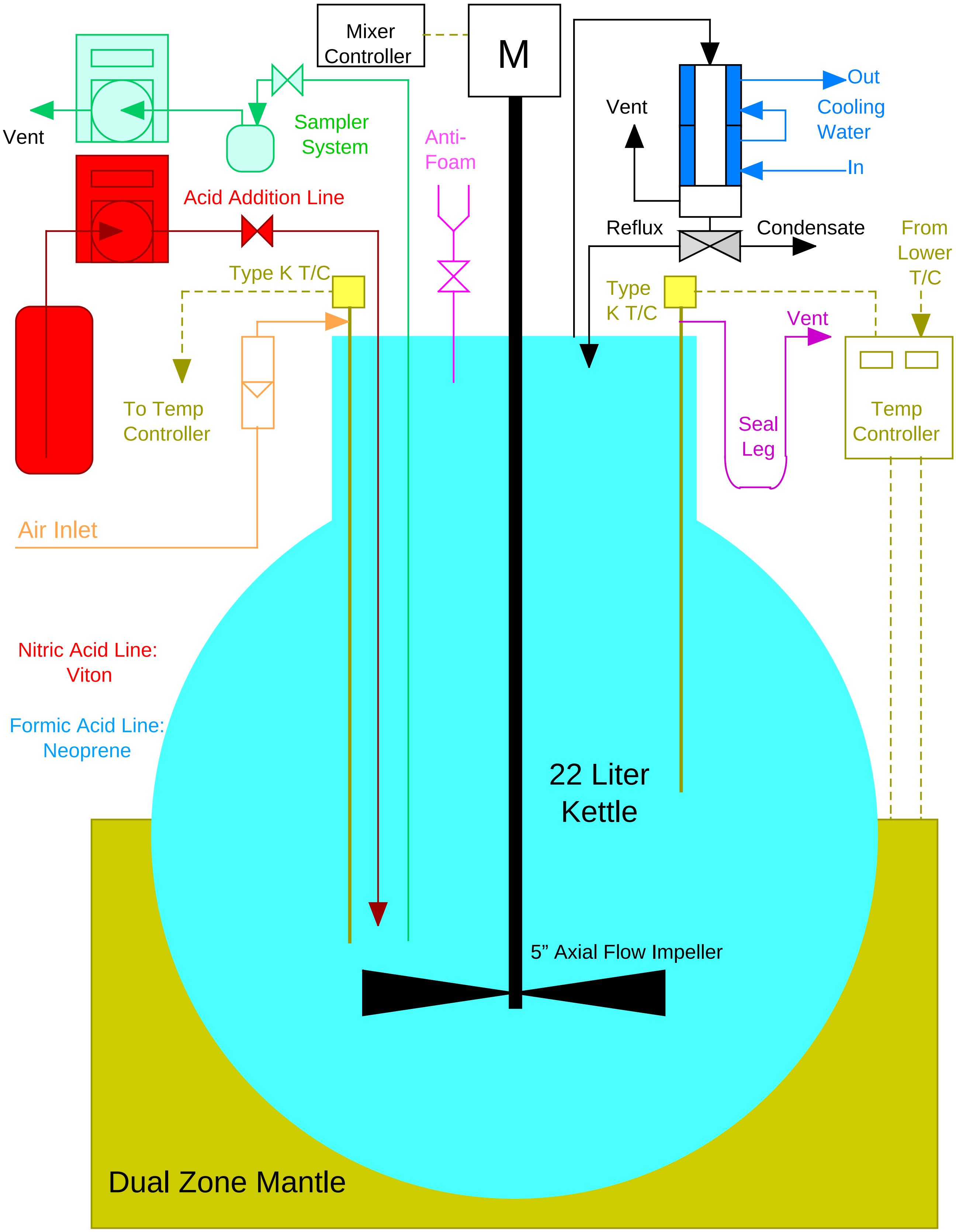
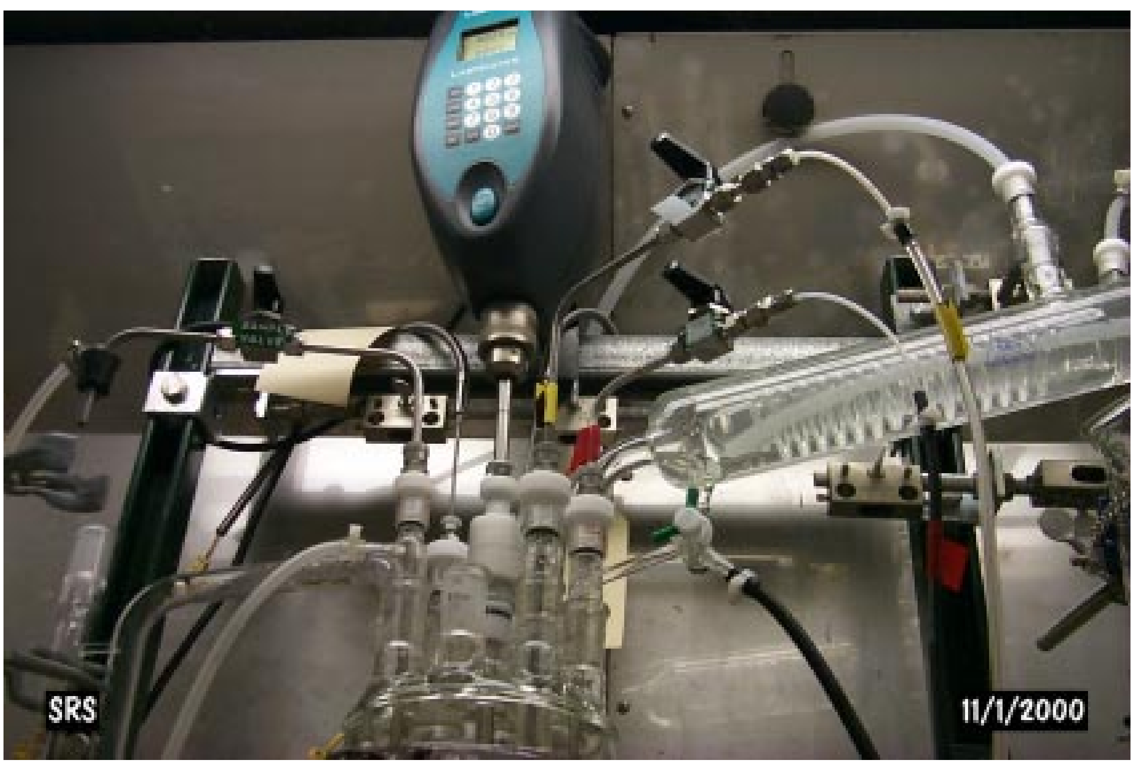


Figure 2 -- Large SRAT Vessel

Figure 3 -- Large SRAT Vessel Upper Assembly



APPENDIX A: MACROBATCH 3 SRAT PRODUCT PREPARATION

Appendix A-1	Macrobatch 3 SRAT Product Preparation for Melt Rate Tests: 15-Liter Batch (U), SRT-PTD-2000-00082, October 31, 2000.
Appendix A-2	Run Plan for Preparation of Macrobatch 3 Reducing SRAT Product for Melt Rate Tests (U), SRT-PTD-2000-00099, December 11, 2000.
Appendix A-3	Run Plan for Preparation of Macrobatch 3 Overwashed SRAT (U), SRT-PTD-2000-00104, December 13, 2000.
Appendix A-4	Run Plan for Preparation of Macrobatch 3 Underwashed SRAT (U), SRT-PTD-2000-00105, December 13, 2000.

APPENDIX B: MACROBATCH 3 SLUDGE PREPARATION

Appendix B-1	Run Plan for Preparation of Macrobatch 3 Overwashed Sludge (U), SRT-PTD-2000-00097, December 7, 2000.
Appendix B-2	Run Plan for Preparation of Macrobatch 3 Underwashed Sludge (U), SRT-PTD-2000-00098, December 7, 2000.

4.0 REFERENCES

[1] WSRC.TR-2000-0395, DWPF Macrobatch 2 Melt Rate Tests, M. E. Stone, D. P. Lambert, October 5, 2000.

[2] Technical Task Request #HLW/DWPF/TTR-00-0044, DWPF Macrobatch 3 Melt Rate Study.

[3] WSRC-RP-2001-00183, Increase Melting Rate of DWPF Feed -- Task Technical & QA Plan, D. P. Lambert, D. K. Peeler, November 13, 2000 (first issued as WSRC-RP-2000-00080). TFA TTP# SR-1-6-WT-31

[4] WSRC-TR-2000-00398, Revision 0, Sludge Batch 2 (Macrobatch 3) Flowsheet Studies With Simulants (U), D. C. Koopman, October 9, 2000.

[5] HLW-SDT-2000-00128, Revision 0, Position Paper on Sludge Batch 2 Qualification Strategy and Simulant Composition, May 9, 2000.

[6] SRT-WHM-2000-004, Rev. 0, 1, and 2, Specification for the Procurement of Tank 40 and Tank 8 Sludge Feed Simulants, M. R. Poirier, June 29, 2000.

[7] Manual L27, Procedure 2.02, Rev 0, Laboratory Scale Chemical Process Cell Simulations, June 8, 1998.

[8] Lab notebooks WSRC-NB-2000-00106 and WSRC-NB-2001-00009.

[9] WSRC-TR-2000-00211, Rev. 2, Technical Basis for the 242-16H Evaporator Cleaning Process, C. S. Boley, M. C. Thompson, W. R. Wilmarth, K. G. Brown, December 6, 2000.

SRS

WESTINGHOUSE SAVANNAH RIVER COMPANY
INTEROFFICE MEMORANDUM

SRT-PTD-2000-00082

October 31, 2000

To: S. L. Marra, 704-1T

From: M. E .Stone, 704-1T

Macrobatch 3 SRAT Product Preparation for Melt Rate Tests: 15-Liter Batch (U)

Reference: SRT-PTD-2000-0071, Run Plan for Preparation of Macrobatch 3 SRAT Product for Melt Rate Tests: 3-Liter Batch (U), October 5, 2000.

This document provides the necessary instructions for preparation of Slurry Mix Evaporator Tank (SRAT) product to be utilized during melt rate tests on Macrobatch 3 sludge simulant. Macrobatch 3 is assumed to be a 48% Tank 8 and 52% Tank 40 blend (based on air-dried solids content). The 22-liter laboratory scale SRAT kettle will be configured as shown in Attachment One. The calculations are based on SRT-PTD-2000-0071 and a scaling factor of 6:1. Acid additions also take into account a 2:1 dilution factor which will be used to increase the acid flowrate to allow subsurface feeding.

Samples of the slurry will be taken after the initial boil down, after refluxing, and at the completion of the run.

The following sequence will be utilized to process the sludge:

SRAT Cycle Sequence

NOTE: Sufficient rinse water should be used after each addition to ensure complete transfer of the material added. Record amount of each rinse water addition in the laboratory notebook.

1. Verify that adequate formic and nitric acid is available and that sample results for the acid match the molarities shown in Table 2.
2. Weight out the required amounts of nitric acid and formic acid and dilute each with the amounts of dilution water shown in Table 1.
3. Verify that leak checks are complete on experimental apparatus.
4. Transfer amount of Tank 40 sludge to SRAT kettle shown in Table 1.
5. Transfer amount of Tank 8 sludge to SRAT kettle shown in Table 1.
6. Turn on SRAT agitator at speed indicated in Table 2.
7. Turn on air purge to SRAT kettle at flowrate indicated in Table 2.
8. Add trim chemicals to SRAT kettle in amounts shown in Table 1.

9. Turn on cooling water to SRAT condensor at temperature shown in Table 2.
10. Add initial amount of antifoam to slurry in amount shown in Table 2.
11. Turn on mantle to heat vessel to temperature indicated in Table 2 for acid additions.
12. Once acid addition temperature is reached, add nitric acid as shown in Table 2.
    - Add required amount of nitric acid to nitric acid addition funnel
    - Open nitric acid addition valve.
    - Pump nitric acid into kettle at flowrate shown in Table 2.
    - Rinse nitric acid addition funnel with 25 ml of DI water.
13. When nitric acid addition is complete, add formic acid as shown in Table 2.
    - Add required amount of formic acid to formic acid addition funnel
    - Open formic acid addition valve.
    - Pump formic acid into kettle at flowrate shown in Table 2.
    - Rinse formic acid addition funnel with 25 ml of DI water.
14. If required, adjust agitator speed to maintain mixing.
15. When addition is complete, bring kettle to boiling by setting temperature controllers to the boilup temperature shown in Table 2 and add antifoam in same amount as initial addition, as shown in Table 2.
16. If required, reset agitator speed to value shown in Table 2.
17. Dewater the kettle by removing the amount of condensate specified by researcher.
18. Reflux the kettle for the time specified in Table 2.
19. Dewater the amount of condensate specified in Table 2.
20. Turn off mantle and allow SRAT kettle to cool overnight.
21. Turn off remaining equipment when vessel cools below boiling.
22. Transfer SRAT contents to a poly-bottle labeled as follows plus the date and time the bottle is filled.

    Macrobatch 3 SRAT Product for Melt Rate Tests
    Tank 8/40 Blend
    Sodium Added as Formate and Nitrate
    Predicted Redox: 0.2 (Baseline Process)

23. Housekeep after completion of run.
    - Disassemble apparatus and remove from hood.
    - Clean all glassware and other items in contact with sludge.
    - Store all equipment in proper location.
    - Wipe down hood and replace floor liner.
    - Dispose of waste properly.

c: L. F. Landon, 704-1T
D. C. Witt, 704-1T
D. C. Koopman, 704-1T
D. P. Lambert, 704-1T
D. K. Peeler, 773-43A
T. H. Loirer, 773-23A

Table 1. Sludge and Trim Chemical Additions

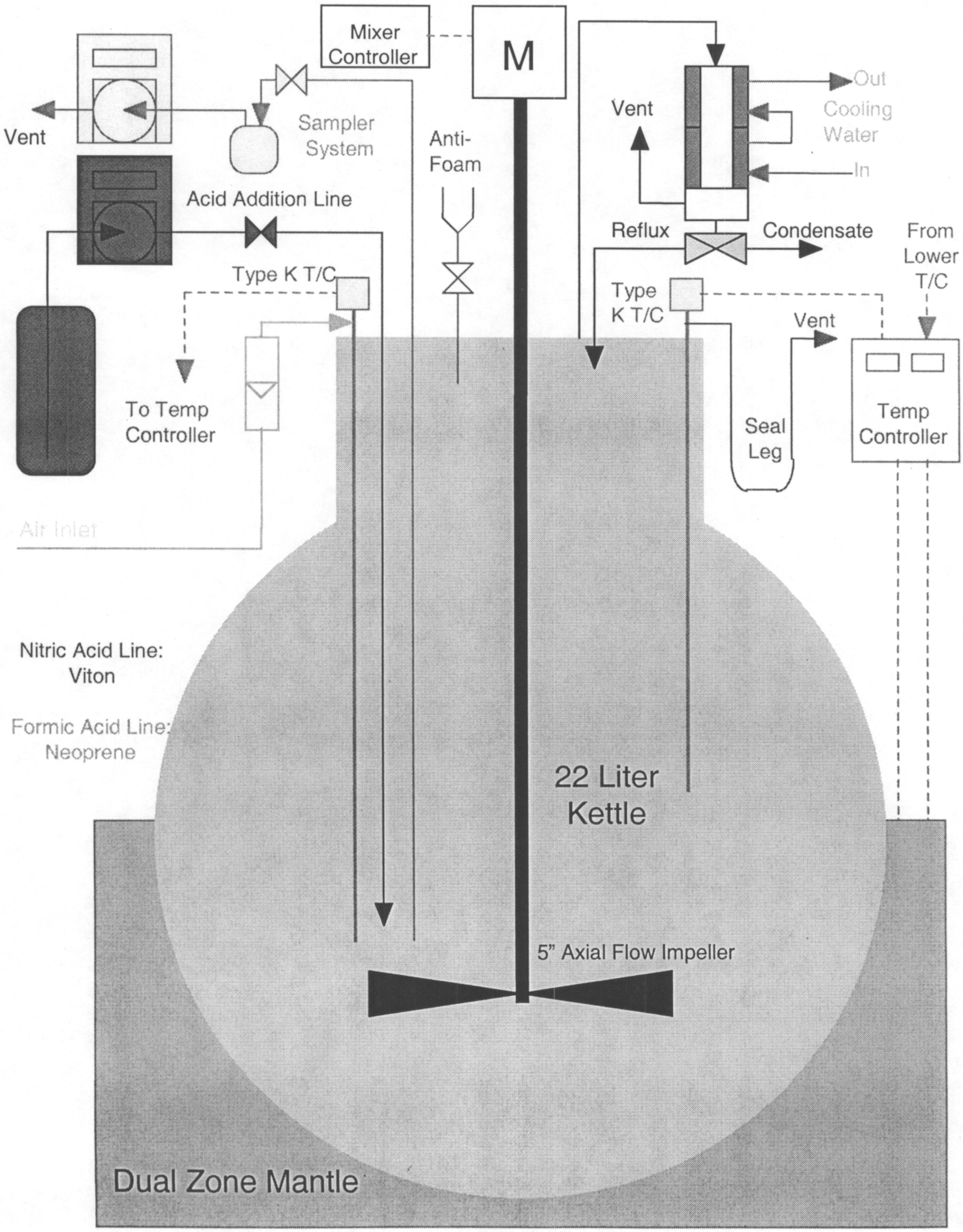
Chemical	Addition Amount (grams)
Tank 40 Sludge Simulant	8,400
Tank 8 Simulant	8, 400
Silver Nitrate	5.2446
Palladium Nitrate Hydrate	16.6800
Rhodium Nitrate Dihydrate	7.8084
Ruthenium Chloride	12.8364
Nickel Chloride Hexahydrate	18.96
Sodium Formate	216.06
Sodium Nitrate	92.22
Manganese Oxide (85%)	36.28

Table 2. Operating Parameters

Parameter	Value	Units
Antifoam Amount (IIT 747, 10%)	21	ml
Nitric Acid Amount (10.15 molar)	90.6	ml
Nitric Acid Dilution Water	90.6	ml
Nitric Acid Flowrate	12.0	ml /min
Formic Acid Amount (22.64 molar)	533.4	ml
Formic Acid Dilution Water	533.4	ml
Formic Acid Flowrate	12.0*	ml /min
Reflux Time	8	hours
Final Dewater Volume	3000	grams
Air Purge Rate	3000	ml /min
Agitator Speed	200#	RPM
Condensor Cooling Water Temperature	15	°C
Acid Addition Temperature (Upper Zone)	88	°C
Acid Addition Temperature (Lower Zone)	93	°C
Boil-up Temperature (Both Zones)	110	°C

* Flowrate adjustments and antifoam additions should be used to minimize foaming.
# Agitator speed should be adjusted to maintain well mixed batch.

Attachment One. SRAT Kettle Equipment Setup



SRS

TM

WESTINGHOUSE SAVANNAH RIVER COMPANY
INTEROFFICE MEMORANDUM

SRT-PTD-2000-0099

December 11, 2000

To: S. L. Marra, 704-1T

From: M. E .Stone, 704-1T *MEAl*

Run Plan for Preparation of Macrobatch 3 Reducing SRAT Product for Melt Rate Tests (U)

This document provides the necessary instructions for preparation of reducing Slurry Receipt and Adjustment Tank (SRAT) product to be utilized during melt rate tests on Macrobatch 3 sludge simulant. Macrobatch 3 is assumed to be a 48% Tank 8 and 52% Tank 40 blend (based on air-dried solids content). Formic acid will be used to adjust the sludge to provide a feed that is as reducing as possible. The four liter laboratory scale SRAT kettle will be configured as shown in Attachment One.

The noble metal additions are based on the chemical process cell tests for this sludge, as documented in SRT-PTD-2000-48. No mercury will be added due to offgas concerns when the material is vitrified. Manganese oxide and nickel chloride additions are based on the baseline run, as documented in SRT-PTD-2000-0071. Calculation of the sodium formate and sodium nitrate additions is shown in Attachment Two. The amount of formic acid required is calculated in Attachment Three.

Samples (30 ml) will be pulled of the sludge prior to acid additions, after completion of the acid additions, after 4 hours of reflux, at the completion of the 8 hours of reflux, and at the completion of the run to allow an evaluation of the manganese oxide.

The following sequence will be utilized to process the sludge:

SRAT Cycle Sequence

1. Verify that adequate formic is available and that sample results for the acid match the molarity shown in Table 1.
2. Verify that leak checks are complete on experimental apparatus.
3. Transfer 1,400 grams of Tank 40 sludge to SRAT kettle.
4. Transfer 1,400 grams of Tank 8 sludge to SRAT kettle.
5. Turn on SRAT agitator at speed indicated in Table 2.
6. Turn on air purge to SRAT kettle at flowrate indicated in Table 2.
7. Add trim chemicals to SRAT kettle in amounts shown in Table 1.
8. Turn on cooling water to SRAT condensor at temperature shown in Table 2.
9. Add initial amount of antifoam to slurry in amount shown in Table 2.
10. Turn on mantle to heat vessel to temperature indicated in Table 2 for acid additions.
11. Add formic acid as shown in Table 2.
12. If required, adjust agitator speed to maintain mixing.
13. When addition is complete, bring kettle to boiling by setting temperature controller to the boilup temperature shown in Table 2.
14. Dewater to remove the amount of condensate specified by researcher.
15. Reflux the kettle for the time specified in Table 2.
16. Dewater the amount specified in Table 2.
17. Turn off mantle and allow SRAT kettle to cool.
18. Turn off remaining equipment.

S. L. Marra
SRT-PTD-2000-0099
December 11, 2000
Page 2 of 5

19. Transfer SRAT contents to a poly-bottle labeled as follows plus the date and time the bottle is filled.

Macrobatch 3 Reducing SRAT Product for Melt Rate Tests
Tank 8/40 Blend
Predicted Redox: 0.26

Table 1. Sludge and Trim Chemical Additions

Chemical	Addition Amount (grams)
Tank 40 Sludge Simulant	1,400
Tank 8 Simulant	1, 400
Silver Nitrate	0.8741
Palladium Nitrate Hydrate	2.7800
Rhodium Nitrate Dihydrate	1.3014
Ruthenium Chloride	2.1394
Nickel Chloride Hexahydrate	3.16
Sodium Formate	29.67
Sodium Nitrate	23.29
Manganese Oxide (85%)	6.047

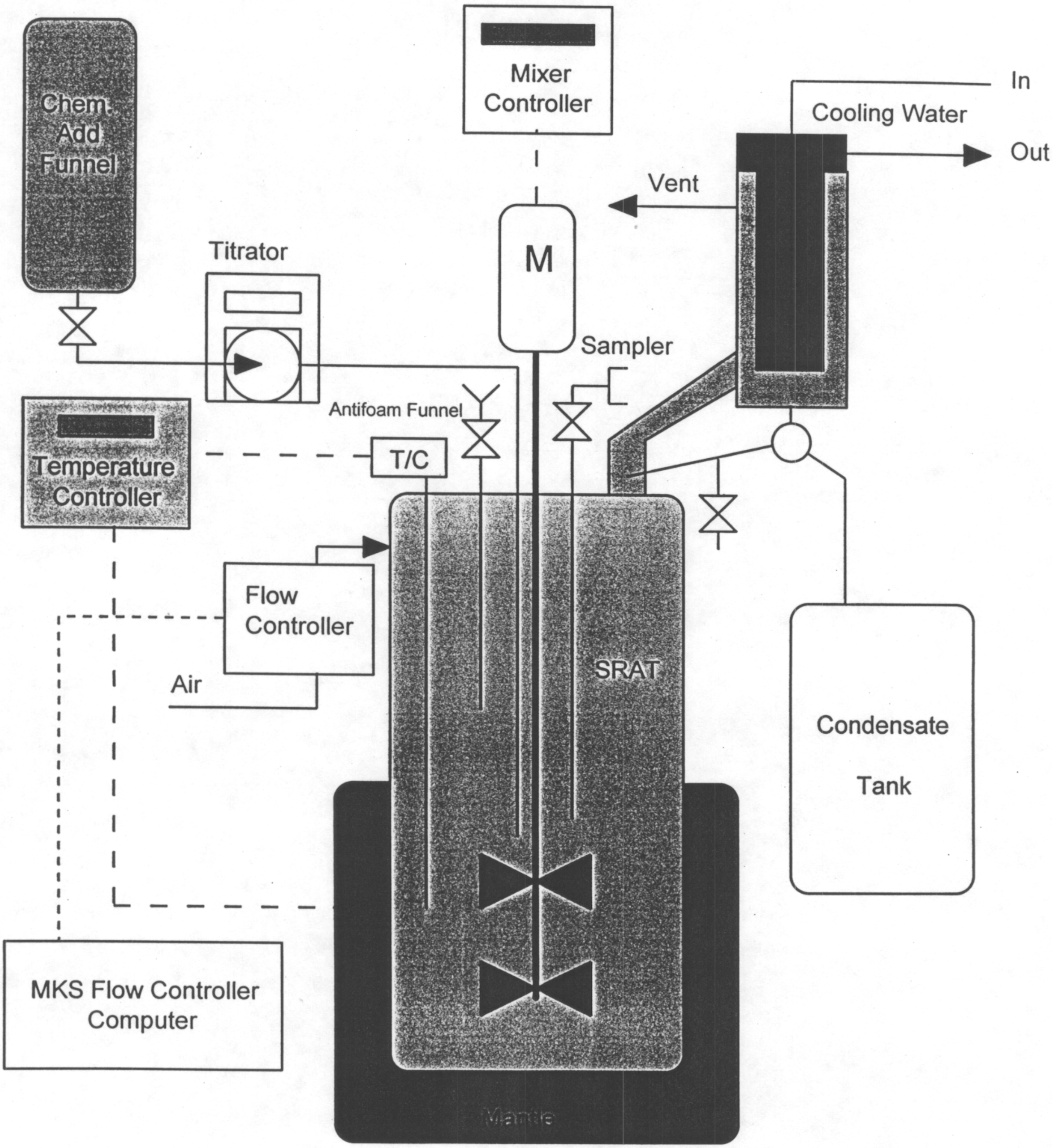
Table 2. Operating Parameters

Parameter	Value	Units
Antifoam Amount (IIT 747, 10%)	3.5	grams
Formic Acid Molarity	22.64	molar
Formic Acid Amount	95.67	ml
Formic Acid Flowrate	1.0*	ml /min
Initial Dewater Volume	TBD	liters
Reflux Time	8	hours
Final Dewater Volume	500	ml
Air Purge Rate	500	ml /min
Agitator Speed	200	RPM
Condensor Cooling Water Temperature	10	°C
Acid Addition Temperature	93	°C
Boil-up Temperature	115	°C

* Flowrate adjustments and antifoam additions should be used to minimize foaming.

c: D. P. Lambert, 704-1T
D. C. Witt, 704-1T
M. F. Williams, 704-1T
PTD Files
D. K. Peeler, 773-43A
T. H. Loirer, 773-23A
J. E. Josephs, 704-1T
Melt Rate Laboratory Notebook

Attachment One. SRAT Kettle Equipment Setup



Attachment Two. Sodium Formate Addition Calculations

Sodium Adjustment				Redox Prediction		
	Value	Units			Value	Units
Initial concentration	6.04	wt %		SRAT Formic Acid Addition	95.67	ml
Desired concentration	9.47	wt %		SME Formic Acid Addition	0.0000	ml
Sludge Quantity	2800.00	grams		Formic Acid Concentration	22.64	molar
Sludge Volume	2.523	liters		Formic Acid Destruction	35	%
Solids Loading	15.40	wt %		Formate from Formic Acid	1.408	moles
Sodium Mol. Wt.	23.00	g/mol		Nitric Acid	0.00	ml
Initial Amount of Na	26.04	grams		Nitric Acid Concentration	10.15	molar
Sodium Addition	0.71	moles		Sludge Nitrite Concentration	8500	mg/kg
Final Concentration	9.47	wt %		Sludge Nitrate Concentration	3800	mg/kg
Ratio of Formate/Total	0.6143	g/g		Conversion - Nitrite to Nitrate	35	%
Amount of Sodium Formate	0.4363	moles		Sludge Density	1.11	kg/L
Amount of Sodium Nitrate	0.2740	moles		Nitrate from Sludge	0.306	moles
Sodium Formate Addition	**29.67**	**grams**		Redox	0.255	Fe+2/Fe
Sodium Nitrate Addition	**23.29**	**grams**		* Redox Calculated per WSRC-RP-97-34		

S. L. Marra

Attachment Three. Calculation of Formic Acid Addition

Raw Sludge Parameters				Stoichiometric Acid		Actual Acid	
Weight % Solids	15.40	wt% of sludge			Moles / L	Moles / L	
Density	1.11	kg / L sludge					
Nitrite	8500	mg / (kg sludge)		Nitrite	0.1538		
Nitrate	3800	mg / (kg sludge)					
Manganese (% of solids)	2.520	wt % of solids		Mn	0.0941		
TIC (Carbonate)	0.000	mg / L		TIC	0.0000		
Hydroxide	0.439	Molar (sludge)		OH-	0.4390		
Mercury (% of solids)	0.000	wt%		Hg	0.0000		
				Total	0.6869	0.8587	
Assumed Parameters							
				Redox Determination			
Conversion of nitrite to nitrate	35.00	%				Moles	
Destruction of formate	35.00	%					
Overall Acid Stoichiometry	125.00	%		Formic Amount		0.859	
Nitric Acid Molarity	10.15	Molar		Nitric Amount		0.000	
Formic Acid Molarity	22.64	Molar		Nitrate from Nitrite		0.0718	
				Nitrate (w/o Na addition)		0.1398	
Redox Target	0.200	Fe+2 / Fe		Predicted Redox *		0.255	
Ratio of Formic to Total Acid	1.000	mole / mole		* Redox Calculated per WSRC-RP-97-34			
Molecular Weights							
Nitrite	46.00	g / mole		Acid Volumes		ml / L	
Nitrate	62.00	g / mole					
Manganese	54.94	g / mole		Formic Acid		37.926	
Carbonate	60.01	g / mole		Nitric Acid		0.000	
Mercury	200.60	g / mole					
Factors for Acid Calculation*				Batch Calculations			
Acid Requirement for Nitrites	0.75	mole / mole		Batch Size		2.52	liters
Acid Requirement for Mn	1.20	mole / mole		Batch Weight		2.800	kg
Acid Requirement for Hg	1.00	mole / mole		Formic Acid Addition		95.6698	ml
Acid Requirement for Hydroxide	1.00	mole / mole		Nitric Acid Addition		0.0000	ml
Acid Requirement for Carbonate	2.00	mole / mole					
* Factors are based on WSRC-RP-92-1056							

WESTINGHOUSE SAVANNAH RIVER COMPANY
INTEROFFICE MEMORANDUM

SRT-PTD-2000-0104

December 13, 2000

To: S. L. Marra, 704-1T

From: M. E .Stone, 704-1T

Run Plan for Preparation of Macrobatch 3 Overwashed SRAT Product (U)

This document provides the necessary instructions for preparation of reducing Slurry Receipt and Adjustment Tank (SRAT) product to be utilized during melt rate tests on Macrobatch 3 sludge simulant. Macrobatch 3 is assumed to be a 48% Tank 8 and 52% Tank 40 blend (based on air-dried solids content). Formic acid will be used to adjust the sludge to provide a feed that is as reducing as possible. The four liter laboratory scale SRAT kettle will be configured as shown in SRT-PTD-2000-00097.

The noble metal additions, manganese oxide and nickel were added per SRT-PTD-2000-0097. Calculation of the sodium formate and sodium nitrate additions is shown in Table Three. The amounts of nitric and formic acid required is calculated in Attachment One. Two samples (30 ml) will be pulled of the sludge at the completion of the run.

The following sequence will be utilized to process the sludge:

SRAT Cycle Sequence

1. Verify that adequate formic and nitric acid is available and that sample results for the acid match the molarity shown in Table 1.
2. Turn on SRAT agitator at speed indicated in Table 2.
3. Turn on air purge to SRAT kettle at flowrate indicated in Table 2.
4. Add trim chemicals to SRAT kettle in amounts shown in Table 1.
5. Turn on cooling water to SRAT condensor at temperature shown in Table 2.
6. Add initial amount of antifoam to slurry in amount shown in Table 2.
7. Turn on mantle to heat vessel to temperature indicated in Table 2 for acid additions.
8. Add nitric acid as shown in Table 2.
9. Add formic acid as shown in Table 2.
10. If required, adjust agitator speed to maintain mixing.
11. When addition is complete, bring kettle to boiling by setting temperature controller to the boilup temperature shown in Table 2.
12. Dewater to remove the amount of condensate specified by researcher.
13. Reflux the kettle for the time specified in Table 2.
14. Dewater the amount specified in Table 2.
15. Turn off mantle and allow SRAT kettle to cool.
16. Turn off remaining equipment.
17. Transfer SRAT contents to a poly-bottle labeled as follows plus the date and time the bottle is filled.

    Macrobatch 3 Overwashed SRAT Product for Melt Rate Tests
    Tank 8/40 Blend
    Predicted Redox: 0.20

Table 1. Sludge and Trim Chemical Additions

Chemical	Addition Amount (grams)
Sodium Formate	36.50
Sodium Nitrate	14.75

Table 2. Operating Parameters

Parameter	Value	Units
Antifoam Amount (IIT 747, 10%)	3.5	grams
Nitric Acid Molarity	10.15	Molar
Nitric Acid Amount	17.01	Ml
Nitric Acid Flowrate	1.0	ml/min
Formic Acid Molarity	22.64	molar
Formic Acid Amount	65.62	ml
Formic Acid Flowrate	1.0*	ml /min
Initial Dewater Volume	TBD	liters
Reflux Time	8	hours
Final Dewater Volume	500	ml
Air Purge Rate	500	ml /min
Agitator Speed	200	RPM
Condensor Cooling Water Temperature	10	°C
Acid Addition Temperature	93	°C
Boil-up Temperature	115	°C

* Flowrate adjustments and antifoam additions should be used to minimize foaming.

Table 3. Sodium Formate Addition Calculations

Insoluble Sodium Adjustment				Redox Prediction		
	Value	Units			Value	Units
Initial concentration	6.04%	wt %		SRAT Formic Acid Addition	65.62	ml
Desired concentration	9.47%	wt %		SME Formic Acid Addition	0.0000	ml
Sludge Quantity	2800.00	grams		Formic Acid Concentration	22.64	molar
Sludge Volume	2.523	liters		Formic Acid Destruction	35	%
Solids Loading	15.40%	wt %		Formate from Formic Acid	0.966	moles
Sodium Mol. Wt.	23.00	g/mol		Nitric Acid	17.01	ml
Initial Amount of Na	26.04	grams		Nitric Acid Concentration	10.15	molar
Sodium Addition	0.71	moles		Sludge Nitrite Concentration	4520	mg/kg
Final Concentration	9.47	wt %		Sludge Nitrate Concentration	2650	mg/kg
Ratio of Formate/Total	0.7556	g/g		Conversion - Nitrite to Nitrate	35	%
Amount of Sodium Formate	0.5367	moles		Sludge Density	1.11	kg/L
Amount of Sodium Nitrate	0.1736	moles		Nitrate from Sludge	0.191	moles
Sodium Formate Addition	**36.50**	**grams**		Redox	0.200	Fe+2/Fe
Sodium Nitrate Addition	**14.75**	**grams**		* Redox Calculated per WSRC-RP-97-34		

c:

D. P. Lambert, 704-1T
D. C. Witt, 704-1T
M. F. Williams, 704-1T
PTD Files

D. K. Peeler, 773-43A
T. H. Loirer, 773-23A
J. E. Josephs, 704-1T
Melt Rate Laboratory Notebook

Attachment One. Calculation of Formic Acid Addition

Raw Sludge Parameters				Stoichiometric Acid		Actual Acid	
Weight % Solids	14.30	wt% of sludge			Moles / L	Moles / L	
Density	1.11	kg / L sludge					
Nitrite	4520	mg / (kg sludge)		Nitrite	0.0818		
Nitrate	2650	mg / (kg sludge)					
Manganese (% of solids)	3.160	wt % of solids		Mn	0.1096		
TIC (Carbonate)	0.000	mg / L		TIC	0.0000		
Hydroxide	0.335	Molar (sludge)		OH-	0.3346		
Mercury (% of solids)	0.000	wt%		Hg	0.0000		
				Total	0.5259	0.6574	
Assumed Parameters							
				Redox Determination			
Conversion of nitrite to nitrate	35.00	%				Moles	
Destruction of formate	35.00	%					
Overall Acid Stoichiometry	125.00	%		Formic Amount		0.589	
Nitric Acid Molarity	10.15	Molar		Nitric Amount		0.068	
Formic Acid Molarity	22.64	Molar		Nitrate from Nitrite		0.0382	
				Nitrate (w/o Na addition)		0.1541	
Redox Target	0.200	Fe+2 / Fe		Predicted Redox *		0.200	
Ratio of Formic to Total Acid	0.896	mole / mole		* Redox Calculated per WSRC-RP-97-34			
Molecular Weights							
Nitrite	46.00	g / mole		Acid Volumes		ml / L	
Nitrate	62.00	g / mole					
Manganese	54.94	g / mole		Formic Acid		26.014	
Carbonate	60.01	g / mole		Nitric Acid		6.744	
Mercury	200.60	g / mole					
Factors for Acid Calculation*				Batch Calculations			
Acid Requirement for Nitrites	0.75	mole / mole		Batch Size		2.52	liters
Acid Requirement for Mn	1.20	mole / mole		Batch Weight		2.800	kg
Acid Requirement for Hg	1.00	mole / mole		Formic Acid Addition		65.6217	ml
Acid Requirement for Hydroxide	1.00	mole / mole		Nitric Acid Addition		17.0108	ml
Acid Requirement for Carbonate	2.00	mole / mole					
* Factors are based on WSRC-RP-92-1056							

SRS™

WESTINGHOUSE SAVANNAH RIVER COMPANY
INTEROFFICE MEMORANDUM

SRT-PTD-2000-0105

December 13, 2000

To: S. L. Marra, 704-1T

From: M. E .Stone, 704-1T

Run Plan for Preparation of Macrobatch 3 Underwashed SRAT Product (U)

This document provides the necessary instructions for preparation of reducing Slurry Receipt and Adjustment Tank (SRAT) product to be utilized during melt rate tests on Macrobatch 3 sludge simulant. Macrobatch 3 is assumed to be a 48% Tank 8 and 52% Tank 40 blend (based on air-dried solids content). Formic acid will be used to adjust the sludge to provide a feed that is as reducing as possible. The four liter laboratory scale SRAT kettle will be configured as shown in SRT-PTD-2000-00098.

The noble metal additions, manganese oxide and nickel were added per SRT-PTD-2000-0098. Calculation of the sodium formate and sodium nitrate additions is shown in Table Three. The amounts of nitric and formic acid required is calculated in Attachment One. Two samples (30 ml) will be pulled of the sludge at the completion of the run.

The following sequence will be utilized to process the sludge:

SRAT Cycle Sequence

1. Verify that adequate formic and nitric acid is available and that sample results for the acid match the molarity shown in Table 1.
2. Turn on SRAT agitator at speed indicated in Table 2.
3. Turn on air purge to SRAT kettle at flowrate indicated in Table 2.
4. Add trim chemicals to SRAT kettle in amounts shown in Table 1.
5. Turn on cooling water to SRAT condensor at temperature shown in Table 2.
6. Add initial amount of antifoam to slurry in amount shown in Table 2.
7. Turn on mantle to heat vessel to temperature indicated in Table 2 for acid additions.
8. Add nitric as shown in Table 2.
9. Add formic acid as shown in Table 2.
10. If required, adjust agitator speed to maintain mixing.
11. When addition is complete, bring kettle to boiling by setting temperature controller to the boilup temperature shown in Table 2.
12. Dewater to remove the amount of condensate specified by researcher.
13. Reflux the kettle for the time specified in Table 2.
14. Dewater the amount specified in Table 2.
15. Turn off mantle and allow SRAT kettle to cool.
16. Turn off remaining equipment.
17. Transfer SRAT contents to a poly-bottle labeled as follows plus the date and time the bottle is filled.

    Macrobatch 3 Underwashed SRAT Product for Melt Rate Tests
    Tank 8/40 Blend
    Predicted Redox: 0.20

Table 1. Sludge and Trim Chemical Additions

Chemical	Addition Amount (grams)
Sodium Formate	35.21
Sodium Nitrate	16.37

Table 2. Operating Parameters

Parameter	Value	Units
Antifoam Amount (IIT 747, 10%)	3.5	grams
Nitric Acid Molarity	10.15	molar
Nitric Acid Amount	11.14	ml
Nitric Acid Flowrate	1.0	ml /min
Formic Acid Molarity	22.64	molar
Formic Acid Amount	102.42	ml
Formic Acid Flowrate	1.0*	ml /min
Initial Dewater Volume	TBD	liters
Reflux Time	8	hours
Final Dewater Volume	500	ml
Air Purge Rate	500	ml /min
Agitator Speed	200	RPM
Condensor Cooling Water Temperature	10	°C
Acid Addition Temperature	93	°C
Boil-up Temperature	115	°C

* Flowrate adjustments and antifoam additions should be used to minimize foaming.

Table 3. Sodium Formate Addition Calculations

Insoluble Sodium Adjustment				Redox Prediction		
	Value	Units			Value	Units
Initial concentration	6.04%	wt %		SRAT Formic Acid Addition	102.42	ml
Desired concentration	9.47%	wt %		SME Formic Acid Addition	0.0000	ml
Sludge Quantity	2800.00	grams		Formic Acid Concentration	22.64	molar
Sludge Volume	2.523	liters		Formic Acid Destruction	35	%
Solids Loading	15.40%	wt %		Formate from Formic Acid	1.507	moles
Sodium Mol. Wt.	23.00	g/mol		Nitric Acid	11.14	ml
Initial Amount of Na	26.04	grams		Nitric Acid Concentration	10.15	molar
Sodium Addition	0.71	moles		Sludge Nitrite Concentration	9160	mg/kg
Final Concentration	9.47	wt %		Sludge Nitrate Concentration	5890	mg/kg
Ratio of Formate/Total	0.7289	g/g		Conversion - Nitrite to Nitrate	35	%
Amount of Sodium Formate	0.5177	moles		Sludge Density	1.11	kg/L
Amount of Sodium Nitrate	0.1926	moles		Nitrate from Sludge	0.411	moles
Sodium Formate Addition	**35.21**	**grams**		Redox	0.200	Fe+2/Fe
Sodium Nitrate Addition	**16.37**	**grams**		* Redox Calculated per WSRC-RP-97-34		

c: D. P. Lambert, 704-1T
D. C. Witt, 704-1T
M. F. Williams, 704-1T
PTD Files
D. K. Peeler, 773-43A
T. H. Loirer, 773-23A
J. E. Josephs, 704-1T
Melt Rate Laboratory Notebook

Attachment One. Calculation of Formic Acid Addition

Raw Sludge Parameters		
Weight % Solids	16.70	wt% of sludge
Density	1.11	kg / L sludge
Nitrite	9160	mg / (kg sludge)
Nitrate	5890	mg / (kg sludge)
Manganese (% of solids)	2.659	wt % of solids
TIC (Carbonate)	0.000	mg / L
Hydroxide	0.498	Molar (sludge)
Mercury (% of solids)	0.000	wt%
Assumed Parameters		
Conversion of nitrite to nitrate	35.00	%
Destruction of formate	35.00	%
Overall Acid Stoichiometry	125.00	%
Nitric Acid Molarity	10.15	Molar
Formic Acid Molarity	22.64	Molar
Redox Target	0.200	Fe+2 / Fe
Ratio of Formic to Total Acid	0.953	mole / mole
Molecular Weights		
Nitrite	46.00	g / mole
Nitrate	62.00	g / mole
Manganese	54.94	g / mole
Carbonate	60.01	g / mole
Mercury	200.60	g / mole
Factors for Acid Calculation*		
Acid Requirement for Nitrites	0.75	mole / mole
Acid Requirement for Mn	1.20	mole / mole
Acid Requirement for Hg	1.00	mole / mole
Acid Requirement for Hydroxide	1.00	mole / mole
Acid Requirement for Carbonate	2.00	mole / mole
* Factors are based on WSRC-RP-92-1056		

Stoichiometric Acid		Actual Acid	
	Moles / L	Moles / L	
Nitrite	0.1658		
Mn	0.1076		
TIC	0.0000		
OH-	0.4978		
Hg	0.0000		
Total	0.7712	0.9640	
Redox Determination			
		Moles	
Formic Amount		0.919	
Nitric Amount		0.045	
Nitrate from Nitrite		0.0774	
Nitrate (w/o Na addition)		0.2276	
Predicted Redox *		0.200	
* Redox Calculated per WSRC-RP-97-34			
Acid Volumes		ml / L	
Formic Acid		40.601	
Nitric Acid		4.417	
Batch Calculations			
Batch Size		2.52	liters
Batch Weight		2.800	kg
Formic Acid Addition		102.4165	ml
Nitric Acid Addition		11.1432	ml

SRS

WESTINGHOUSE SAVANNAH RIVER COMPANY

INTEROFFICE MEMORANDUM

SRT-PTD-2000-00097

December 7, 2000

To: S. L. Marra, 704-1T

From: M. E .Stone, 704-1T *mES*

Run Plan for Preparation of Macrobatch 3 Overwashed Sludge (U)

This document provides the necessary instructions for preparation of Macrobatch 3 overwashed sludge to be utilized during melt rate tests on Macrobatch 3 sludge simulant. Macrobatch 3 is assumed to be a 48% Tank 8 and 52% Tank 40 blend (based on air-dried solids content). The four liter laboratory scale SRAT kettle will be configured as shown in Attachment One. The basis for the washing sequence is shown in Attachment Two.

The sludge will be washed by the following sequence:

1. Addition of 2800 grams of Tank 8/40 Blend sludge to vessel.
2. Allow solids to settle.
3. Decant 500* ml of supernate from vessel.
4. Add 1075 grams of DI water to vessel.
5. Mix contents of vessel thoroughly.
6. Allow solids to settle.
7. Decant 1075 ml of supernate from vessel.
8. Add 500 grams of DI water to vessel.

* If 500 ml of supernate cannot be removed, contact researcher for instructions.

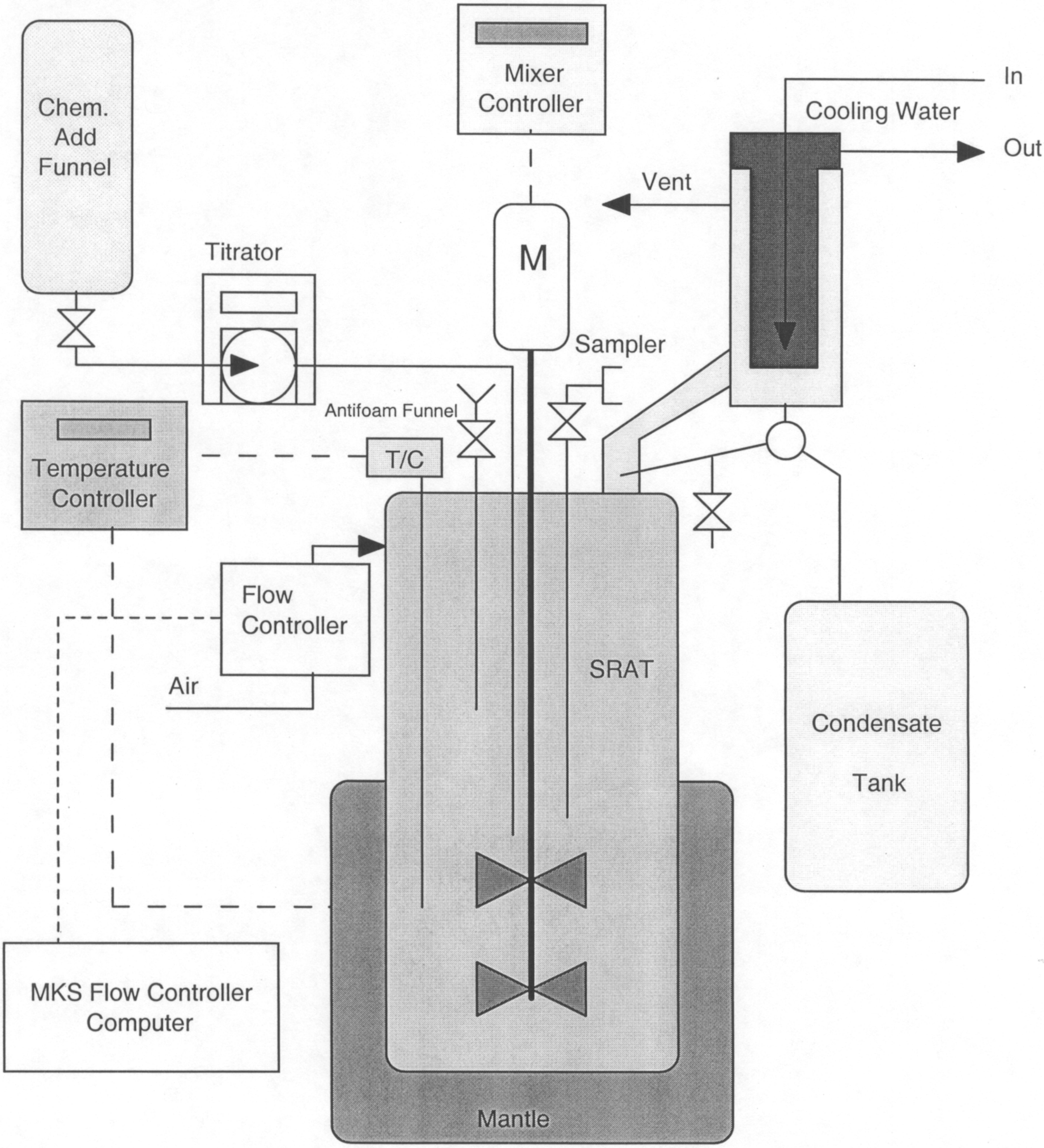
After washing, the sludge will be trimmed as shown in Table 1. The noble metal additions are based on the chemical process cell tests for this sludge, as documented in SRT-PTD-2000-48 and shown in Table 1. No mercury will be added due to offgas concerns when the material is vitrified. Addition of the manganese oxide and nickel chloride hexahydrate are based on the 3L baseline run, as documented in SRT-PTD-2000-0071.

Table 1. Trim Chemical Additions

Chemical	Addition Amount (grams)
Silver Nitrate	0.8741
Palladium Nitrate Hydrate	2.7800
Rhodium Nitrate Dihydrate	1.3014
Ruthenium Chloride	2.1394
Nickel Chloride Hexahydrate	3.16
Manganese Oxide (85%)	6.047

After trim chemical addition, the sludge will be mixed thoroughly and a 40 ml sample pulled. Analysis of the sample must be expedited to allow the acid addition calculations to be performed.

c: L. F. Landon, 704-1T
D. K. Peeler, 773-43A
J. E. Joseph, 704-1T
D. P. Lambert, 704-1T
D. C. Koopman, 704-1T
PTD Files
D. C. Witt, 704-1T
T. H. Loirer, 773-23A
Melt Rate Notebook


Chem.
Add
Funnel
Mixer
Controller
In
Cooling Water
Out
Vent
M
Titrator
Sampler
Antifoam Funnel
T/C
Temperature
Controller
Flow
Controller
Air
SRAT
Condensate
Tank
MKS Flow Controller
Computer
Mantle

Attachment Two: Calculation of Washing Requirements

Given

Supernate Na concentration of washed sludge	0.50	molar
Desired supernate Na concentration	0.25	molar
Sludge weight percent solids	15.4	wt%
Sludge density	1.11	g/ml
Density of supernate	1.03	g/ml

Calculation

Solids weight in 1L of sludge	171	grams
Water weight in 1L of sludge	939	grams
Water volume	939	ml
Initial Decant	200	ml
Moles of sodium in supernate	470	millimoles
Moles of sodium in initial decant	100	millimoles
Moles of sodium remaining after initial decant	370	millimoles
Wash volume	430	ml
Sodium concentration after 1st wash addition	0.316	molar
Second decant volume	430	ml
Moles of sodium in second decant	136	millimoles
Moles of sodium remaining after second decant	234	millimoles
Final addition	200	ml
Final supernate volume	939	ml
Final sodium concentration in wash sludge	0.249	molar

Full Batch Calculations

Batch Size	2.5	liters

Initial Decant	500	ml
First wash volume	1075	ml
Second Decant volume	1075	ml
Final Addition volume	500	ml

SRS

WESTINGHOUSE SAVANNAH RIVER COMPANY
INTEROFFICE MEMORANDUM

SRT-PTD-2000-00098

December 7, 2000

To: S. L. Marra, 704-1T

From: M. E .Stone, 704-1T MCAL

Run Plan for Preparation of Macrobatch 3 Underwashed Sludge (U)

This document provides the necessary instructions for preparation of Macrobatch 3 underwashed sludge to be utilized during melt rate tests on Macrobatch 3 sludge simulant. Macrobatch 3 is assumed to be a 48% Tank 8 and 52% Tank 40 blend (based on air-dried solids content). The four liter laboratory scale SRAT kettle will be configured as shown in Attachment One.

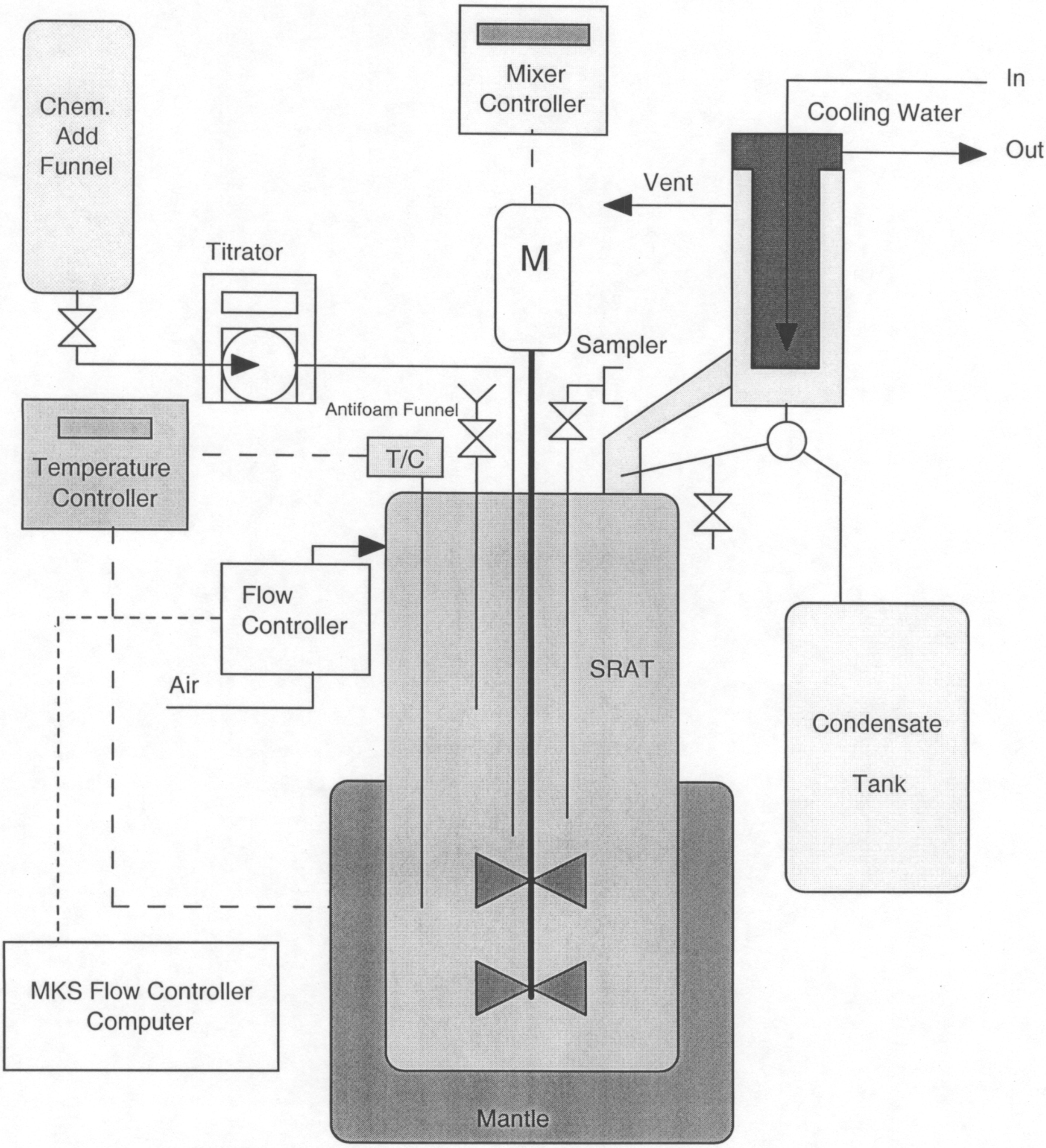
After addition to the kettle the sludge will be trimmed as shown in Table 1. The noble metal additions are based on the chemical process cell tests for this sludge, as documented in SRT-PTD-2000-48 and shown in Table 1. No mercury will be added due to offgas concerns when the material is vitrified. Addition of the manganese oxide and nickel chloride hexahydrate are based on the 3L baseline run, as documented in SRT-PTD-2000-0071. Calculation of the addition amount for the soluble sodium species and rinse water amount is shown in Attachment Two.

Table 1. Trim Chemical Additions

Chemical	Addition Amount (grams)
Silver Nitrate	0.8741
Palladium Nitrate Hydrate	2.7800
Rhodium Nitrate Dihydrate	1.3014
Ruthenium Chloride	2.1394
Nickel Chloride Hexahydrate	3.16
Manganese Oxide (85%)	6.047
Sodium Nitrate	14.05
Sodium Nitrite	20.19
Sodium Hydroxide	10.18
Deionized Water	236

After trim chemical addition, the sludge will be mixed thoroughly and a 40 ml sample pulled. Analysis of the sample must be expedited to allow the acid addition calculations to be performed.

c: L. F. Landon, 704-1T
D. K. Peeler, 773-43A
J. E. Joseph, 704-1T
D. P. Lambert, 704-1T
D. C. Koopman, 704-1T
PTD Files
D. C. Witt, 704-1T
T. H. Loirer, 773-23A
Melt Rate Notebook


Chem.
Add
Funnel
Mixer
Controller
In
Cooling Water
Out
Vent
M
Titrator
Sampler
Antifoam Funnel
Temperature
Controller
T/C
Flow
Controller
Air
SRAT
Condensate
Tank
MKS Flow Controller
Computer
Mantle

Attachment Two. Underwashed Sludge Sodium Trim (One less wash)

Given		
Supernate Na concentration of washed sludge	0.50	molar
Supernate Na concentration of underwashed sludge	0.73	molar
Sludge weight percent solids	15.4	wt%
Sludge density	1.11	g/ml
Density of supernate	1.03	g/ml
Concentration of hydroxide in sludge	0.2	molar
Concentration of nitrate in sludge	0.13	molar
Concentration of nitrite in sludge	0.23	molar
Calculation of Soluble Sodium Addition per Liter		
Solids weight in 1L of sludge	171	grams
Water weight in 1L of sludge	939	grams
Water volume in 1L of sludge	939	ml
Change in Na concentration	0.230	molar
Moles of Na required per liter of sludge	0.216	moles
Ratio of nitrite to nitrate	1.77	mol/mol
Ratio of hydroxide to nitrate	1.54	mol/mol
Moles of sodium nitrate required per liter of sludge	0.0501	moles
Moles of sodium hydroxide required per liter of sludge	0.0771	moles
Moles of sodium nitrite required per liter of sludge	0.0887	moles
Grams of sodium nitrate required per liter of sludge	4.26	grams
Grams of sodium hydroxide required per liter of sludge	3.09	grams
Grams of sodium nitrite required per liter of sludge	6.12	grams
Total Solids after solids addition	184.4	grams
Total weight after solids addition	1123.5	grams
Weight % solids after solids addition	16.41	wt %
Supernate Addition per Liter	94.43	ml
Moles of sodium nitrate in supernate addition	0.02	moles
Grams of sodium nitrate in supernate addition	1.36	grams
Grams of sodium nitrite in supernate addition	1.95	grams
Grams of sodium hydroxide in supernate addition	0.98	grams
Total Solids after supernate addition	188.7	grams
Total weight after supernate addition	1222.2	grams
Weight % solids after supernate addition	15.44	wt %
Total Additions per Liter		
Water	94.43	ml
Sodium Nitrate	5.62	grams
Sodium Nitrite	8.07	grams
Sodium Hydroxide	4.07	grams
Full Batch Calculations		
Batch Size	2.5	liters
Grams of water required	236.064	grams
Grams of sodium nitrate required	14.05	grams
Grams of sodium hydroxide required	10.18	grams
Grams of sodium nitrite required	20.19	grams