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SUMMARY OF RESULTS FROM MINIMELTER RUN WITH MACROBATCH 3 BASELINE FEED USING FRIT 320 (U)

D. H. Miller

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

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D. H. Miller

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Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

APPROVALS

D. H. Miller, Author
Immobilization Technology Section

5/2/02
Date

M. E. Smith, Technical Reviewer
Immobilization Technology Section

5/6/02
Date

S. L. Marra, Manager
Glass Form & Process Development Group

5/15/02
Date

E. W. Holtzscheiter, Manager
Immobilization Technology Section

5/15/02
Date

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

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1.0 INTRODUCTION

This report covers the testing completed as the melter was transitioned from a simulated blend of Tank 8/40(sludge Batch 2) with frit 200 to a simulated blend of Tank 8/40 with frit 320. The same sludge was used to produce both batches of feed in the Glass Feed Preparation System(GFPS). This sludge is referred to as both Sludge Batch 2 and Macrobatch 3 in different reports. The testing was outlined in Task Technical and QA Plan: WSRC-RP-2002-00075, Mini-melter Run with Frit 320 and Sludge Batch 2. The run plan detailing the steps for testing was SRT-GPD-2002-00014: Run Plan for Minimelter with Macrobatch 3 Baseline Feed Using Frit 320 (U). The laboratory notebook used for recording the observations and results was WSRC-NB-2000-00186: 786-A Minimelter. The feed for this melter run was prepared in the GFPS under Task Technical and QA Plan WSRC-RP-2002-00065, GFPS Runs for Minimelter Feed Preparation with Frit 320 (U).

2.0 SUMMARY

The general objectives of the run were:

- Determine a relative melt rate for the mini-melter using frit 320.
- Determine off-gas composition under a variety of test conditions.
- Establish operational differences between the use of frit 200 and frit 320.

The objectives of the task plan were met during testing. General melt rate values indicate that frit 320 allows approximately a 20% higher melt rate when compared to frit 200. The glass made with frit 320 has a different set of power requirements, but no adverse process problems were discovered. The off-gas generation rates were determined at several plenum temperatures. Data was collected that can be used to estimate the difference between actual and indicated plenum temperature.

3.0 DISCUSSION

The 786-A minimelter is joule heated with a one-foot diameter Carborundum Monofax K-3 refractory pot. The vertical electrodes and plenum are made of Inconel 690. There are two vertical Kanthal™ lid heaters that are capable of supplying 5000 watts each. The overflow spout is heated by a split clam shell 1500W resistance heater. The melter is kept under partial vacuum with an air eductor and pressure is controlled with the addition of air. The off-gas passes through a quencher/scrubber and then through a mist eliminator prior to exiting a stack. Sample ports allow the off-gas to be sampled at the melter exit and after the condensate tank. Two gas chromatographs (GC) are used for analysis. A sketch of the melter system is shown in

Attachment A. The campaign was conducted with very few problems caused by feed line pluggage. Continuous pouring at a slightly negative pressure allowed the melter to operate with a minimum number of high glass level alarms.

3.1 Melter Turnover

At the start of testing, the melter was full of glass made using frit 200. The specifications for frit 200 and frit 320 are shown in Attachment C. The melter was initially fed for 5 hours at a rate of ~ 50 cc/min. This was the standard rate used during previous testing and would represent normal operation during a transition between different types of feed. The rate was gradually increased until approximately one melter volume had been poured. Feed samples were taken at least once a day, usually during the transfer from the hold tank to the feed tank. Glass samples were taken at the end of the last pour each day. Feed rates up to 67 cc/min were used during this transition period. This value was subsequently reduced during the continuous feed portion of the testing. A typical analysis of the two feed batches is shown in Attachment B.

3.2 Short Term Melt Rate

The test was conducted by feeding 150 cc/min for 5 minutes and visually determining the time required to burn off the cold cap. A VCR recorded the surface for comparison to other tests. The duration of volatile generation was also recorded. The melt pool and plenum temperatures were in auto control at 1150°C and 850°C respectively. The purge air flow was zero and the dilution flow was 160 standard liters per minute. The burn off was determined visually by the absence of any remaining feed on the surface. There is a residual texture to the surface for a long period after feeding, but this was not considered during this test. The feed used during this testing had a 46.9 wt % solids which is 0.5% lower than that used in the previous run with frit 200. When all the feed was introduced, there was nearly complete cold cap coverage.

The volatile concentrations were measured using the gas chromatographs (GC). The sample point selected was directly after the addition of the dilution air. Due to low concentrations and short sample times the absolute value of the reading is probably not accurate. Durations were counted for the period that the concentration was above the background value. The duration for both the volatile concentration and visual observation are shown in Table 1 below. The average feed rate is based on the data recording system.

Table 1 -Volatile Generation During Short Term Melt Rate Test

Test #	Feed Rate cc/min	NO Duration (min)	CO2 Duration (min)	Visual Cold Cap Burn Off(min)
1	147	9	12	18
2	144	0	16	24
3	150	4	21	27
Average	147	4.3	16.3	23
Previous Frit 200 Run Average	142	12	8.3	19.3

Another observation recorded during the testing was the time required for each volatile component to be detected. Since a water flush is required prior to each feed initiation, there may be a spike in a plot of feed rate vs. time. This can also vary depending on the timing of the flush vs. the one minute frequency of data collection. For purposes of this report, the feed initiation was considered to start at the first of 5 consecutive readings in the range of the set point for feed rate. Table 2 shows the time delay between the initiation of feed and the detection of the volatile component.

Table 2 - Volatile Initiation During Short Term Melt Rate Test

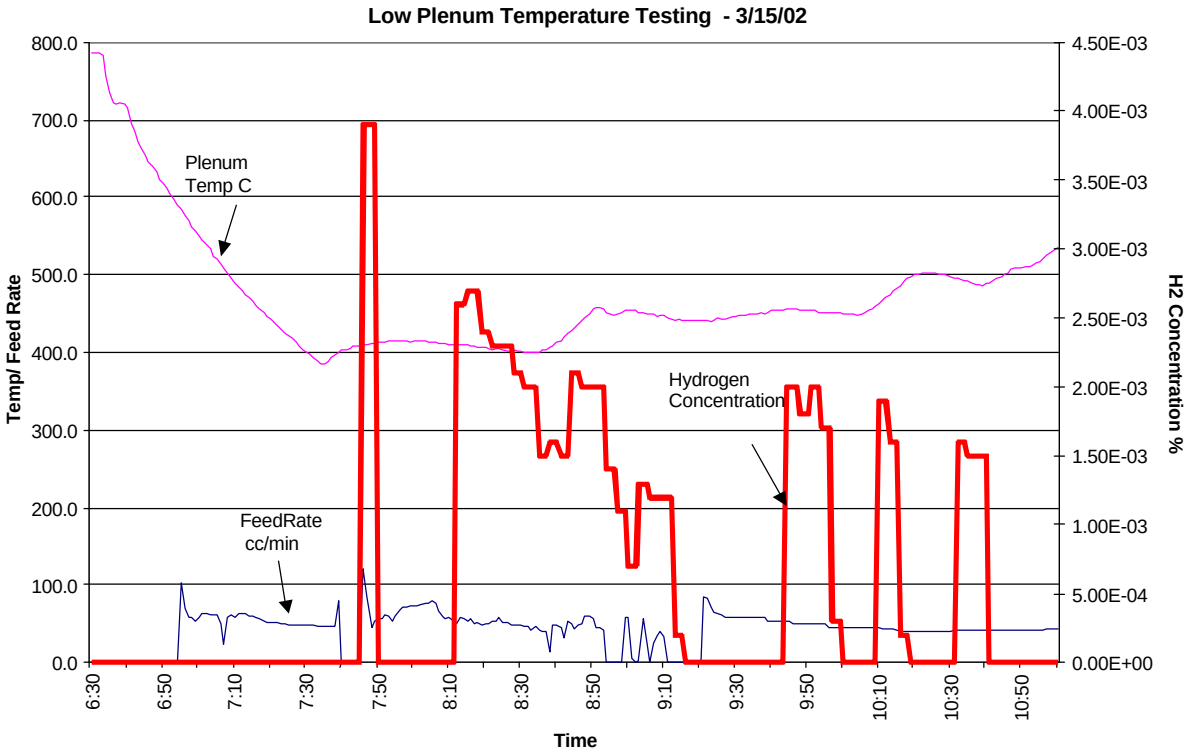
Test #	NO Initiation (min)	CO2 Initiation (min)
1	8	5
2	na	6
3	15	6
Average	11.5	5.7
Previous Frit 200 Run Avg.	2	8

The results of the short term testing are difficult to interpret due to problems detecting volatile components during short sample times. The subjective nature of visual observation also makes comparisons difficult. This test appears to be of limited value and will probably be modified or omitted from future runs.

3.3 Low Plenum Temperature

This test was performed in an attempt to gather data for flammability calculation verification. The plenum temperature during normal operation is 800-850° C. There is no hydrogen predicted or detected at these elevated temperatures. Verification of the model requires measurements in the temperature ranges that have predicted hydrogen concentrations. Excess air must be introduced into the plenum to obtain the desired temperatures. This was initially accomplished by removing the sight glass and later by adjusting the melter purge air. After feeding for approximately 30 minutes at 400 °C, the plenum temperature was raised to ~ 450 °C by energizing the lid heaters. Off-gas data was collected for approximately 30 minutes before raising the plenum temperature to 500 °C. A feed rate of ~ 43 cc/min was maintained while the plenum was operating at that temperature. Several problems with the feed system were encountered during the testing. Replacement of the peristaltic pump hose corrected most of the difficulty. A plot of hydrogen generation versus plenum temperature is shown in Figure 1**.** Based on the information during this testing, the hydrogen generated at the three temperatures is similar. The verification of the offgas model will be covered under a separate report.

Figure 1



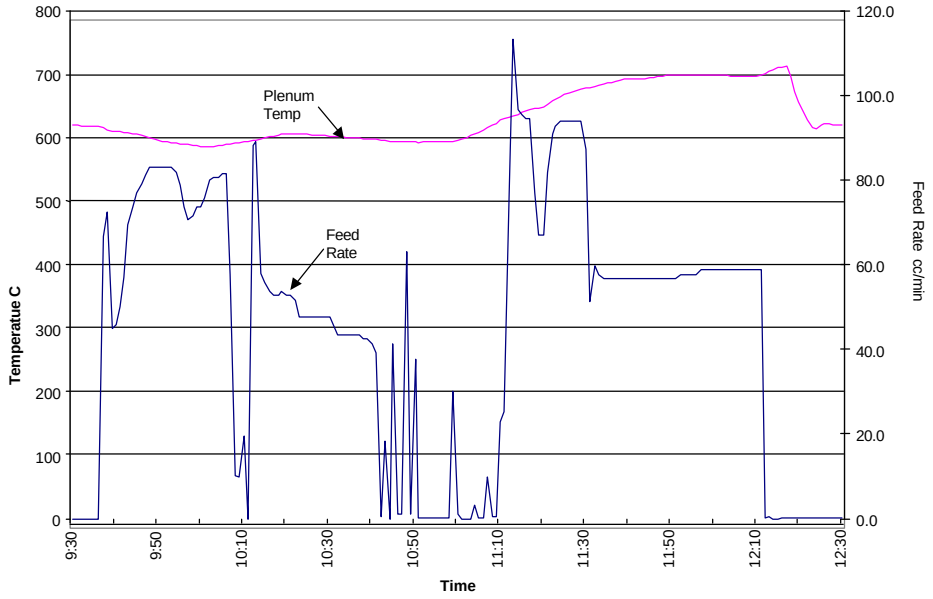
3.4 Melt Rate vs. Plenum Temperature

In addition to the low temperature plenum testing described above, two additional runs were made to determine relative melt rate at different temperatures. The plenum temperature was initially lowered to 600 °C by adding purge air. Once the target temperature was reached, the lid heaters were energized with a set point of 600 °C. The feed rate was initially high to build up the cold cap. The goal during the testing was to maintain a consistent cold cap coverage of ~90%. This is a subjective measurement but generally the presence of several vent holes or a small area of glass constitutes normal coverage. Once steady state was obtained, the plenum temperature was raised to 700 °C and the test was repeated.

The testing indicated that a steady rate of ~ 44 cc/min could be maintained with a plenum temperature of 600°C and the glass pool in automatic control with a set point of 1150 °C. The increase to a 700°C plenum temperature yielded an estimated steady state feed rate of ~58 cc/min.

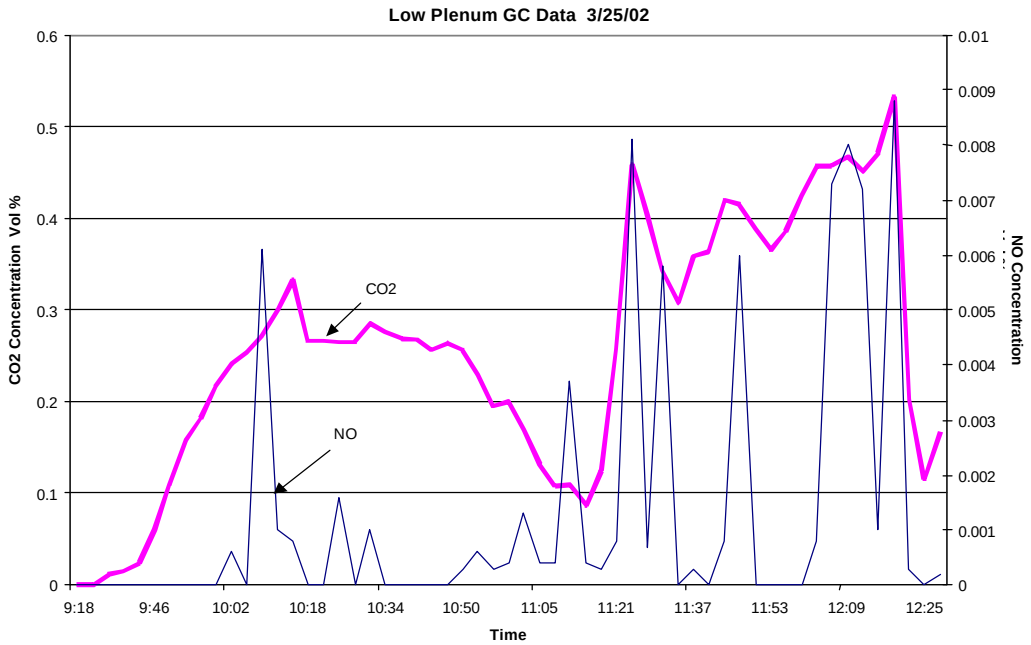
The feed rate vs. plenum temperature for this test is shown in Figure 2. There were feed problems experienced during the testing, especially at the lower temperature. Replacing a pump hose seemed to solve the problem.

Figure 2



The offgas conditions were monitored during this testing. There were only a couple of very small hydrogen peaks observed during the three hours of testing. A plot of the CO2 and NO concentrations is shown in Figure 3.

Figure 3



3.5 Extended Melt Rate

Continuous operation was started by initially feeding with a melter pressure of -4 inwc. Feeding continued until glass began to drip from the pour spout. The pressure was changed to + 5 inwc to initiate a steady stream. The set point was then placed at -0.5 inwc and feeding continued for 6 more hours. No problems were observed using the new feed. The cold cap was maintained with similar coverage in both runs. The melter was capable of sustaining a feed rate of ~64 cc/min using the feed made with frit 320. This is an improvement over the last campaign using frit 200, where a maximum sustainable feed rate was ~ 54 cc/min. Similar feed rates for the frit 320 were also observed during the melter turnover portion of the test. Operating conditions for both runs are shown in Table 3. The glass pool and plenum temperatures were maintained in automatic mode for both tests. Due to an error in the previous report, the plenum temperature for the frit 320 test was held at 850° C rather than 800°C. This is not believed to have a major effect on the melt rate comparison. This is based on the decreasing slope in melt rate when plotted against increasing plenum temperature. A rate of 63 cc/min at 800°C is estimated when graphing the rates at different temperatures.

Table 3 - Melt Rate Conditions

Variable	Unit	Frit 320	Frit 200
Feed Rate	cc/min	64	55
Feed Density	g/cc	1.21	1.21
Wt % Solids	%	47	46.1
Calcine Ratio		.92	.915
Glass Melt Rate	lb/hr	4.4	3.7
Glass Temperature	°C	1150	1150
Plenum Temperature	°C	850	800
Feed Duration	hr	6	5

3.6 Actual vs. Indicated Plenum Temperature

The actual gas temperature in a melter can differ from the measured temperature for a variety of reasons. The configuration of the thermowell and shine from the glass are two factors. Studies have been conducted to calculate the difference in several melters. Data gathered during a variety of plenum temperatures and feed conditions will allow an estimate of this parameter in the minimelter. Examples of the data to be collected are summarized in Table 4. The actual raw data collected during this testing will be used to calculate the temperature difference. Several data points to complete the table are not available at this time and will be supplied as they become available. This information will be used in off- gas modeling and will be covered in a separate report.

Table 4 - Plenum Temperature Conditions

Date-Start/ Finish	Plenum Temp 3A(°C)	Off-gas Temp T103(°C)	Off-gas Flow scfm	Purge Air slm	Dilution Air slm	CO2 flow #Mole/min	Feed Rate cc/min	Feed % Solids	Feed Density g/cc *	Formate mg/l
3/14 -11:30/17:30	850.1	127.5	9.4	0	161	5.0 E-2	64.2	46.9	1.35	20400
3/15 - 9:25/10:05	450.7	135.8	12.1	150	28	2.9 E-2	52.7	46.9	1.35	20400
3/25 - 11:30/12:10	694.7	155.5	7.6	108	30	3.8 E -2	57.7	46.1	1.35	22900
3/13 – 15:27/20:20	849.8	108.7	7.5	0	161	4.7 E -2	65.2	46.9	1.35	21000

* Density is estimated from weight and displacement in SRAT and hold tank

3.7 Air Flow Measurement

The off-gas flow was calculated using a helium tracer gas that was detected by the gas chromatograph. A known amount of helium was introduced into the off-gas line prior to the sample port. The helium concentration was measured and the total flow calculated. The known air supplies to the off-gas line prior to the sample port are the melter purge and the dilution air. Testing prior to feeding allowed the inleakage to be estimated since it would be the only other source of air not being intentionally supplied. MKS flow meters were calibrated and installed to measure the purge and dilution air flows. Since the flow meters were calibrated with equipment certified at STP, a correction factor had to be used. The helium flow meter was checked using water displacement in a graduated flask. Actual helium flow was higher than indicated and a correction factor was added in the calculation. The correction factors used are shown in Attachment E. Testing after the run indicates that there is approximately a 2 scfm inleakage in the melter.

3.8 Process Parameters

Data collection occurred automatically during testing. Additional information was recorded in the laboratory notebook, including setpoint and output changes. Table 5 represents average values during normal and idle operation for both frits. The idle parameters were taken from a 24 hour period several days after the completion of testing for each frit. The feeding parameters were taken during the extended melt rate portion of each test. The power settings required during idle and feeding periods indicate a difference in electrical resistivity between the two glasses. Glass made with frit 320 exhibits a lower resistance. Samples have been submitted to PNNL for electrical resistivity measurements.

Table 5 - Average Melter Parameters

Variable	Unit	Frit 200 Idle	Frit 320 Idle	Frit 200 Operating	Frit 320 Operating
Electrode	Kw	3.9	4.6	3.9	5.1
Electrode	Amp	141.5	174.7	143.6	186.3
Electrode	Volt	29.4	25.2	27.2	25
Glass Pool	° C	1158	1141	1150	1150
Lid Heater	Kw	NA	NA	6.1	5.9
Lid Heater	Amp	NA	NA	76.1	76.6
Lid Heater	Volt	NA	NA	79.5	79.9
Plenum Temp	° C	780	804	800	850
Feed Rate	cc/min	NA	NA	55	64
Date of Data		7/30/01	4/02/02	7/26/01	3/14/02
Duration	Hours	24	24	5	6

3.9 Material Balance

In order to confirm the data collected during the run, a material balance was performed at several conditions. Calculations for two conditions are shown below. A feed slurry density of 1.4 was assumed for both all calculations.

CO_2 Balance
Test date 3/13 from 15:27 To 20:24

CO_2 generation from feed:

$$\frac{65.2\text{ml}}{\text{min}} \times \frac{1.4\text{ g}}{\text{ml}} \times \frac{21000\text{ mg COOH}}{\text{kg total}} \times \frac{1\text{ kg}}{1000\text{g}} \times \frac{1\text{mol CO2}}{45\text{ g}} \times \frac{\text{g}}{1000\text{mg}} = \frac{4.2\text{ E-2 mole}}{\text{min}}$$

CO_2 detected in off-gas:

$$\frac{0.005034\text{ mol CO}_2}{\text{mol off-gas}} \times \frac{7.5\text{ scf off-gas}}{\text{min}} \times \frac{\text{lbmol}}{359\text{ scf}} \times \frac{453.6\text{ mol}}{\text{lb mol}} = \frac{4.7\text{ E-2 mole}}{\text{min}}$$

CO_2 Balance:
Test date 3/14 from 11:30 to 17:30

CO_2 generation from feed:

$$\frac{64.2\text{ ml}}{\text{min}} \times \frac{1.4\text{ g}}{\text{ml}} \times \frac{20500\text{ mg COOH}}{\text{kg total}} \times \frac{1\text{kg}}{1000\text{g}} \times \frac{\text{mol CO2}}{45\text{ g}} \times \frac{\text{g}}{1000\text{mg}} = \frac{4.0\text{ E-2 mol}}{\text{min}}$$

CO_2 detected in off-gas:

$$\frac{0.00426\text{ mol CO}_2}{\text{mol off-gas}} \times \frac{9.4\text{ scf off-gas}}{\text{min}} \times \frac{\text{lbmol}}{359\text{ scf}} \times \frac{453.6\text{ mol}}{\text{lb mol}} = \frac{5.0\text{ E-2 mole}}{\text{min}}$$

3.10 Predicted Properties

The analyses from 4 pour samples of the run were used to predict the properties of the glass. The results indicate that the estimated values for liquidus, viscosity, homogeneity, and durabilty all fall within the accepted ranges using the PAR criteria. Both the current and new liquidus models were used. The increasing sample number indicates transition between frit 200 and frit 320 glass. Attachment D shows the sample log kept during the run. The results for all properties are shown in Attachment F for reference. The values predicted by the new liquidus model indicate that higher sludge loading could be achieved with frit 320 and still stay within the limits. The new model incorporates sodium, lithium and potassium into the calculation. The effect of this can be seen in the relationship between Σalkali and liquidus. The new model is described in WSRC- TR-2001 -00520 and the limits for the parameters are listed in WSRC-TR-1995-00364, Rev 3.

3.11 General Observations

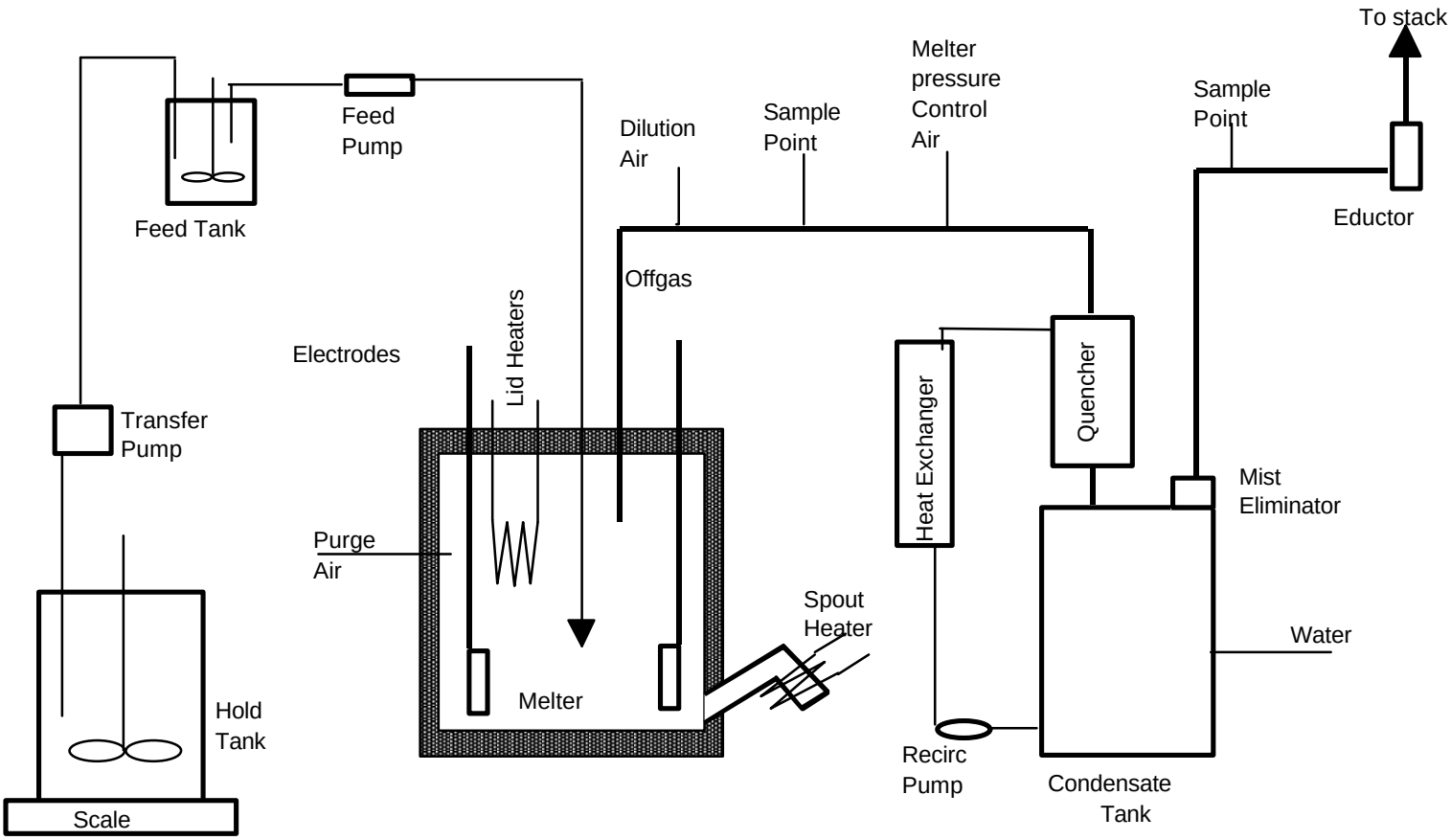
The use of the new feed tube during the testing greatly improved the reliability of the system. Long runs could be made without the need to rod the feed tube. Continuous pouring also allowed the operation of the melter with very few high glass pool level alarms. Operation for long periods at low plenum temperature still continues to be a problem. Two water leaks were discovered during the run. One was on the #1 electrode clamp, which required the water to be shut off during most of the testing. Occasionally during the high feed rate test, the water was turned on briefly to bring the clamp temperature below the alarm point. There was a second leak in the rotometer supplying the feed tube flush water. This valve had to be opened manually prior to and after each feed initiation or stoppage. The rotometer can be replaced during an idle period, but the clamp leak will require a melter outage.

4.0 CONCLUSIONS

The results indicate that glass made with frit 320 melts at a rate ~ 20% faster than that using frit 200. This is in line with results from small scale testing. The run also showed that there were no operational problems associated with processing the frit 320. The glass produced has predicted properties within the acceptable range. A lower electrical resistance is verified by the power settings on the melter during both idle and feeding conditions.

Attachment A

786-A Minimelter System



Attachment B – Simulated Feed Composition

Oxide Wt %	**Frit 200 Feed Sample MMF009**	**Frit 320 Feed Sample MMF024**
Al	2.77	2.67
B	2.66	1.87
Ba	0.092	0.084
Ca	0.924	0.688
Cr	0.924	0.083
Cu	0.046	0.030
Fe	7.85	8.61
K	0.143	<0.010
Li	1.56	2.73
Mg	0.915	0.026
Mn	0.866	0.828
Na	8.33	9.22
Ni	0.433	0.469
P	0.035	0.029
Pb	0.062	0.058
Pd	0.011	<0.010
Rh	0.006	<0.010
Ru	0.012	0.017
Si	23.1	25.9
Zn	0.126	-
Zr	0.202	0.165

Attachment C - Frit Specification

Component	Frit 200 (wt %)	Frit 320 (wt %)
B2O3	12	8
Li2O	5	8
Na2O	11	12
SiO2	70	72
MgO	2	0
Total	100	100

Attachment D - Sample Log

MMF 021	3/13/02	Macrobatch 3 feed simulant with frit 320 -collected at feed tank
MMG 022	3/13/02	Glass pour sample after one melter turnover
MMF 023	3/14/02	Macrobatch 3 feed simulant collected at feed tank
MMF 024	3/14/02	Transfer to feed tank after transfer from drum to hold tank
MMG 025	3/14/02	Power pour at end of continuous testing
MMG 026	3/20/02	Frit 304 preshipment sample
MMG 027	3/20/02	Frit 320 final preshipment sample
MMG 028	3/20/02	Glass from bucket under spout after compeltion of testing(3/15/02)- drips
MMF 029	3/25/02	Feed during transfer from hold to feed tank
MMF 030	3/25/02	Upper feed tank during 600 degree plenum test - thick
MMG 031	3/25/02	Pour sample at end of run

Attachment E

Corrections To 786-A Melter Air Purge and Helium Tracer Flows

Correction to He tracer flow, based on volume per time data taken.

500 ml of He was collected in 84 seconds.

The flowmeter read 295 ml/min. The temperature of the He was approximately 29.4 °C. The correction factor *f* is then:

$$f = \frac{\frac{500\,ml}{84\,\text{sec}} \times \frac{60\,\text{sec}}{\text{min}} \times \frac{273.16\,K}{273.16 + 29.4\,K}}{295\,ml/\text{min}} = \mathbf{1.093}$$

The resulting flow reading for the He tracer is then in standard ml/min, where standard conditions are 1 atm and 0 °C.

The MKS flowmeters for measuring the Melter Purge Air and Dilution Air were calibrated with 29.92 inHg (1 atm) and 70 °F as the standard conditions. The correction factor *k* is then:

$$k = \frac{273.16\,\text{K}}{273.16 + 21.11\,\text{K}} = \mathbf{0.9283}$$

The resulting flow readings for the air purges are then in standard L/min (slpm), where standard conditions are 1 atm and 0 °C. To convert to scfm, divide by 28.316847.

Example using data collected on 7/19/01:

Dilution Air flowrate reading ~6.0 cfm
Melter Air purge flowrate reading = 0
He tracer flowrate reading = 295 ml/min
He concentration in offgas from gas chromatograph = 0.2 ± 0.05 vol%
Melter pressure > 0 inwc (no air inleakage)

Corrected Dilution Air flowrate = **5.57** scfm

Corrected He tracer flowrate = 322.4 ml/min

$$\text{Calculated Offgas Flowrate} = \frac{322.4\ \text{ml/min}}{\frac{0.20\ \text{vol\%}}{100}} \times \frac{\text{L}}{1000\ \text{ml}} \times \frac{\text{ft}^3}{28.316847\ \text{L}} = \mathbf{5.69}\,\text{scfm}$$

Attachment F – Predicted Glass Properties

Sample ID	**Viscosity** (Poise)	**Homogeneity**	**Pred NL*** [B(g/L)]
MMG 020	92.90	232.9	0.375
MMG 022	71.47	232.2	0.577
MMG 025	62.09	232.0	0.686
MMG 028	56.08	230.9	0.832

***** Predicted PCT

.

Sample ID	**Current Liquidus**	**New Liquidus**	**Al2O3**	**SAlkali**
	(°C)	(°C)	(mass fraction)	(mass fraction)
MMG 020	990.5	1003.8	0.0614	0.1546
MMG 022	987.6	919.6	0.0559	0.1696
MMG 025	990.6	900.2	0.0529	0.1753
MMG 028	988.7	907.5	0.0514	0.1809