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PRODUCTION OF SIMULATED SLUDGE BATCH 2 MELTER FEED CONTAINING FRIT 320 IN THE 1/240TH GLASS FEED PREPARATION SYSTEM (U)

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EXECUTIVE SUMMARY

This report was prepared to fulfill a deliverable for HLW/DWPF/TTR-2002-00008, Rev. 0, “Glass Feed Preparation System (GFPS) Runs for Minimelter Feed Preparation with Frit 320 (U)”, 1/10/2002. Approximately 50 gallons of Sludge Batch 2 simulated melter feed containing Frit 320 were produced from 58 gallons of a simulated Sludge Batch 2 slurry. Two three-day runs of 29 gallons feed each were required. Frit 320 produced by Specialty Glass was used in both runs. The final melter feed product was about 47 wt. % total solids. The goal was to be close to, but greater than, 46.5 wt. %.

Significant findings include:

- There were no operational concerns noted that were directly attributable to Frit 320 during the GFPS campaign.
- Frit 320 did not appear to promote foaming, however a change in antifoam source during the two runs did seem to indicate that the more recent DeBourg antifoam obtained from the DWPF used in the second run was inferior to the older 747 antifoam prepared at IIT in 9/00.
- Planned rheological measurements were complicated by the presence of trapped air bubbles. Frit 320 SME products appear to be similar rheologically to Frit 200 SME products for Sludge Batch 2. Trapped air bubbles were also a problem with Frit 200 melter feed rheology samples.
- SME product waste loading and frit loading were as targeted. They matched the results of the GFPS runs with Frit 200 and Sludge Batch 2.
- SME product at 48-49 wt. % total solids was very difficult to mix with the 4-inch impeller, side-mounted, drum mixer at 2000 rpm, whereas 46 wt. % was readily mixed.
- Hydrogen generation rates were broadly comparable to the baseline bench-scale tests of 2000 for Sludge Batch 2 at 125% acid stoichiometry (identical noble metal concentrations). Carbon dioxide generation rates were also similar to the bench-scale baseline run.
- A comparison between the two SRAT boiling sequences “concentrate-reflux” and “reflux-concentrate” was made to look for variations in mercury stripping and recovery. No significant difference was detected, but the results are not conclusive.
- SRAT and SME product samples were checked for density. Shaking the sample bottles produced results that paralleled DWPF experience since starting Sludge Batch 2. Subsequent measurements of SRAT product density ran about 0.9 g/ml and SME product density ran about 1.2 g/ml due to the presence of trapped air. Estimated densities from GFPS vessel volumes and cumulative mass additions were roughly 1.13 g/ml for the SRAT product and 1.42 g/ml for the SME product. (The GFPS agitator does not create a vortex during sludge slurry processing.)

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INTRODUCTION

The Defense Waste Processing Facility (DWPF) is planning to switch from Frit 200 to an alternative frit, Frit 320, in an effort to increase the melt rate in the DWPF waste vitrification melter. Lab-scale testing has indicated that Frit 320 will increase melt rate. A pilot-scale demonstration of the processing characteristics of simulated melter feed containing Frit 320 was requested prior to implementing the change in DWPF. This report summarizes the first phase of the pilot-scale testing which included preparation of simulated melter feed using the 1/240th-scale Glass Feed Preparation System (GFPS) to perform a prototypical process simulation of the DWPF Sludge Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) process steps.

This report was prepared to fulfill a deliverable for HLW/DWPF/TTR-2002-00008, Rev. 0, “Glass Feed Preparation System (GFPS) Runs for Minimelter Feed Preparation with Frit 320 (U)”, 1/10/2002. Other deliverables included a Task Technical/Quality Assurance Plan reviewed and approved by the customer[4], an Analytical Study Plan[5], a detailed schedule (which was included in the TT/QAP[4]), frequent updates to WD Engineering once processing had begun, and the preparation of 45-50 gallons of simulated melter feed containing Frit 320 to be tested in the Joule-heated 786-A Minimelter.

EXPERIMENTAL APPROACH

Two runs of the GFPS were needed to produce any amount over 25 gallons of melter feed simulant. The Minimelter team requested 45-50 gallons for their tests. GFPS run conditions were designed to match those used during the two previous runs of the GFPS with Sludge Batch 2 simulant waste and Frit 200. Results of those two runs were documented by Koopman[3]. This included a description of the preparation of the Sludge Batch 2 simulated feeds for those and for future tests. These were blends of Tank 8 simulant and Tank 40 simulant (Tank 40 prior to the addition of Tank 8) prepared at the University of South Carolina-Columbia in the Spring of 2000.

The first GFPS run in the Frit 320 campaign, Frit 320 Run 1, used the sludge described in Koopman[3], Table 1, as “GFPS-G”. The second GFPS run, Frit 320 Run 2, used the sludge described in Koopman[3] as “GFPS-F”. The two Frit 200 run used drums “GFPS-C” and “GFPS-D” in Koopman[3]. The Sludge Batch 2 – Frit 200 melter feed simulants were used by the Minimelter team to obtain a melt rate data baseline. The Sludge Batch 2 blend originally made in drum “GFPS-F” in Koopman[3] was transferred into the drum labeled “GFPS-C” in 2001, i.e. after the two Frit 200 runs. Consequently “Drum C” appears in the Run Plan[7] for Frit 320 Run 2, but the contents are as indicated above.

The analytical data for the above drums were used to prepare the initial run plan[6] corresponding to the nominal bench-scale run at TNX during the Sludge Batch 2 Flowsheet Study[1]. A few small adjustments were made to the run plan compared to the

Frit 200 run plans due to the objectives laid out in the technical task request[10]. These changes can be summarized as follows:

- Add 100 ppm antifoam between nitric acid and formic acid addition to conform to the Shielded Cells tests with real Sludge Batch 2 waste.
- Substitute Frit 320 for Frit 200 in the SME cycle on a pound per pound basis.
- Monitor the SME cycle for unusual foaming behavior that might relate to the presence of Frit 320.
- Monitor the SMECT following the SRAT cycle for mercury.
- Sample the SME product for rheological comparison to Frit 200 melter feed results obtained in Koopman[2].

There was one further change made solely in Frit 320 Run 2. The order of concentration and reflux during the SRAT cycle was reversed for Frit 320 Run 2 per the TTR[10]. Thus, in Frit 320 Run 2, the SRAT was refluxed for about 10.5 hours, and then concentrated to the target SRAT product solids content during the last 1.5 hours at boiling. The SRAT feed mercury content for Sludge Batch 2 already meets the DWPF SRAT product mercury specification. Consequently, this processing variation was not anticipated to have any significant impact on the final SME product. This was seen as an opportunity to make a preliminary attempt to discern whether the order of concentration-reflux affected the mercury content of the SRAT product and condensate. To support this goal, both the mercury water wash tank (MWWT) and the slurry mix evaporator condensate tank (SMECT) were purged and filled with fresh process water prior to the start of each run.

New batches of ~50% nitric acid and ~90% formic acid were prepared for the two GFPS runs. These were titrated for acid strength at the Aiken County Technical Laboratory facility, 999-1W. These results were input into the run plan acid calculations. SRAT and SME nitric acid and formic acid addition masses were prepared at 773-A and transferred to the GFPS in 786-A. Frit 320 was weighed and transferred to 786-A per the run plan calculation. Acid batching was at 125% of stoichiometry per the bench-scale baseline run made in 2000[1]. The glass property acceptability calculations were made by Kevin Brown prior to running the process simulations. No problems were indicated. The results can be found in Appendix A.

Run Chronology

GFPS Feed Drums G and C had been stored outside 786-A (Thermal Fluids Lab). GFPS Feed Drum G was moved into 786-A first. The contents of Drum G were transferred into the GFPS SRAT on 1/13/02. The net transferred drum sludge was determined by difference to be 124.65 kg. The SRAT contents were trimmed with mercury oxide and noble metals per the run plan to match the concentrations in the nominal Sludge Batch 2

Flowsheet study run, SB2-1, in Koopman[1]. A somewhat larger amount of flush water was used than in the first Sludge Batch 2 GFPS run with Frit 200. This was done to keep the SRAT/SME slurries more fluid, since the first Frit 200 GFPS run was very hard to mix during processing. This was due to the high solids content throughout combined with the higher apparent viscosity compared to Sludge Batch 1B simulant.

Both Frit 320 runs were processed more like the second Frit 200 run in that they carried about 1-4 extra gallons of water until the final concentration of the SME product. The advantages of wetter processing were summarized in Koopman[3]. The addition of trim chemicals, flush water, etc. brought the SRAT mass to 138.57 kg at a corrected volume of 125.2 liters. This gave a crude specific gravity of 1.107. Once the sludge had settled, it was observed that the supernate had a green color (transparent, but green). This disappeared after a few days, and the supernate took on the normal pale yellow color observed in the past.

SRAT processing for the Frit 320 Run 1 proceeded on 2/25/02 with heat-up to 93°C, nitric acid addition, and formic acid addition. Details of the nominal 125% acid stoichiometry are given in Koopman[6]. The sludge was very viscous during acid addition. The vessel mixer was supplemented by the recirculation pump throughout acid addition to increase turnover in the SRAT contents. Mixing was suspect at the thermocouple measuring SRAT liquid temperature. When the sludge thickens, a stagnant zone seems to form near the thermocouple. Steam was controlled manually during acid addition to avoid a repeat of the accidental boiling episode that occurred during acid addition in the second Frit 200 Sludge Batch 2 run. Following acid addition the SRAT was taken to boiling. The SRAT was boiled for slightly over twelve hours total. Concentration occurred first and lasted 2.3 hours. Reflux followed and lasted 9.8 hours. The boiling period was split over two days (2/25-2/26). There were approximately 120 kg of SRAT product in a little over 106 liters for a crude specific gravity of 1.13.

After completion of the SRAT cycle, an abbreviated SME cycle was performed on 2/26-2/27/02. The cycle was abbreviated in the sense that only about three hours of boiling followed each of the two frit additions, and there was no analog to decontamination frit additions and concentrations performed in DWPF. Frit 320 (22.85 kg) was added with 8-liters of flush water plus 481 g at 87.17 wt.% formic acid. The flush water was used to rinse down the SME vessel walls in order to see inside well enough to perform a test of foaminess (under reflux). The flush water was then boiled off. Frit 320 was added a second time with flush water and formic acid (same amounts as in the first addition and flush water was used to rinse the walls again). Foaminess was evaluated a second time. The slurry was then concentrated to about ten kg less than the ultimate target wt. % total solids content of 46.5 wt. % based on a mass balance model. The SME product was approximately 126 kg in about 89 liters for a crude specific gravity of 1.42. The SME product was transferred into a drum. Ten kg of water were then used to flush the SME vessel solids still trapped in the vessel into the drum. Not all solids held on the coils were recovered. About three kg of solids (wet basis) were estimated to be in the vessel. They were allowed to remain there for the second run.

GFPS Feed Drum C was moved into 786-A for Frit 320 Run 2. Its contents were transferred into the GFPS SRAT on 2/28/02. The transferred sludge mass was determined to be 123.59 kg by difference. The SRAT contents were trimmed with identical quantities of mercury and noble metals compared to the Frit 320 Run 1. A slightly adjusted run plan was issued[7], to reflect the altered sequence of concentration and reflux during the SRAT cycle. (The addition of trim chemicals, flush water, etc. brought the SRAT mass to 132.74 kg at a corrected volume of 121.7 liters giving a crude specific gravity of 1.091. This does not take into account the solids left over from the Frit 320 Run 1. About 2-3 kg of left-over material would bring the specific gravity up to that of the first run.) Initiation of the run was attempted on 3/4/02, but gas chromatograph (GC) problems prevented it. The run started 3/5/02. SRAT refluxing preceded concentration during the second run and lasted about 10.5 hours. Refluxing was split over two days (3/5-3/6/02). The SRAT was then concentrated for about 1.5 hours before cooling down. The first day of the SRAT cycle went more smoothly than in Frit 320 Run 1, so more of the SRAT cycle was completed during the first day (13.5 hour days).

SRAT product samples were taken after completion of the SRAT cycle on 3/6/02. Final SRAT product was roughly 122 kg in 107 liters for a crude specific gravity of 1.14. Processing proceeded immediately to the SME cycle. The SME cycle was completed on 3/7/02. A final SME volume about 10 liters larger than in the Frit 320 Run 1 was targeted and met, but the SME product sample was higher in total solids than in the first run. This was apparently due to the solids left over from the first run. Final SME mass was estimated to be 136 kg in roughly 96 liters for a crude specific gravity of 1.42. Other SME cycle testing duplicated what was done in Frit 320 Run 1 (foam tests, off-gas monitoring). Frit 320 Run 2 was successfully completed on 3/7/02. Drum work, vessel rinsing, and sampling activities continued into 3/8/02.

Off-gas composition was monitored by GC during both runs. A regulated helium flow was added to the SRAT/SME as an internal standard. GC calibration was checked before and after each run. The sampling pump of the GC used in Frit 320 Run 1 failed completely during the loading and trimming of the second sludge batch. A second GC was prepared and then used for Frit 320 Run 2. The sensitivity of the second GC to low concentrations of CO_2 and N_2O appeared to be better than that of the first GC. This may be sampling pump related. All raw GC scans were saved on two different computer hard disks. This was fortunate, since there was a data registration problem between the laptop GC controller and the GFPS process controller. As a result, the CO_2 and N_2O data from Frit 320 Run 1 did not get properly stored into the run data file. This data can be recovered at a later date, if desired, from the raw GC scans. The GC data registration issue was caused by a hard drive failure on the original laptop GC controller used for GFPS runs. The GC method had to be reconstructed from scratch, and this one small point was overlooked. This was caught between runs and corrected prior to the second run.

Antifoam was added prototypically during acid addition and before going to boiling. This entailed a 200 ppm addition prior to nitric acid addition, a 100 ppm addition prior to formic acid addition, and a 500 ppm addition prior to boiling. Antifoam mass was adjusted for %-effectiveness, which was taken as 75% for the two 747 antifoams used during testing. A basis of 125 kg was used for the slurry when determining antifoam addition mass. So 100 ppm was 12.5 grams of neat antifoam before adjusting for %-effectiveness, and was 16.67 grams of neat antifoam after adjusting for %-effectiveness. Antifoam was diluted to a net 1:10 solution in water on a 100% effective basis. Antifoam solution was flushed in with an equivalent mass of water, i.e. a 100 ppm addition included 125 grams of 1:10 aqueous solution containing 16.67 g of neat 75% effective antifoam and 125 grams of flush water.

Frit 320 Run 1 was completed with IIT 747 antifoam at 75% activity prepared by Alex Nikolov at IIT and dated 9/25/2000. There was very little left after the first run, but enough remained to prepare antifoam for the two additions made during nitric acid and formic acid addition in Frit 320 Run 2. The 500 ppm antifoam addition made prior to going to boiling was a composite of the 9/25/2000 antifoam (24.6%), some previously diluted to 1:10 antifoam believed to be from that lot (17%), and Lot 11949 of 747 antifoam prepared by DeBourg Corp. and dated 9/01 (58.4%). Additional antifoam added during Frit 320 Run 2 came exclusively from Lot 11949 material obtained from DWPF.

GFPS run details are documented in the same laboratory notebook[8] that was used for the two runs with Frit 200 in November and December, 2000.

Rheological Methods

A sample of melter feed from each run was measured on a Haake RS-150 Rheostress rheometer. The concentric cylinder geometry was used initially. A rotating cylinder 60 mm high with a diameter of 38.02 mm was placed inside a beaker with an internal diameter of 43.4 mm. The sample was sheared from 0 $\sec^{-1}$ to 800 $\sec^{-1}$ over a five minute period (linear rate ramp), held at 800 $\sec^{-1}$ for one minute, and then returned to 0 $\sec^{-1}$ over an additional five minute period. Some additional tests on the samples were performed in the parallel plate geometry for comparison purposes. A gap of 1.5 mm was used for SME product samples. The 60 mm diameter plates were used. Samples pulled during acid addition of the SRAT cycle of Frit 320 Run 1 were measured using a rotating cylinder 60 mm high with a diameter of 41.42 mm in the 43.4 mm diameter beaker. These samples were sheared from 0 $\sec^{-1}$ to 990 $\sec^{-1}$ over a five minute period (linear rate ramp), held at 990 $\sec^{-1}$ for one minute, and then returned to 0 $\sec^{-1}$ over an additional five minute period. All testing was done under temperature control at 25°C.

Flow curves were not corrected for non-Newtonian fluid behavior. Data were fit to the following Bingham plastic rheological equation of state using data in the mid-shear rate ranges.

$$t = t_o + h\dot{g}$$

Where: t = shear stress (dynes/cm^2)
$\dot{g}$ = shear rate, (1/seconds)

t_o = Bingham model yield stress (dynes/cm^2)
h = Bingham model consistency, or Bingham plastic viscosity, (cP)

Yield stress and consistency, or plastic viscosity, were determined by linear regressions (intercept and slope, respectively). Supporting sample pH measurements were made at room temperature with an Accumet AR50 pH meter calibrated with pH 7 and pH 10 buffer solutions.

DISCUSSION OF RESULTS

A more in-depth analysis of the GFPS data was made for the two Frit 320 runs than was attempted for the two Frit 200 runs in 2000[3]. These two Frit 320 GFPS runs were the first full process simulations of the SRAT and SME cycles for Sludge Batch 2 simulant combined with Frit 320. The discussion to follow will split the data into SRAT results followed by SME results. The discussion covers foaminess and antifoam performance, gas generation, effects of processing variations, Frit 320 analytical data, and SRAT and SME cycle product analytical results. A final section discusses the results of rheological measurements on some of the GFPS samples.

SRAT Cycle Discussion

SRAT Cycle Foaminess

SRAT processing occurred in the 14-18 wt. % total solids range. Post-run data indicate that the wt. % total solids was fairly similar between the two runs. 747 antifoam was used in both runs. Two supplies were available. One was made at the Illinois Institute of Technology (IIT) in September of 2000. The other was made by DeBourg corporation in 2001. The IIT 747 antifoam was used until it was gone. The DeBourg antifoam was used to finish Frit 320 Run 2. There was one modification made to the antifoam addition strategy followed for the Frit 200 runs. An additional 100 ppm antifoam addition was made between nitric acid and formic acid addition.

A 200 ppm antifoam addition was made prior to acid addition. Mild foaming was nevertheless observed during nitric acid addition in both runs. Long-lived bubbles were formed, but they did not occupy a large enough volume to be of concern. Individual bubbles the diameter of baseballs were observed. The new addition of 100 ppm antifoam following nitric acid addition effectively eliminated this foam. An addition of 500 ppm antifoam was made following formic acid addition while the SRAT was being heated from 93°C to boiling.

Foaminess results during SRAT processing at boiling will be reported using the following formula:

$$Volume\ expansion\ factor = \frac{Volume\ of\ slurry\ and\ foam\ at\ boiling}{Volume\ of\ non-boiling\ slurry}$$

The volume expansion factor is dimensionless. Volume expansion factors on the order of 1.05-1.10 have been common in the GFPS when there is a monolayer of bubbles due to boiling, but no stacked foam observable. This appears to simply be a small expansion of the slurry volume due to increasing temperature, perhaps coupled with the presence of bubbles coming off of the steam coils at boiling. The two previous Frit 200 runs with Sludge Batch 2 could be characterized by a volume expansion factor that was probably less than about 1.10 throughout the two SRAT cycles. The non-boiling slurry volume is typically measured at as high a temperature as possible short of boiling, to mitigate the expansion in volume associated with heating from 50°C to 99°C. Results for the SRAT cycles of Frit 320 Runs 1 and 2 are given in Table 1. Arrows denote changes with respect to time.

During the first SRAT cycle (concentration followed by reflux, and 9/00 antifoam) there were no real issues with foaming during boiling. No additional antifoam was added beyond the 500 ppm added before going to boiling until foam testing during the SME cycle two days later. This includes the second day of the SRAT cycle (2/27) at which point the antifoam had been in the process for nearly a full day (not all at temperature).

Table 1: SRAT Foaming

Frit 320 Run # and Date	Volume Expansion Factor
Reflux of Run 1, 2/26	1.10
Reflux of Run 1, 2/27	1.05-1.14
Reflux of Run 2, 3/5	1.19 → 1.13 → 1.25
Reflux of Run 2, 3/5 after +100 ppm antifoam	1.08
Reflux of Run 2, 3/6	1.08-1.11

The observed amount of foam was more than expected during the Frit 320 Run 2 SRAT cycle on the first day, 3/5, based on previous runs with Sludge Batch 2 feed. The foam subsided somewhat once the process settled down to a steady reflux (1.19→1.13). This happened over about the first hour at boiling. Then the apparent volume began expanding gradually and unexpectedly over about four hours (1.13→1.25). This was more foam than had been seen in the three previous runs with Sludge Batch 2 simulant, although it was less than had been seen in the antifoam testing work in 1999 with Dow Corning 544 antifoam.

An additional 100 ppm addition of DeBourg antifoam was made after about six hours at boiling. The stacked foam layer was essentially eliminated (1.08). A measurable, but controlled, foam did redevelop the next day, 3/6, during the completion of the SRAT cycle, but no additional antifoam was added. The boil-up flux was cycled through six different values, and the foam height was measured. The entire test lasted twenty minutes. Testing was under reflux with the SRAT recirculation pump on. The baseline

SRAT level was last observed near the 146 liter mark, but this was adjusted to the 150.26 liter mark based on an extrapolation to zero boil-up flux giving no foam (~0.9 inch shift in the baseline level).

The foam height data were then fit to the model of Pilon, Federov, and Viskanta[9], assuming constant bubble diameter:

$$H_{\infty} = 2905 \frac{s}{r_0^{2.60}} \frac{[m\,(j - j_m)^{0.80}]}{(r\ g)^{1.80}}$$

where H_∞ is foam height, σ is surface tension, μ is fluid viscosity, ρ is fluid density, g is gravitational acceleration, r_0 is the mean bubble radius, j is gas flux, and j_m is the minimum gas flux to produce a nonzero foam height.

The data taken on the GFPS were unable to determine a meaningful nonzero value of j_m. The adjustment of the SRAT level at the onset of boiling was made to obtain a zero foam height at zero boil-up while giving a dependence of $H_\infty \propto j^{0.8}$. This was deemed reasonable given the difficulty of discriminating between the mean foam height and the maximum or minimum foam height. Foam height was read the same way each time, but it is unclear what height was actually represented by the reading (mean, minimum, maximum, or something else). In addition, it was difficult to read the SRAT level just prior to boiling, because the liquid phase thermocouple often read 3-6°C below the bulk temperature until boiling has been initiated for 10-20 minutes. If the level is read too soon, then the value read is too small for the pre-boiling level.

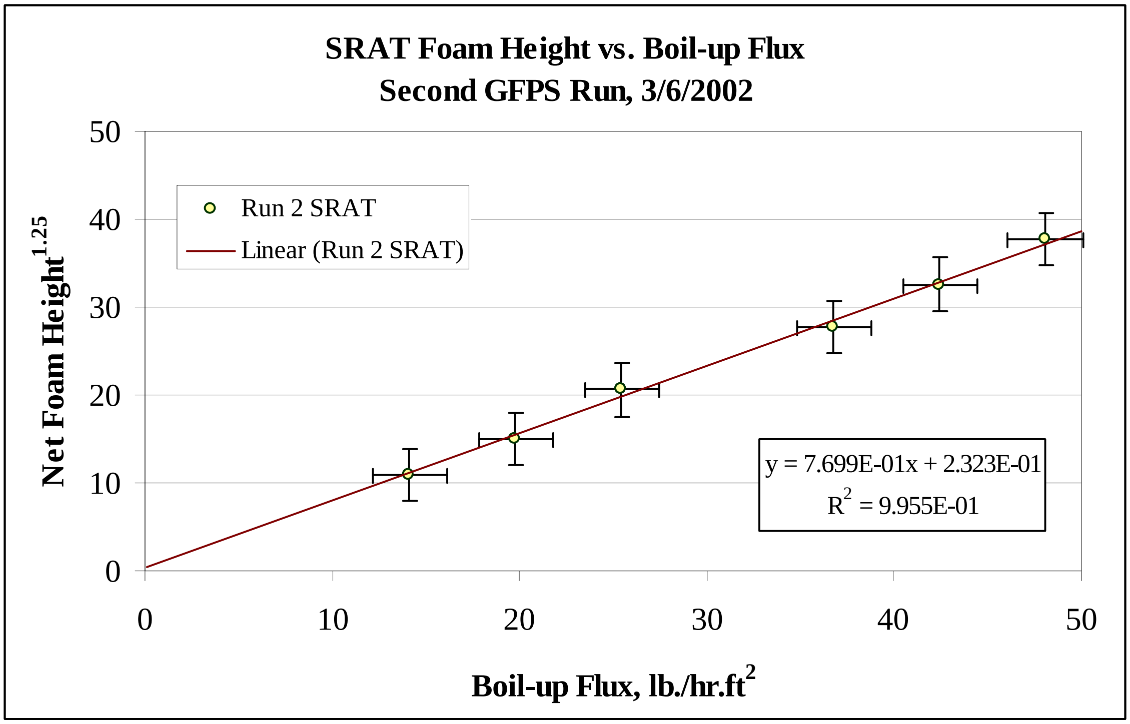
The net foam height used to fit the foam data was just the difference between the read SRAT foam level and the baseline SRAT level. The effective viscosity of the slurry was assumed to be independent of boil-up flux induced shear rate changes as a first approximation. The actual slurry viscosity is a function of position in the vessel (through the variation of shear rate with position). Table 2 gives the mean data used to prepare Figure 1. The volume expansion factor was adjusted for the adjusted SRAT non-boiling level, ~150. Net foam height is the difference between level readings. The level markings are in liters assuming no vessel internals. In the region covered by the data below, a unit difference in level reading is 0.237 inches, or 0.60 cm, on average. The boil-up flux is given in units of lb./hr.ft^2.

Table 2: SRAT Data for Foam Study

Boil-up Flux	SRAT Foam Level	Net Foam Height	Vol. Exp. Factor
14.15	157	6.7 (1.7 in.)	1.05
19.81	159	8.7 (2.1 in.)	1.06
25.46	161.5	11.2 (2.7 in.)	1.08
25.46	161.5	11.2 (2.7 in.)	1.08
36.78	164.5	14.2 (3.4 in.)	1.10
42.44	166.5	16.2 (3.9 in.)	1.11
48.10	168.5	18.2 (4.3 in.)	1.12

The maximum net foam height of 18.2 corresponds to about 4.3 inches of foam. Foam levels of 3-7.5 inches were observed with IIT 747 antifoam during SRAT processing at this flux in the Sludge Batch 1B testing. The Sludge Batch 1B testing had only 200 ppm total antifoam in the SRAT cycle, while the present tests had 800 ppm total antifoam.

Figure 1: Foaming in Second GFPS SRAT Cycle



The data in Figure 1 showed very good agreement with the model. The projected j_m value was essentially zero, but this was partially achieved by adjusting the baseline level (volume). The foam height data gave a fairly linear response, when plotted in Figure 1 raised to the 0.8^{-1}, or 1.25, power versus boil-up flux. The boil-up flux error bars were created to allow for a possible systematic bias between steam flow and boil-up flux.

They are not expected to be randomly distributed, i.e. the entire data set could be biased left or right. This helps to explain the observed linearity relative to the size of the error bars. The boil-up flux was indirectly in feedback control through the controlled steam flowrate to the SRAT coil. Most of the possible random error was in the net foam height determination.

There were five known processing differences in the SRAT cycle of Frit 320 Run 2 that could have impacted foaminess. These are:

- Reflux came before concentration.
- The antifoam supply lot changed.
- A heel from the SME cycle of Frit 320 Run 1 was present.
- Gas generation rates were higher.
- Wt. % total solids was lower.

One concern prior to testing was that Frit 320 solids could somehow impact foaming tendencies. Based on the Frit 320 Run 1 SME cycle, this did not appear to be the case. Although it can not be ruled out that frit left over from the Run 1 contributed something to the foaming, prevailing wisdom seems to be that large, frit-sized particles do not promote foaming. It would require more data to make a case that any contribution due to frit was due to the frit being Frit 320 rather than Frit 200. Measured gas generation rates were higher in the second SRAT cycle than in the first SRAT cycle, but this did not correlate with the time that the increased foaming was observed (see Figure 3 and Figure 4 below for gas generation data).

Wt. % total solids ran about 15.8% during the Frit 320 Run 2 reflux period (which was prior to concentration). Wt. % total solids ran about 17.7% during the Frit 320 Run 1 reflux period (which was after concentration). Since no foaming is expected at 0 wt. % solids or 100 wt. % solids, there must be at least one wt. % solids at which there is a maximum in foaming tendency. It is not known precisely where this point is relative to the two operating conditions just given. There is some evidence that a point of maximum foaming tendency lies between 12 and 24 wt. % total solids. There is insufficient data to rule out the possibility that the second run was operated closer to a point of maximum foaming tendency than the first run.

The tentative conclusion is that the foaming tendency of Frit 320 Run 2 was due to a somewhat inferior antifoam preparation. Similar problems with foam control were noted in recent runs of the 22-liter SRAT at the Aiken County Technical Laboratory (ACTL). At least part of the ACTL foaming problem seems to have been due to antifoam mixing issues, rather than to lower quality antifoam.

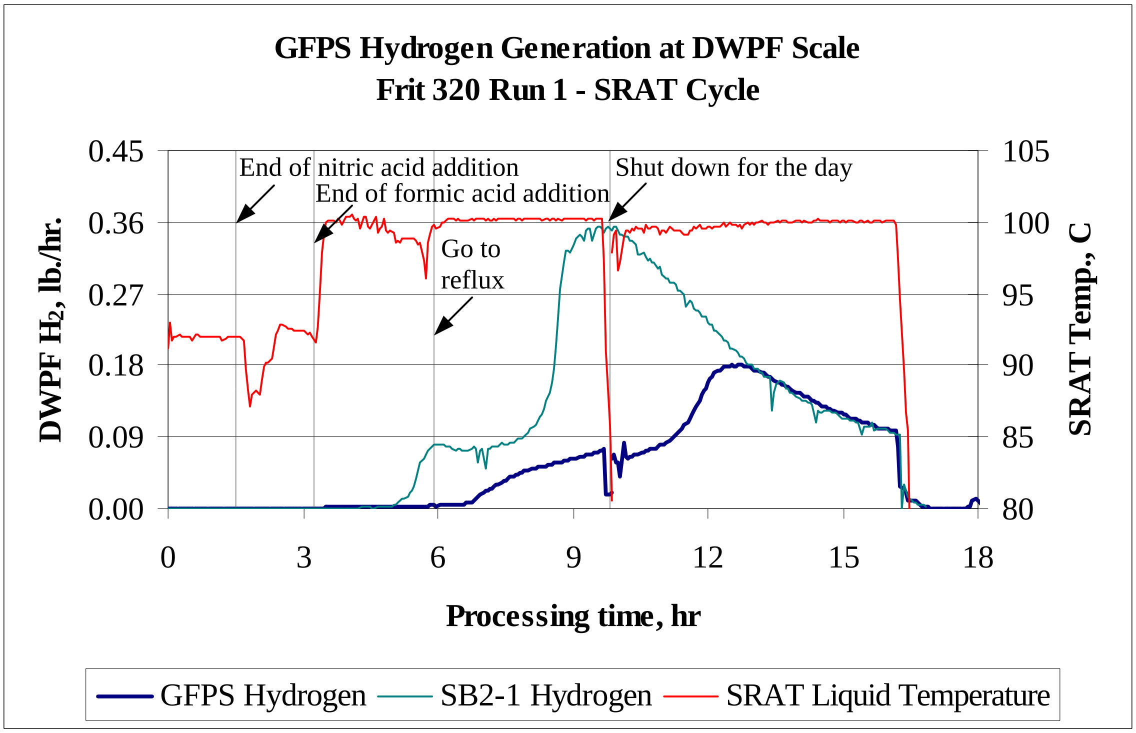
SRAT Cycle Gas Generation

GC data were examined for both Sludge Batch 2 runs with Frit 320. GC calibration data from before and after each run were used along with the calibration gas cylinder composition to produce corrected concentrations for all of the analyzed gases of interest.

These values were combined with helium flow rate data to produce time-dependent flow rates for the other gases. These were then converted into equivalent lbs./hr. for a 6000 gallon batch.

Figure 2 compares the GFPS hydrogen generation rate at DWPF-scale with the bench-scale data from the baseline run (SB2-1) for Sludge Batch 2 sludge[1]. The run marks and SRAT liquid temperature are from the GFPS run, but were close in time to the bench-scale run events as well. Considerable analysis was required to convert the GC data into the DWPF basis flow. Small drifts in the hydrogen, helium, carbon dioxide, and nitrous oxide calibrations were factored out of the raw composition data. The contribution of the SMECT sparge air to the total flow was extracted. Uncondensed moisture was ignored. The peak hydrogen volume % observed occurs in the SRAT condenser outlet upstream of the junction with the SMECT purge air under these assumptions. This was calculated to have reached 0.28 % at the peak in Figure 2 at about the 12.5 hour mark.

Figure 2: H_2 in First GFPS Run

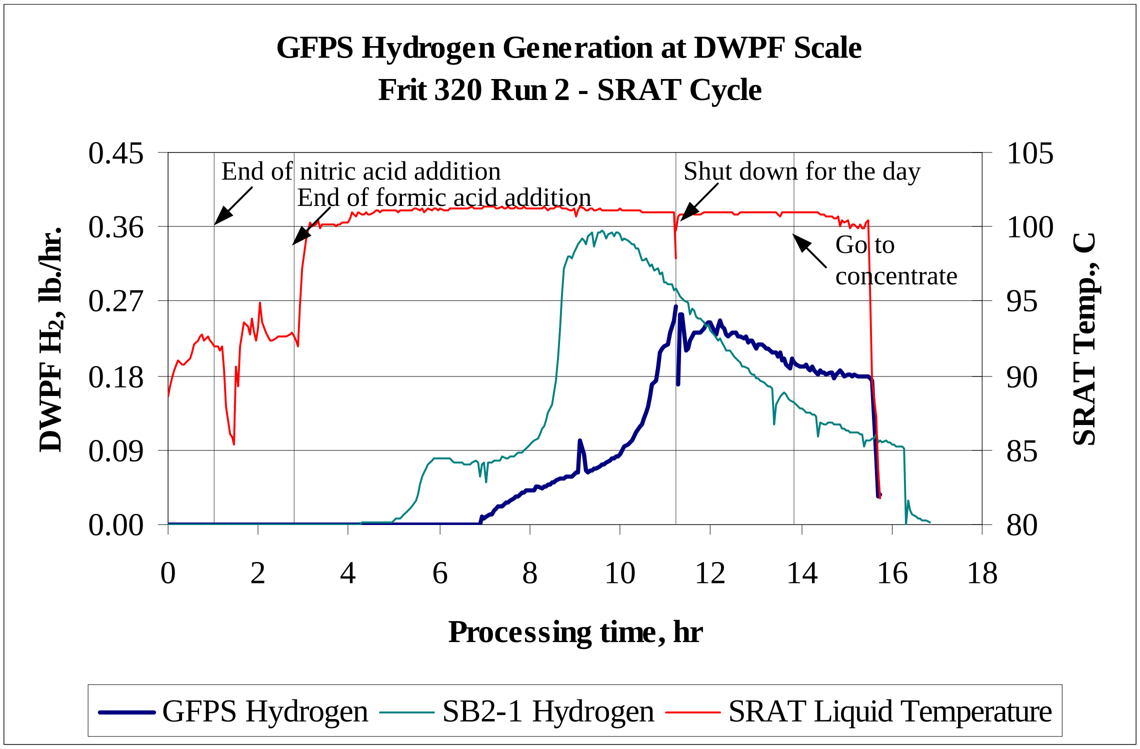


GFPS peak hydrogen levels for Frit 320 Run 1 did not reach those seen in the bench-scale run. This was almost certainly the case for the SRAT cycles run in 11/2000 and 12/2000 using Sludge Batch 2 sludge. The 11/2000 Frit 200 run seems generally comparable to the two 2002 GFPS runs described here. The 12/2000 Frit 200 GFPS run, however, did not appear to have peaked by the end of the SRAT cycle, and did not reach as high a concentration (uncorrected) as the 11/2000 run.

There could be processing explanations for the lower observed gas generation rates in the GFPS relative to the bench-scale work, such as lower-intensity mixing in the GFPS. Hydrogen generation rate data did exhibit a double rise that was typical of the bench-scale work. The first rise was during processing hours 7-9 and the second rise was during processing hours 11-12. (Processing hours are hours at normal temperatures, about 93°C for acid addition and 100°C for boiling.)

Figure 3 shows equivalent data for Frit 320 Run 2.

Figure 3: H_2 in Second GFPS Run

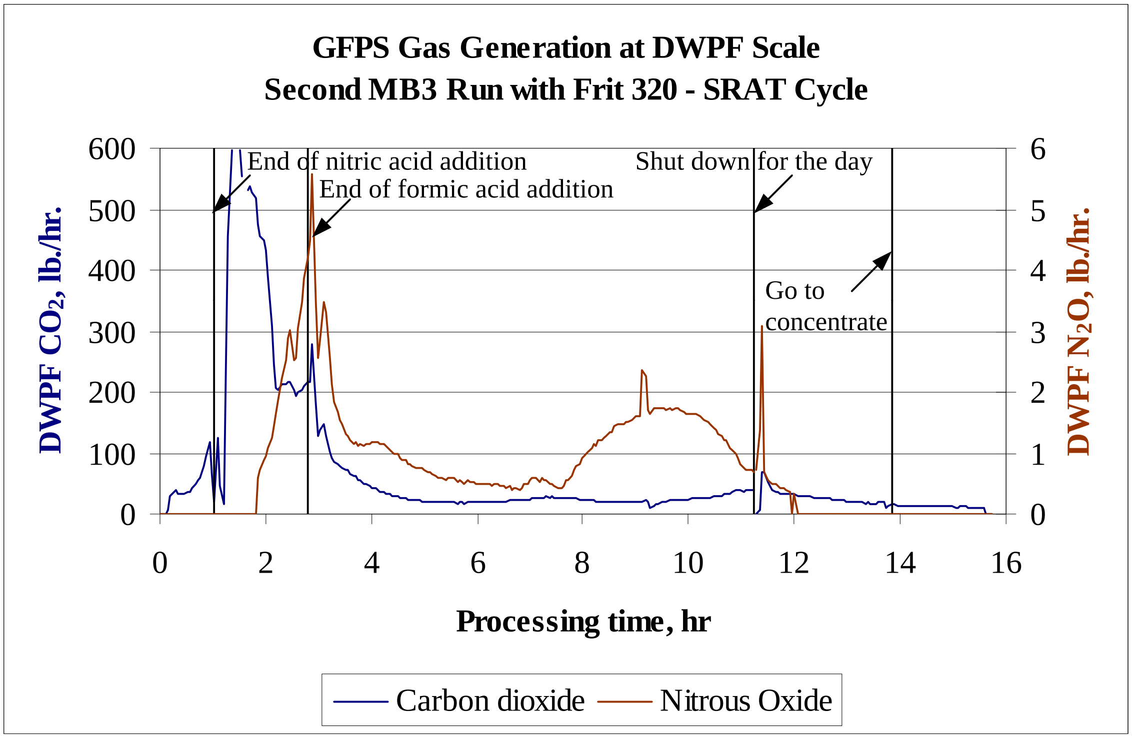


The total SRAT processing time for Frit 320 Run 2 was about an hour shorter because acid addition pumping went more smoothly and the time in-between steps time was kept shorter. Even though indicated SRAT temperature readings were below 90°C, these numbers are not considered valid. One explanation is that dead zones, which are cool relative to the bulk material, can form near the vessel wall (which is where the thermocouple is located) and give false readings of the bulk SRAT temperature. These dead zones typically occur when the sludge thickens (see rheology discussion below). The temperature uncertainty is observed during acid addition, but not during boiling. Boiling supplies additional agitation and seems to remix the wall regions.

The hydrogen generation data in Figure 3 are higher in magnitude from that in Figure 2, but it took longer to be detected. Trace amounts of hydrogen appeared in Frit 320 Run 1 shortly after going to boiling, whereas it took nearly four hours of boiling to detect hydrogen in Frit 320 Run 2. Examination of the GC scans showed that there was no hydrogen peak on the GC scans during this period in the second run, i.e. that this was not a GC peak identification problem. The cause for the delayed onset in hydrogen generation is not known. The second run also showed a double rise in hydrogen generation rate. Hydrogen generation rate initially increased from about the seven hour mark and then increased even more rapidly around the 10-11 hour mark before starting to decrease. The spike in hydrogen generation in Figure 3 at about the 9 hour mark coincides with the addition of more antifoam. It is possible that the peak hydrogen generation rate was impacted by the SRAT cycle interruption that occurs following 12.5 hours. This interruption seems to have coincided with the peak hydrogen generation rate in both runs.

GC data for carbon dioxide and nitrous oxide were only analyzed for Frit 320 Run 2. The DWPF-scale flow rates derived from the data are shown in Figure 4. The bench-scale gas generation data showed a correlation between a second peak in nitrous oxide generation and the second increase in hydrogen generation rate.

Figure 4: CO_2 and N_2O in Second GFPS Run



The first nitrous oxide peak occurred at about the three hour mark. A second peak occurred at about the 9-10 hour mark. The acceleration in hydrogen generation, which occurred during the 10-11 hour period, thus came shortly after the second nitrous oxide peak. This is the first time this phenomena has been quantified in the GFPS. Nitrous oxide peaked initially at about 9 lb./hr. in the baseline bench-scale run, and then peaked again at about 2 lb./hr. Results here are very similar, except that the bench-scale second peak occurred about 2-3 hours earlier than the GFPS second peak.

Carbon dioxide does not go off-scale in Figure 4 at about the 1.5 hour mark, but three data points were lost because of GC issues while the generation rate was just under 600 lb./hr. Carbon dioxide peaked in the baseline bench-scale run several times. The first was near the end of nitric acid addition at about 160 lb./hr., the second was early in formic acid addition at about 600 lb./hr., the third was near the end of formic acid addition just before the onset of boiling at about 300 lb./hr., and the fourth was at the peak in hydrogen generation at about 50 lb./hr. Corresponding values for the GFPS Frit 320 Run 2 are about 120, 600, 215, and 40 lb./hr. in Figure 4 at 0.95, 1.5, 2.5 and 11.2 hours respectively. The general conclusion can be made that Frit 320 Run 2 processed very much like the baseline bench-scale run except for some time delays in the events that occurred after acid addition.

The following conclusions summarize some of the SRAT gas generation data:

- Hydrogen peaked in the first SRAT cycle at 0.18 lb./hr.
- Hydrogen peaked in the second SRAT cycle at 0.26 lb./hr.
- Hydrogen generation was less intense than in the corresponding bench-scale run.
- Carbon dioxide and nitrous oxide generation in the second SRAT cycle were comparable to past bench-scale data.
- Most carbon dioxide seen early was attributed to carbonate destruction. Later carbon dioxide was attributed to reduction reaction chemistry.
- Carbon dioxide was evolved at about seven times the hydrogen evolution rate during the latter part of the SRAT cycle on a molar basis. (Presumably 1/7 of the carbon dioxide came from catalytic decomposition of formic acid to CO_2 and H_2. The other 6/7 could be due to reduction reactions or an alternate decomposition stoichiometry.)
- GFPS gas generation events seemed to occur later in time than in bench-scale runs, suggesting a possible mass transfer limitation with increasing scale.

SRAT Cycle Analytical Results

Table 3 gives the analytical results for SRAT product samples from each of the two Frit 320 runs. A portion of each GFPS SRAT product sample was calcined at 900°C for the ICP elemental analysis to conform to the treatment given to the 2000 samples. Calcining the slurry prior to ICP analysis was adopted during the Sludge Batch 2 simulant preparation work. This was driven by a highly variable value for the waters of hydration associated with Fe_2O_3, as Fe_2O_3ÌxH_2O, during microwave and oven drying measurements of wt. % total solids. The case $x = 3$ corresponds to $Fe(OH)_3$. This was the presumed form in the simulant recipes provided to the University of South Carolina – Columbia for simulant preparation. Oven drying results suggested that $x = 0.5$-1.0 was more likely, giving a smaller mass of total and insoluble solids than originally predicted. Calcining samples takes x to near zero, and eliminates much of the ambiguity associated with interpreting the ICP results.

Results for the two Frit 320 runs are in the columns labeled “GFPS Run 1 Sample 2-26-02” and “GFPS Run 2 Sample 3-6-02”. A calculated Frit 320 Run 2 result is also given which attempts to correct the contribution of frit left in the GFPS SRAT/SME vessel from the first run, and then give a frit-free set of elemental results for the second run. The cumulative data for the two runs suggests that the starting sludge for both runs was more nearly 15.2-15.3 wt. % total solids than the previously measured 15.5 wt. % total solids. Solids have been measured consistently using oven drying at 110-115°C. Results from the current campaign are compared to bench-scale results from Koopman[1] in the column labeled “Based on Bench-Scale ICP Results”. Results are also compared to average GFPS results from the two Sludge Batch 2 runs with Frit 200 from Koopman[3] in the column labeled “GFPS Mean 2000 Results”.

Table 3: SRAT Product Sample Results

	Based on Bench-Scale ICP Results	GFPS Mean 2000 Results	GFPS Run 1 Sample 2-26-02	GFPS Run 2 Sample 3-6-02	GFPS Run 2, corrected for Run 1 heel
Total Solids (wt. %)	-	18.1	17.7	18.2	-
Insoluble Solids (wt. %)	-	12.0	12.4	13.0	-
Soluble Solids (wt. %)	-	6.1	5.3	5.2	-
Calcined Solids (wt. %)	-	13.9	13.5	13.9	-
Chloride (mg/kg)	-	2530	2600	2550	-
Nitrite (mg/kg)	-	<100	<100	<100	-
Nitrate (mg/kg)	-	24600	16800	17600	-
Formate (mg/kg)	-	22200	24300	21300	-
Sulfate (mg/kg)	-	400	311	373	-
Al (calcined solids wt. %)	10.3	10.1	11.20	10.60	11.20
B (calcined solids wt. %)	0.0	0.0	0.0	**0.10**	0.0
Ba (calcined solids wt. %)	0.21	0.26	0.30	0.28	0.30
Ca (calcined solids wt. %)	3.2	3.35	2.03	2.36	2.49
Cr (calcined solids wt. %)	0.28	0.26	0.28	0.26	0.28
Cu (calcined solids wt. %)	0.19	0.16	0.17	0.16	0.17
Fe (calcined solids wt. %)	34.9	36.1	35.10	32.00	33.82
K (calcined solids wt. %)	0.03	0.12	0.11	0.14	0.15
Li (calcined solids wt. %)	0.0	0.0	**0.15**	**0.313**	0.08
Mg (calcined solids wt. %)	0.18	0.14	0.14	0.14	0.15
Mn (calcined solids wt. %)	3.2	2.78	3.68	3.51	3.71
Na (calcined solids wt. %)	10.6	10.6	6.04	**7.35**	7.18
Ni (calcined solids wt. %)	1.8	1.09	2.01	1.89	2.00
P (calcined solids wt. %)	-	0.04	0.067	0.065	0.069
Pb (calcined solids wt. %)	0.22	0.24	0.28	0.26	0.27
Si (calcined solids wt. %)	1.27	1.40	1.42	**3.35**	1.17
Sr (calcined solids wt. %)	-	0.12	0.10	0.11	0.12
Zn (calcined solids wt. %)	0.30	0.32	0.39	0.38	0.40
Zr (calcined solids wt. %)	0.56	0.61	0.79	0.73	0.77

The numbers in the last column are from a calculation. The calculation attempts to remove some B, Li, Na, and Si from the sample results in the 3-6-02 column in the same proportions as they are present in Frit 320. The changes over time in the results for calcium, manganese, and nickel are hard to explain as anything other than analytical uncertainty. There are no signs of deposits in the GFPS feed drums other than a thin band of dried sludge solids near the gas-liquid interface that forms where sludge dries out. The lithium in the 2-26-02 results was completely unexpected. The vessel was fairly clean prior to Frit 320 Run 1, and no known source of lithium was introduced to the SRAT. Some improvements in the overall quality of the results have been made through improved sample preparation methods. This has impacted some of the results above, making them harder to compare to older data. A sum of oxides check was made for the calcined solids in the last three columns of Table 3. It was assumed that all iron was Fe^{+3} and all manganese was Mn^{+2}. The results for 2002 samples were about 97%, whereas the previous results summed to about 100%.

The frit balance on the Frit 320 Run 2 sample required about 6.5% of the calcined solids mass to be due to residual frit from Frit 320 Run 1. This corresponds to retention of 2% of the frit added in the first run, or nearly a kilogram. Allowing for some sludge and water in proper proportions leads to the conclusion that about 2-3 kg of the first run SME product remained in the vessel for the second run. This was virtually the same result for residual solids mass following the first run that was obtained for the two runs with Frit 200[3].

The overall material balance on Frit 320 Run 1 closed to within about 1%, once an adjustment for retained mass was made, versus to within about 3% without an adjustment. The overall material balance for Frit 320 Run 2 closed to within about 3%. This includes adjustments for sludge left in the vessel after the first run and for sludge left in the vessel after the second run. Conversion of SMECT level into mass is probably the major source of uncertainty in the overall mass balance. There is some indication that the extent of solid to gas conversion is being underestimated somewhat in all of the SRAT data for Sludge Batch 2 obtained in bench-scale and GFPS runs. This shows up as a lower than predicted wt. % total solids in the SRAT product. The difference is less than 1 wt. % relative to SRAT products of about 17-18 wt. % total solids. It may be related to the carbon dioxide produced during hydrogen evolution that is in excess of 1:1 on a mole basis with molecular hydrogen.

Nitrite destruction was good indicating that aspect of SRAT processing was not adversely affected by lower intensity mixing in the GFPS. Other anion results were comparable to past data, except for nitrate which was at about two-thirds of the levels seen in 2000. The soluble solids content was lower in these runs by nearly 1%. This may be related to gas evolution or analytical uncertainty. Accurate slurry density data were not obtained. The SRAT product samples, once shaken, formed a bubble-rich three-phase slurry that was not typical of processing at boiling. Densities of about 0.9 g/ml were measured for the three-phase slurry.

The following conclusions can be made concerning the SRAT analytical data:

- SRAT processing was comparable between the two Frit 320 runs in 2002.
- SRAT processing in 2002 was comparable to the previous two runs with Sludge Batch 2 simulant in 2000.
- Nitrite destruction was excellent.
- Density measurement was a problem area.

- The frit heel can be essentially eliminated from the second run by better flushing between runs, but only at the expense of losing that material from the final product.

Mercury and SRAT Concentration

One request for this task was to study mercury during the SRAT cycle. Sludge Batch 2 sludge is a low mercury sludge. The starting sludge passes the SRAT product specification for mercury even with no stripping. Each nominal GFPS batch was trimmed with 58.9 g of mercury as 63.6 g of HgO. Unusual results were obtained in the bench-scale work with regard to mercury stripping efficiency[1]. Some sample results indicated that mercury content increased during stripping. The GFPS has been run six times prior to these runs. Process lines are expected to be contaminated with mercury by now. Sample results indicating low mercury content are as likely to be due to normal processing as to be due to contamination by residual mercury.

The GFPS SRAT/SME was rinsed before the tests several times. The SMECT and the MWWT were drained and rinsed before both tests, and then inventoried with process water. The SMECT was also doped with 35 ml of concentrated nitric acid to simulate a processing history including condensation in the presence of NO_x. The SMECT and SRAT/SME sample points are now purged routinely before pulling samples for analysis. Elemental mercury is now collecting in the SMECT sample line during processing (a low point). This is a new observation. There were a number of small beads of mercury in the sample point purge beaker, but none in the submitted SMECT samples themselves. The hidden reservoirs of mercury may, nonetheless, be affecting the SMECT dissolved mercury content. Elemental mercury was seen in the bottom of the MWWT following each run, but not in large amounts (<1 gram).

Two different concentration strategies were followed during the SRAT cycle. In Frit 320 Run 1 concentration occurred after acid addition and was followed by reflux. In Frit 320 Run 2 reflux occurred after acid addition, while concentration occurred at the very end of the SRAT boiling period. SRAT product samples, SMECT samples after the SRAT cycle, and SME product samples were submitted for mercury analysis. SMECT, MWWT, and FAVC condensate samples following the SME cycle were archived for possible analysis if there is interest.

SRAT product results combined with SRAT mass at the time of sampling indicated the presence of 18.5 g Hg following Frit 320 Run 1 and 15.1 g Hg following Frit 320 Run 2. The SMECT sample result, combined with SMECT mass following the Frit 320 Run 1 SRAT cycle, indicated the presence of 1.01 g of Hg. The equivalent result for Frit 320 Run 2 was 0.97 g of Hg. The SRAT and SMECT results are very similar between runs, and they do not seem to be inconsistent. However, the initial results of the SME product samples indicated 25.6 g of Hg in the Frit 320 Run 1 SME product and 64.5 g of Hg in the Frit 320 Run 2 SME product. These are higher than in the SRAT products, and, in the second case, exceed the nominal mercury added to the run.

A pair of equivalent SME samples, pulled for rheological measurements, were submitted for mercury in an attempt to clarify the results above. Essentially the same result was obtained for Frit 320 Run 1, but the mercury in the Frit 320 Run 2 SME product fell to 29.4 g. This is much closer to the result for Frit 320 Run 1. This suggests that the samples and/or equipment are being studied at Hg concentrations where the analytical results are not a very reliable indication of processing. Another possibility is that small beads of mercury may be getting into the samples. For example, the Frit 320 Run 1 SME product sample results came from sampling the product drum (clean equipment), while the Frit 320 Run 2 SME results came from samples pulled at the process sample point (presumably Hg-contaminated equipment).

The GFPS mercury balance is not well understood at this time. The following conclusions have been drawn concerning mercury stripping during these runs:

- The initial Hg concentration was quite low (0.29 wt. % in air-dried solids).
- About two-thirds of the SRAT mercury was stripped in both runs. Some of the stripped mercury was found as the elemental liquid.
- The Hg data are sufficiently uncertain that it does not prove that the two concentration strategies are equivalent or interchangeable.
- The Hg data did not indicate, however, a detectable problem associated with either concentration strategy.
- It is quite possible that the SME product Hg concentration increased relative to the SRAT product because of non-prototypical refluxing of the SME through the MWWT.
- It is recommended that the GFPS MWWT be drained between the SRAT and SME cycles and refilled with water in future tests involving mercury measurements. It is possible that non-prototypical refluxing of the GFPS SME through the MWWT may be recycling stripped Hg into the SME slurry.
- Any future comparison of mercury stripping efficiency from the two SRAT boiling strategies should be based on a much higher initial mercury concentration (1-3 wt. % in air-dried solids).

SME Cycle Discussion

Characterization of Frit 320

Frit 320 was required for the SME cycle simulation. Each run required 45.7 kg of frit. A shipment of 100 kg of Frit 320 arrived during the first run. Shipping delays were related to grinding and sieving the frit to meet the specifications on particle size. A preliminary analysis of composition had been completed several weeks earlier using a pre-shipment sample of Frit 320 that was slightly off specification in particle size. Particle size analysis by screen sieving of a follow-up sample met spec, and the Frit 320 was shipped. A grab sample of the received Frit 320 was analyzed to confirm that the delivered particle size was within specifications. A second grab sample from the as-received Frit 320 was sent to Mobile Lab for a confirmatory elemental analysis. Although the data in Table 4 show sodium oxide in the as-received sample is off-spec by 0.1 wt. %, this is well within the analytical uncertainty for a single analysis.

Frit 320 grab sample analysis was completed between the two GFPS Frit 320 addition periods of Runs 1 and 2. The sample data for composition are given in Table 4. Species not listed in Table 4 were below both the corresponding detection limits and specified maximum impurity levels, e.g. Fe_2O_3, MnO, etc. Detection limits for fluorine and chlorine were higher than the maximum impurity specifications (specification: < 0.05 wt. %, reported result: < 0.20 wt. %).

Table 4: Frit 320 Composition Data

Element oxide:	Specification wt. %	Pre-shipment wt. %	As Received wt. %
Al_2O_3	< 1.89	0.39	0.27
B_2O_3	8.0 ± 0.75	8.2	7.9
Li_2O	8.0 ± 0.5	8.1	7.9
Na_2O	12.0 ± 0.5	11.6	11.4
SiO_2	72.0 ± 1.0	73	73
Cr_2O_3	< 0.15	0.0082	<0.002

A sample of the Frit 320 shipment was also sieved using screens. The following results were obtained for a 50.004 gram sample, of which 0.399 grams stuck to the screens:

Table 5: Frit 320 Screen Data

Screen Range	Clear Opening	Mass, grams	Wt. %
> 80 mesh	> 177 µm	0.046	0.09
80-100 mesh	149-177 µm	3.632	7.32
100-120 mesh	125-149 µm	15.301	30.85
200-120 mesh	74-125 µm	28.666	57.78
< 200 mesh	< 74 µm	1.960	3.95

Specifications are <2 wt. % in the >80 mesh cut and <10 wt. % in the <200 mesh cut. The vendor experienced some difficulties in sizing the frit to meet our specifications. Preliminary problems were due to overly high fines content. It can be seen in Table 5 that the frit seems to be concentrated on the low side of the acceptable size range.

SME Cycle Foaminess

Simulated SME cycle processing occurred in the 30-49 wt. % total solids range from completion of the first frit addition until the end of the cycle. Post-run data indicate that the wt. % total solid contents were not overly different between the two Frit 320 runs. An assessment of foaming was made in both Frit 320 SME cycles at three different boil-up rates following each Frit 320 addition. Foaminess during SME processing is reported using the same formula used for SRAT processing above. Non-boiling slurry volume is taken at as high a temperature as possible, since there was a small expansion in volume associated with heating from 80°C to 99°C. Results are given in Table 6.

Table 6: SME Foaming Results

Frit 320 Run #	Boil-up Flux, lb./hr.ft^2	Volume Expansion Factor
1, after 1st Frit addition	31	1.15
1, after 1st Frit addition	51	1.22
1, after 1st Frit addition	14	1.09
1, after 2nd Frit addition	31	1.08
1, after 2nd Frit addition	51	1.12
1, after 2nd Frit addition	14	1.01
2, after 1st Frit addition	31	1.08
2, after 1st Frit addition	51	1.13
2, after 1st Frit addition	14	1.06
2, after 2nd Frit addition	31	1.07
2, after 2nd Frit addition	51	1.11
2, after 2nd Frit addition	14	1.04

A boil-up flux of 51 lb./hr/ft^2 corresponds to about 5700 lb./hr. in a 12 ft. diameter tank, i.e. is conservative for DWPF. Antifoam capabilities were tested in the SME cycle. The Frit 320 Run 1 SME foam test after the initial frit addition occurred 29.5 clock hours after the last previous antifoam addition (just prior to boiling in the SRAT). In spite of this the foaming tendency was small. It did not appear that there was any "foam", but the data indicate that some expansion of the volume occurred that was dependent on boil-up flux. Antifoam was added between frit additions at 100 ppm during Frit 320 Run 1. No change in SME level was noted following addition. The data for the Frit 320 Run 1 may primarily indicate expansions of the slurry volume due to the presence of more bubbles between the coils and the free liquid surface at higher fluxes.

A foam build-up was observed as soon as the Frit 320 Run 2 SME cycle went to boiling following the first Frit 320 addition. This was about 20 clock hours after the last SRAT addition of which only roughly five hours were at temperatures above 80°C. As the SME went to boiling at a flux of 31 lb./hr.ft^2, the SME level rose from the 140 mark to the 170 mark over about three minutes (6.8 inch increase in foam depth). Adding 100 ppm of DeBourg antifoam produced a level drop to about the 155 mark. The first foam test for Frit 320 Run 2 was then made, see Table 6. The next foam test for Frit 320 Run 2 came after the second frit addition (about 4.5 hours later). No additional antifoam was added. Neither run showed much tendency to foam following the second frit addition, based on the volume expansion factors.

The following conclusions were drawn from the SME cycle foaming work:

- Frit 320 did not promote increased foaming in the first SME cycle, when compared to available Frit 200 data SME cycle data.
- Foaming was more pronounced in Frit 320 Run 2. This SME cycle depended on the DeBourg antifoam. The first run depended on the IIT antifoam from 9/00.
- Antifoam was needed in the SME cycle to prevent foaming.
- The foaming tendency seemed greater after the first frit addition than after the second frit addition.

SME Cycle Gas Generation

Hydrogen generation rate data were calculated for the boiling period following each of the four frit additions (total) made during the two runs. Interesting results were expected, since the SRAT hydrogen generation rate was still high at the end of the SRAT cycles. The four figures below mark processing time from when the SME liquid first reached a boiling temperature during the heat-up following frit addition.

Figure 5: Frit 320 Run 1 Hydrogen After Initial Frit Addition

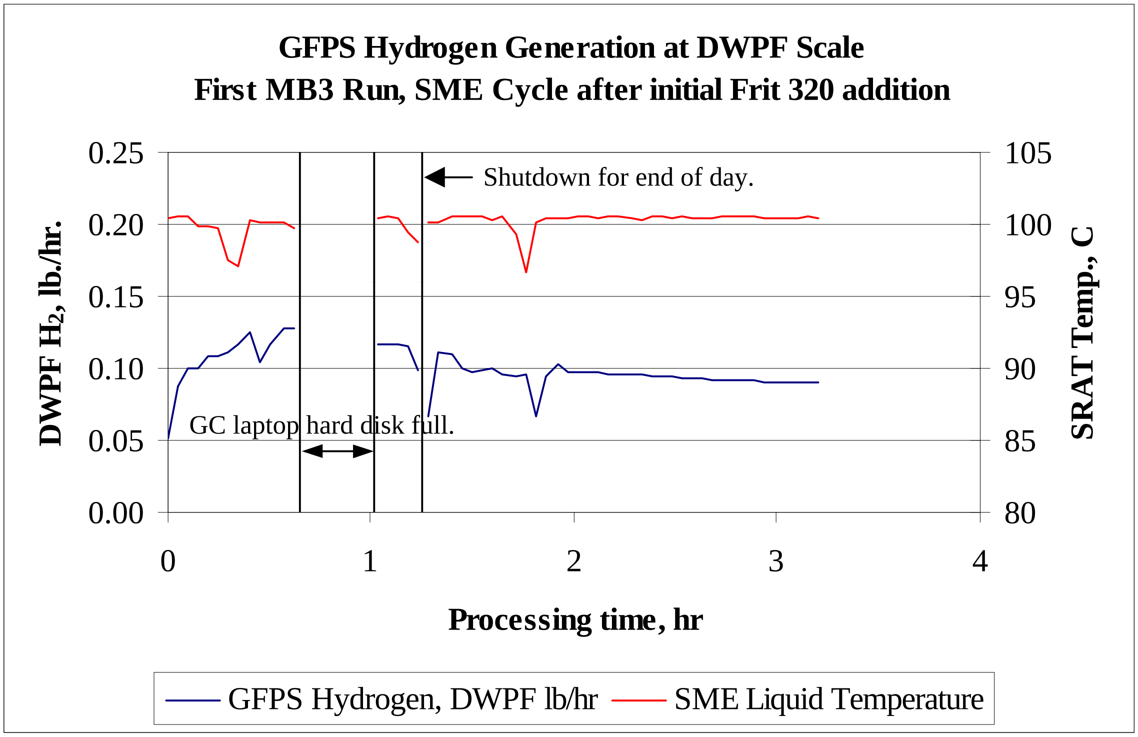


Figure 6: Frit 320 Run 1 Hydrogen After Second Frit Addition

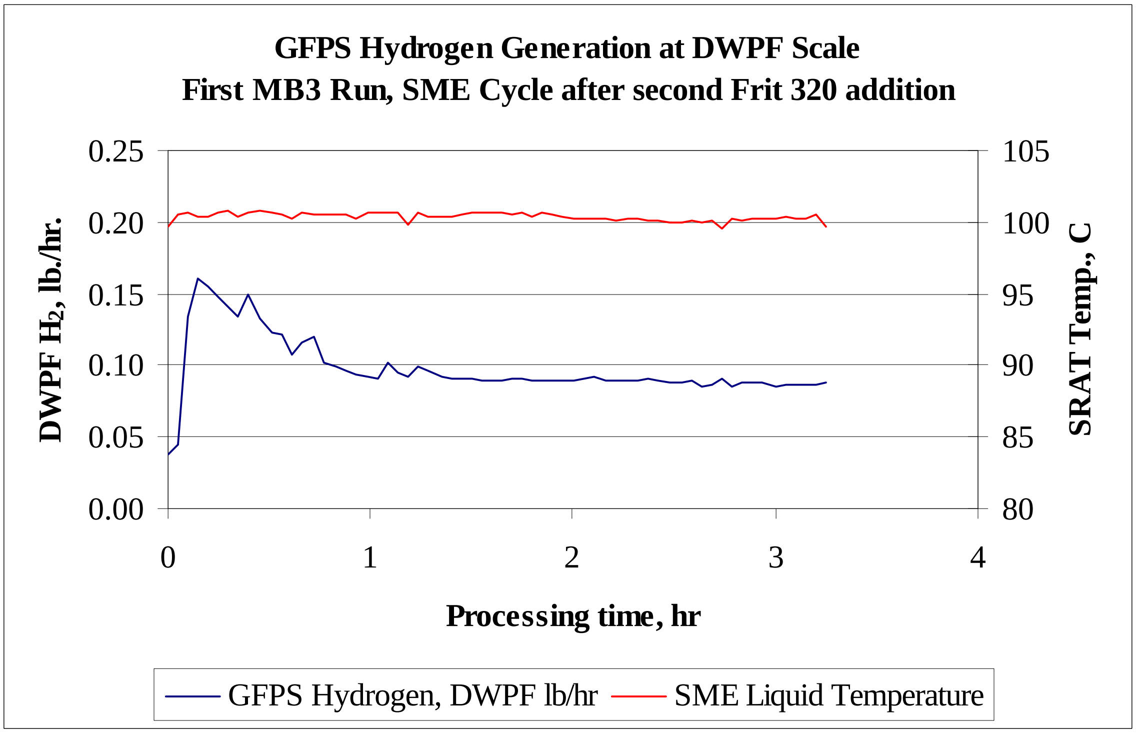


Figure 7: Frit 320 Run 2 Hydrogen After Initial Frit Addition

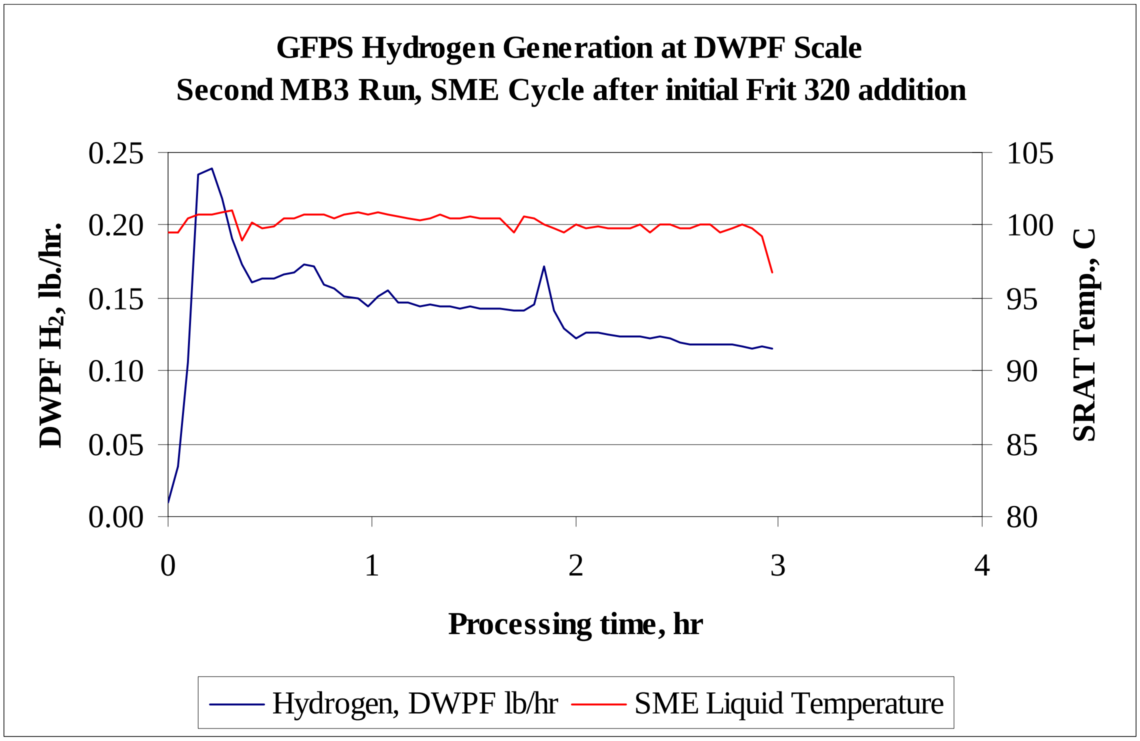
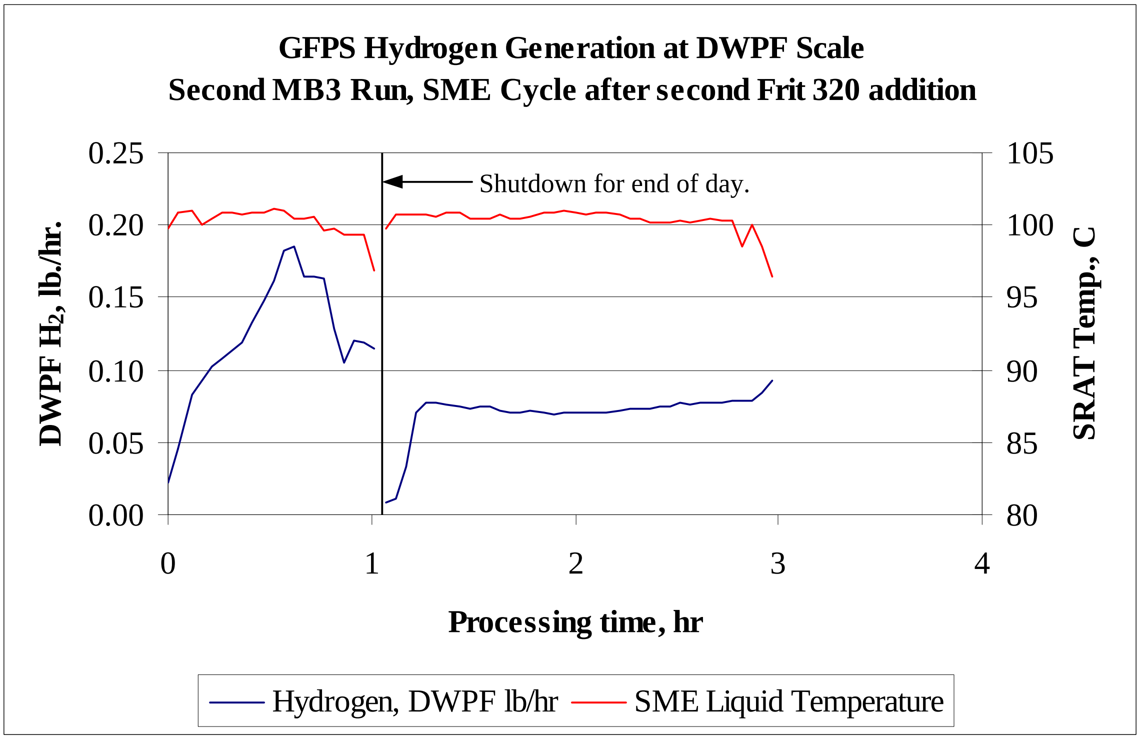


Figure 8: Frit 320 Run 2 Hydrogen After Second Frit Addition



The highest SME hydrogen generation rate was seen in Frit 320 Run 2 following the first frit addition. The SRAT hydrogen generation rate was also greater at the end of the SRAT cycle of Frit 320 Run 2 than at the end of Frit 320 Run 1. Therefore, this finding was expected. This maximum hydrogen generation rate was about 0.24 lb./hr hydrogen. This was very close to the DWPF design basis limit for the SME cycle of 0.223 lb./hr. While this brief peak slightly exceeded the DWPF design basis limit, the conclusion is that DWPF will not see comparable generation rates due to the lower noble metal concentrations in the real waste. Table 7 summarizes the peak hydrogen generation rates following the two Frit 320 additions in each of the two runs.

Table 7: SME Cycle Hydrogen Generation Rates

	Peak Generation Rate
Frit 320 Run 1, after initial frit addition	0.13 lb./hr.
Frit 320 Run 1, after second frit addition	0.16 lb./hr.
Frit 320 Run 2, after initial frit addition	0.24 lb./hr.
Frit 320 Run 2, after second frit addition	0.18 lb./hr.

Several conclusions emerge from the SME cycle gas generation rate data:

- SME hydrogen generation is impacted by end of SRAT cycle hydrogen generation.

- Forecast noble metal concentrations for Tank 8 were sufficiently high to be of concern to DWPF, but actual noble metal concentrations were lower and are probably not of concern.
- Hydrogen generation seemed to peak shortly after boiling was re-established.
- The formic acid added with the frit supplies fresh raw material for the hydrogen generation reaction.

SME Cycle Analytical Results

Table 8 gives the sample results for the two individual SME product samples (melter feeds), plus the results for the composite melter feed blend that was drummed for use by the Minimelter. N/A denotes not analyzed. Note that the Frit 320 Run 1 SME product left a "heel" behind that was stuck to the coils and vessel wall, while the Frit 320 Run 2 SME product contains some of the heel from the first run and left a heel of its own behind. A portion of the SME product samples were vitrified at 1150°C prior to performing the ICP elemental analyses. Also given are the available average results for the Tank 8/Tank 40 blend SME products from the bench-scale flowsheet study[1] and from the Frit 200 GFPS runs[3]. Anion results are for a weighted dilution of a known mass of melter feed slurry.

A calculated melter feed composition was prepared prior to the Frit 320 runs. This was used as input for Kevin Brown's calculation of glass acceptability in Appendix A. Note that Frit 320 has higher Si, Na, and Li, and has lower Mg and B. Frit 320 was added pound for pound like Frit 200. Consequently, the sludge element wt. %'s were not expected to be affected by the change in frit. Based on the GFPS Mean Results – 2000 in Table 8, the following values for B, Li, Mg, Na, and Si were expected in the SME product with Frit 320: B_2O_3 = 5.73%, Li_2O = 5.31%, MgO = 0.06%, Na_2O = 12.3%, and SiO_2 = 57.8%. Composite melter feed results from the Drum Blend Results column in Table 8 were: B_2O_3 = 6.04%, Li_2O = 5.80%, MgO = 0.03%, Na_2O = 12.6%, and SiO_2 = 54.9%. These two sets of numbers match acceptably well.

Table 8: SME Product Sample Results

	SME bench-scale, 2000 (Frit 200)	GFPS Mean Results, 2000 (Frit 200)	GFPS Run 1, 2-28-02, w/rinse (Frit 320)	GFPS Run 2, 3-7-02, pre-rinse (Frit 320)	Drum Blend Results, 3-7-02 (Frit 320)
Total Solids (wt. %)	44.1	48.3	46.2	50.3	46.9
Insoluble Solids (wt. %)	39.5	42.6	41.5	45.3	42.1
Soluble Solids (wt. %)	4.57	5.71	4.75	4.95	4.76
Vitrified Solids (wt. %)	40.7	44.5	42.4	46.2	42.9
Vitrified Solids/Total Solids	0.92	0.92	0.92	0.92	0.91
Chloride (mg/kg)	4310	2080	2050	2110	2060
Nitrite (mg/kg)	<100	<100	<100	<100	<100
Nitrate (mg/kg)	18000	22900	13100	14000	13200
Formate (mg/kg)	34600	22000	21600	22100	22800
Sulfate (mg/kg)	n/a	560	390	520	467
Al_2O_3 (vitrified solids wt. %)	5.68	5.21	5.08	4.91	5.14
B_2O_3 (vitrified solids wt. %)	8.63	8.60	6.05	5.89	6.04
BaO (vitrified solids wt. %)	<0.01	0.097	0.067	0.066	0.066
CaO (vitrified solids wt. %)	1.24	1.22	1.10	1.08	1.17
Cr_2O_3 (vitrified solids wt. %)	0.067	0.11	0.095	0.090	0.097
CuO (vitrified solids wt. %)	0.057	0.056	0.052	0.052	0.054
Fe_2O_3 (vitrified solids wt. %)	12.99	11.82	11.8	11.0	11.7
K_2O (vitrified solids wt. %)	0.13	0.18	<0.010	<0.010	<0.010
Li_2O (vitrified solids wt. %)	3.51	3.32	5.76	5.72	5.80
MgO (vitrified solids wt. %)	2.03	1.67	0.028	0.023	0.026
MnO (vitrified solids wt. %)	1.03	1.09	1.03	0.97	1.07
Na_2O (vitrified solids wt. %)	10.75	11.59	12.5	12.5	12.6
NiO (vitrified solids wt. %)	0.60	0.57	0.56	0.53	0.56
P_2O_5 (vitrified solids wt. %)	n/a	0.013	<0.040	<0.040	<0.040
PbO (vitrified solids wt. %)	0.037	0.072	0.065	0.065	0.067
SiO_2 (vitrified solids wt. %)	54.91	56.23	55.8	55.8	54.9
SrO (vitrified solids wt. %)	n/a	0.13	0.008	0.010	0.010
TiO_2 (vitrified solids wt. %)	0.029	0.067	<0.005	<0.005	<0.005
ZnO (vitrified solids wt. %)	0.034	0.14	0.050	0.044	0.050
ZrO_2 (vitrified solids wt. %)	0.22	0.31	0.284	0.262	0.287
Sum of oxides	101.9	102.5	100.3	99.0	99.6

The most puzzling result was for nitrate ion. This difference between the 2000 and 2002 campaigns appears to be real, but a candidate cause for the difference has not been identified. The nitrate difference was already present in the SRAT product analyses, Table 3. Since identical sludges were used, and identical nitric acid additions were made in the four GFPS runs with Sludge Batch 2 simulant, it would seem likely that nitrate should be nearly constant. None of the other anions appear to have been impacted in any way. Gas evolution data confirmed that the nitric acid was as effective as expected, i.e. there is no reason to suspect the pre-run nitric acid titration result.

Three frit elements (B, Li, and Si) in the melter feed should be present at about 74% of their respective concentrations in pure Frit 320, once the minor contribution of the silicon from the sludge is removed. Table 9 compares the calculated SME frit element wt. %'s to those in pure Frit 320. The last column gives the ratio of the two, ideally expected to be 0.74.

Table 9: Waste Loading Check for Frit Elements

Element:	SME (w/o sludge)	Frit 320	Ratio
B	1.876	2.484	0.755
Li	2.695	3.717	0.725
Si	25.66	33.66	0.762

Given the information in Table 9, the composite drum of combined melter feed made in the two Frit 320 GFPS runs seems to be essentially what was expected to have been produced during the campaign. It has approximately the right proportions of frit to sludge oxides, expected elemental composition, wt. % total solids, etc. The drum containing the composite melter feed of the two runs weighed 653.5 lbs. The tare weight was 65.4 lbs. The net weight of melter feed put in the drum was 608.8 lbs. (An additional 17 lbs. of rinsed melter feed, recovered from the SME, was collected in a separate carboy.)

Redox data were obtained on the composite Frit 320 SME product sample along with an EA glass standard. The results are given in Table 10.

Table 10: Redox of SME Product

	Fe^{2+}	Fe (total)	Fe^{2+}/Fe^{3+}	Fe^{2+}/Fe (total)
EA Glass	0.083	0.467	0.220	0.177
Frit 320 Blend	0.050	1.01	0.052	0.050

Although the Frit 200 SME product samples from the GFPS were not analyzed for redox, subsequent samples of this material, taken from the Minimelter feed tank, were analyzed for redox. These showed that the Frit 200 SME product Fe^{2+}/Fe (total) ran between 0.015 and 0.055.

The following conclusions can be made for the SME cycle analytical data.

- Sludge waste loading was virtually identical to the Frit 200 Sludge Batch 2 product.
- SME product wt. % total solids was virtually identical to the Frit 200 Sludge Batch 2 product.
- Elemental composition shifts for the frit components were as expected.
- SME product redox was comparable to the Frit 200 Sludge Batch 2 product.
- The 2002 runs show lower nitrate than the 2000 runs. This is presently unexplained.

Rheology Tests on GFPS Samples

SME Product Rheology

The impact of Frit 320 on the rheology of the SME product slurry was examined. The anticipated impact was a negligible effect, since Frit 320 is chemically similar to Frit 200 and has similar particle size. Past data have characterized the rheology of the SME product slurry from the bench-scale baseline run with Frit 200 over a range of wt. % total solids[2]. Measurement difficulties with these samples have been known for some time. Vigorous mixing to get a good, uniform suspension of the solid particles entrains a lot of air into the samples. The trapped air is in the form of small bubbles (<1 mm diameter) that are very slow to disengage in a gravitational field. Rheological measurements of these three-phase samples tend to give lower apparent viscosities than when the bubbles are absent.

The first study of the effect of changing to Frit 320 on the rheology of simulated melter feed was carried out by Stone (2001)[11]. Data were taken using a Haake RS-150 Rheostress rheometer. The 60 mm diameter parallel plate geometry was used with a 1.5 mm gap. Measurements were made at 25°C. The two melter feeds were at 43.0-43.4 wt. % total solids with pH's ranging from 7.58-8.16. The yield stress and consistency were 108 dynes/cm^2 and 9.2 cP for the Frit 200 melter feed and 107 dynes/cm^2 and 8.6 cP for the Frit 320 melter feed. The conclusion was that there was no significant effect on the rheological properties. The parallel plate data were not corrected for the presence of a non-Newtonian fluid in a variable shear stress field.

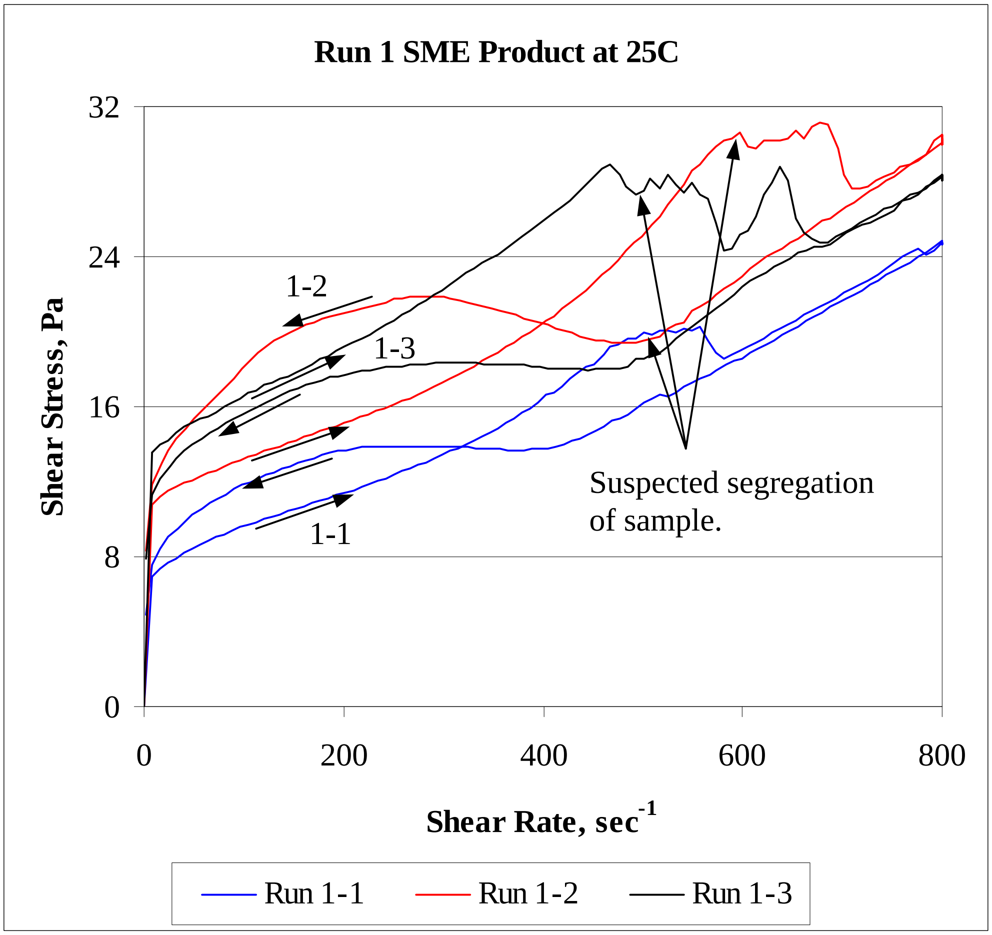
The most directly comparable results from Koopman[2] for melter feed with Frit 200 using the concentric cylinder geometry (RV20 rheometer, MV2 sensor, 25°C) were for a 44.2 wt. % total solids sample at pH 6.8. These gave a yield stress of 130 dynes/cm^2 and a consistency of 17 cP. Additional rheological data were available from this study at five other wt. % total solids, including 41.0, 48.9 and 51.7 wt. %. At 48.9 wt. % total solids the yield stress was 260 dynes/cm^2 and the consistency was 34 cP. So both yield stress and consistency doubled from the 44.2 wt. % sample.

The shear stress field in the concentric cylinder geometry is nearly constant across the annular sample gap for a given rotation rate. It is difficult to compare numbers obtained in the different geometries without considerable analysis. Future studies may show that it is possible to correct the parallel plate data for the differences in the shear stress field.

Conversely, they may show that frit settling and bubble rising issues are exaggerated to such a degree that parallel plate data on SME product slurries is of little value.

Figure 9 shows three concentric cylinder rheograms obtained on the SME product from the Frit 320 Run 1 at 46.23 wt. % total solids. These rheograms are not very pleasing because of all of the variability and unexplained structure seen in the up and down curves. The sample was gently hand-mixed in an effort to minimize bubble entrainment. This was somewhat successful in keeping bubble count down. It appears that the solids were not sheared very well and began to settle during the measurement period. (It is speculated that the erratic structures in the up curves, , at 500-700 sec^{-1} were due to phase segregation within the sample matrix.)

Figure 9: Concentric Cylinder Rheology of Run 1 SME Product



Unreported testing with Frit 200 Sludge Batch 2 SME product from the earlier GFPS runs has shown that a layer of bubbles forms on the inner cylinder during a measurement. These bubbles are very fine, perhaps 0.1 mm in diameter at a guess, but definitely less than 1 mm in diameter. At that size the only way to tell them apart from frit seems to be to rub the sample, and then see if they break or grind on the cylinder surface. The centrifugal forces in the rheometer drive the more dense slurry toward the outer wall, which displaces the less dense bubbles from the slurry matrix. The bubbles then migrate toward the surface of the inner cylinder.

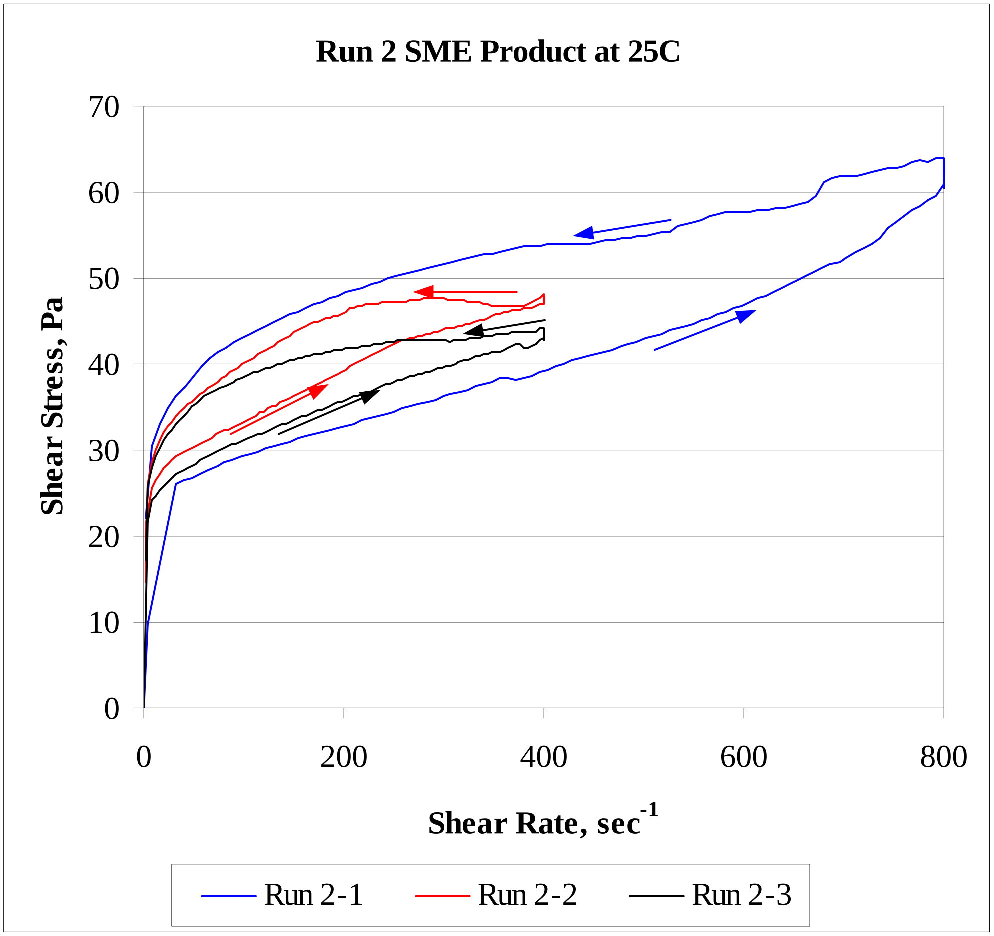
There were a lot of bubble patches on the inner cylinder after Run 1-1 (poured from bottle), but only a few after Run 1-2 (drew sample from middle of bottle for 1-2). The Run 1-3 material was sheared for ten minutes at 400 sec^{-1} before making the flow curve measurement. Ignoring the 1-1 data because of the bubbles, a rough yield stress of 120-140 $dynes/cm^2$ is suggested by the 1-2 and 1-3 data. These values are consistent with the Frit 200 data reported in Koopman[2], and are perhaps a little less viscous.

There is a more qualitative observation that can be made concerning the rheology of both the Frit 320 Run 1 product and the composite product. The GFPS SME product is intentionally over-concentrated right at the end. Water to dilute the SME to the target wt. % total solids is used to rinse the SME vessel walls and coils in order to get a higher percentage of the product into the drum. The amount of rinse water equals the condensate mass from over-concentrating. When the primary transfer of Frit 320 Run 1 SME product to the drum was made, the wt. % total solids can be inferred by mass balance arguments to have been about 1-1.5% higher than the final sample result of 46.23 wt. % total solids.

This higher solids content (47.3-47.8%) melter feed could not really be mixed with the twin 4-inch blade drum mixer, i.e. couldn't get the drum contents to turn over. When the rinse water and recovered solids were added to the drum, the drum contents mixed quite well. (This drum mixer can achieve 2000 rpm without problems, but the GFPS SRAT/SME agitator can only achieve 600 rpm with its pair of 4.5-inch propeller blades.) The composite melter feed in the drum at 46.9 wt. % total solids after the SME product from Frit 320 Run 2 was mixed with the product from Frit 320 Run 1 also had trouble mixing. This was partly due to it being higher in solids and partly due to there being twice as much mass. The drum mixer was moved around to different locations in an attempt to obtain a uniform blend. Drum sampling used the Coliwasa (*co*nsolidated *li*quid *wa*ste *sa*mpler) tube in order to get material from various depths in the drum. (Target total solids was at or above 46.5 wt. % to match the total solids content of the product from the Frit 200 runs.) Once away from the GFPS drum handling area, the portable drum mixing stand was used with its 12 inch impeller to thoroughly mix the melter feed prior to transferring it into the Minimelter feed tank.

Rheological data were also taken on the Frit 320 Run 2 SME product prior to executing the melter feed recovery step (which dilutes the sample). Two of the rheometer tests used a much shorter shear rate range. This was an adaptation caused by the results from testing Frit 320 Run 1 SME product. Nonetheless, the fact that the ramp down curve lies above the ramp up curve was disturbing. This trend was present in the Frit 200 study also[2]. The Bingham plastic rheological equation of state parameters were fit to the up curve data there, and the same procedure was followed here. The raw Frit 200 data were taken from 0 to 350 sec^{-1}.

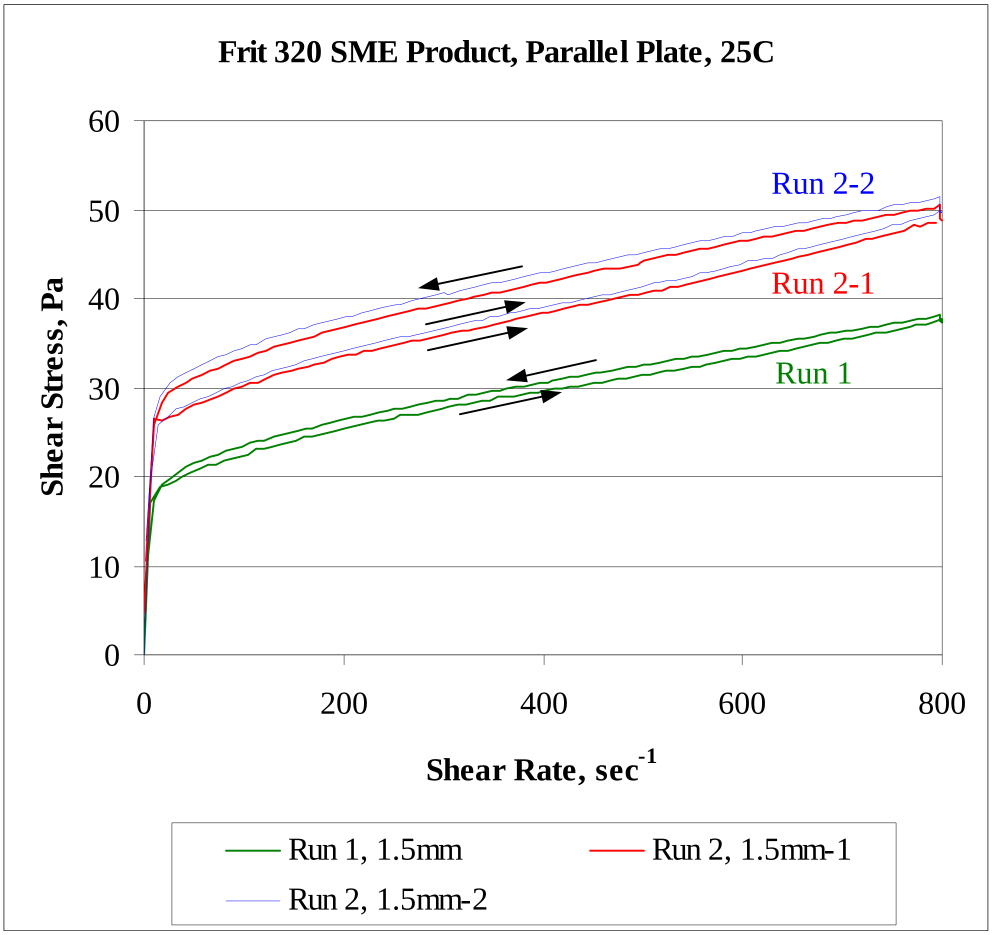
Figure 10: Concentric Cylinder Rheology of Run 2 SME Product



The mean up-curve yield stress obtained over the shear rate interval from 100 sec^{-1} to 350 sec^{-1} was 275 dynes/cm^2. The Frit 320 Run 2 sample was 50.3 wt. % total solids and 45.3 wt. % insoluble solids. The closest data with Frit 200 were at a yield stress of 260 dynes/cm^2 at 48.9 wt. % total solids and 45.3 wt. % insoluble solids[2]. This result agreed well with the Frit 320 Run 2 result, again suggesting that Frit 320 was not affecting rheology significantly differently than Frit 200.

Parallel plate data are shown in Figure 11. The data are more aesthetically pleasing in that the curves are straighter and the up and down ramp curves are closer together. It has not been established that this geometry gives meaningful results for simulated melter feeds. Because of the horizontal fluid gap, any issues with frit settling or bubbles rising are expected to be more significant. Some segregation may have already occurred before the ramp of shear rate begins, e.g. bubbles coming to the underside of the top plate.

Figure 11: SME Product Parallel Plate Rheograms



The yield stress from the parallel plate measurement was noticeably higher than from the concentric cylinder geometry for the Frit 320 Run 1 sample (213 vs. ~130 dynes/cm^2). Consistency was about 20-30 cP for both. Conversely, the Frit 320 Run 2 yield stress results are very comparable (~285 and ~275 dynes/cm^2). Frit 320 Run 2 consistency, however, was only about 63% as large by parallel plate as by concentric cylinder (25 cP vs. ~40 cP). These differences between geometries may be due to the non-Newtonian nature of the samples. In that case, additional analysis to correct for non-Newtonian behavior might be able to bring the data into better agreement with the concentric cylinder geometry, at least for samples that are equally stable over the time scale of the measurements in both geometries. Alternately, the differences may be related to the preparation of low bubble content rheometer samples versus moderately bubbled samples. Differences could also be related to how the data were regressed. This becomes an issue when a region of relatively constant plastic viscosity is not found on the rheogram.

Rheology During Acid Addition

It has been noted in previous bench-scale and GFPS simulations of the SRAT cycle that the slurry seems to thicken during acid addition and then to thin back down toward the end of formic acid addition. A series of samples were pulled to examine this effect. Samples were pulled prior to nitric acid addition, mid-way through nitric acid addition, and following nitric acid addition. Five more samples were pulled 1/5, 2/5, 3/5, 4/5, and all of the way through formic acid addition. Data on these samples are given in Table 11. Rheological data were taken on an RS-150 rheometer using the Z41 cylinder. This gives a narrower gap than the Z38 cylinder used for samples containing frit. (The Z41 and Z38 cylinders are the RS-150 equivalents to the MV1 and MV2 cylinders used on the RV20 and RV30 rheometers. These last two instruments are now used exclusively for radioactive samples.)

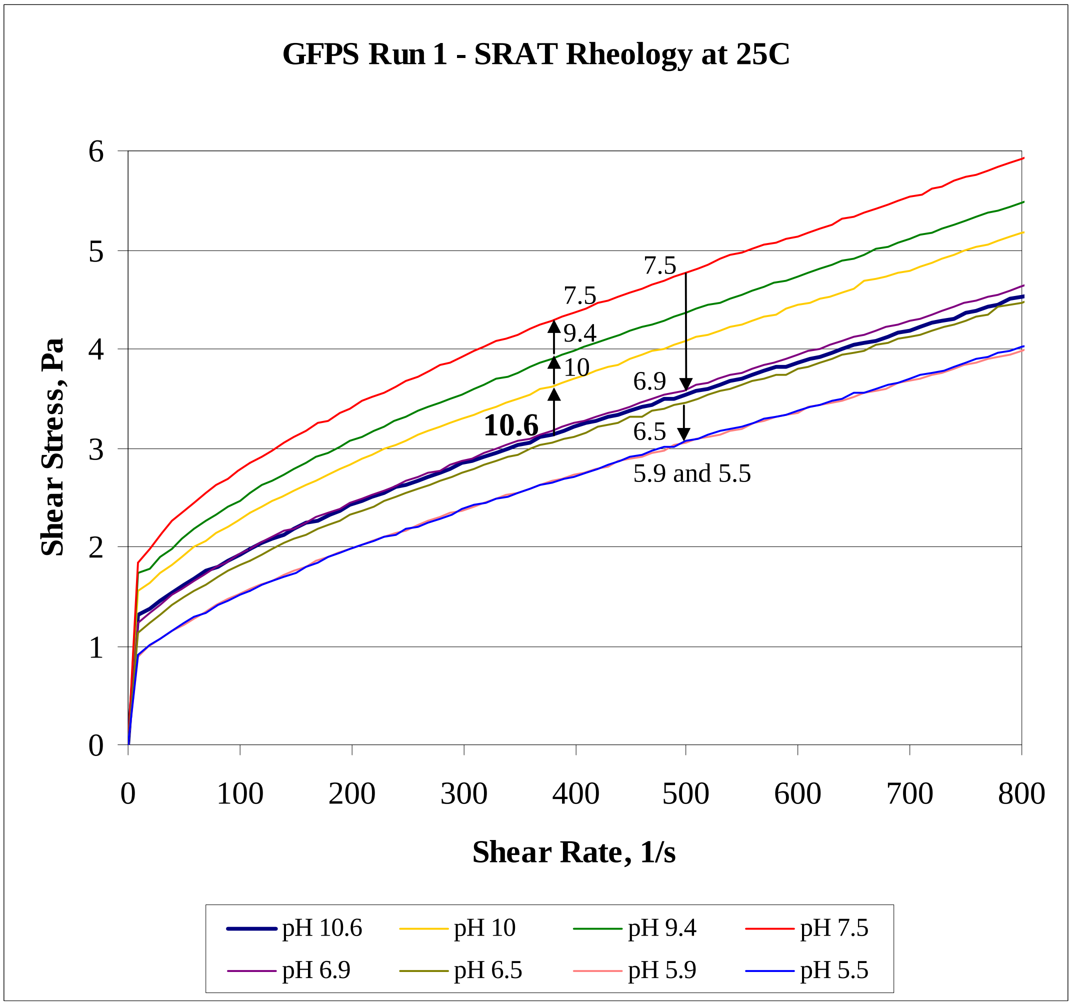
Table 11: Run 1 Acid Addition Sample Results

Acid Addition Sample:	Wt. % Total Solids	Wt. % Insoluble Solids	Wt. % Soluble Solids	Supernate pH	Yield Stress, dynes/cm^2	Consis-tency, cP
Start	13.52	10.77	2.75	10.6	17.4	3.5
mid HNO_3	14.47	11.63	2.84	10.0	20.9	3.9
Between	14.54	11.53	3.01	9.4	23.1	4.0
1/5 formic	14.47	11.16	3.31	7.5	26.1	4.2
2/5 formic	14.42	10.69	3.73	6.9	17.3	3.7
3/5 formic	14.49	10.20	4.29	6.55	16.4	3.6
4/5 formic	14.44	9.53	4.91	5.9	12.6	3.6
End	14.42	9.10	5.32	5.55	12.4	3.6

The yield stress and consistency in Table 11 were for a Bingham plastic rheological equation. The shear rate ramp data were uniformly fit over the interval from 100 to 800 sec^{-1} for the increasing shear rate ramp. The ramp up rheological data are shown in Figure 12 below. Ramp down data were virtually identical. Results for the two samples at pH 5.9 and 5.55 are virtually indistinguishable at this scale. Yield stress varied by more than a factor of two during acid addition. The maximum occurred for the sample at pH 7.5. Consistency, or plastic viscosity, stayed in a narrow range. The data fall below a yield stress of 25 dynes/cm^2 and a consistency of 10 cP. This is due to the operational limits of the GFPS with regard to mixing. Hopefully the samples can be decanted to track a lower water content process and be rerun on the RS-150 at some future time. A similar series of samples was taken during Frit 320 Run 2. These samples have small quantities of Frit 320 in them left over from Frit 320 Run 1.

Wt. % total solids would be projected to increase by about 2% during acid addition in the absence of chemical reactions based on mass balance considerations. A 0.9% increase was measured. A 0.72% increase was projected based on detailed analysis of the corresponding baseline bench-scale run[1]. That analysis estimates carbonate destruction, nitrite destruction, nitrate formation, and water production from chemical reactions. These are then factored into the running GFPS material balance used to track the progress of each run as net lost solids and made water.

Figure 12: Ramp Up Rheograms for Acid Addition



The maximum in yield stress does not align with the maximum in wt. % insoluble solids in Table 11. The conclusion is that there is at least a dual effect on rheological properties at work during acid addition. One is the variation in rheology expected from changing the wt. % insoluble solids. The other is probably the effect of pH on the solvated region around the individual solid particles. The effects may be more cleanly separated with simulants, since aluminum was not co-precipitated with the iron-nickel-manganese solids. Consequently, dissolution of aluminum might not effect other particles in the sludge. Dissolution of aluminum from an agglomerate of aluminum-iron-nickel-manganese could leave a somewhat more porous particle behind without changing the bulk dimensions or numbers of particles.

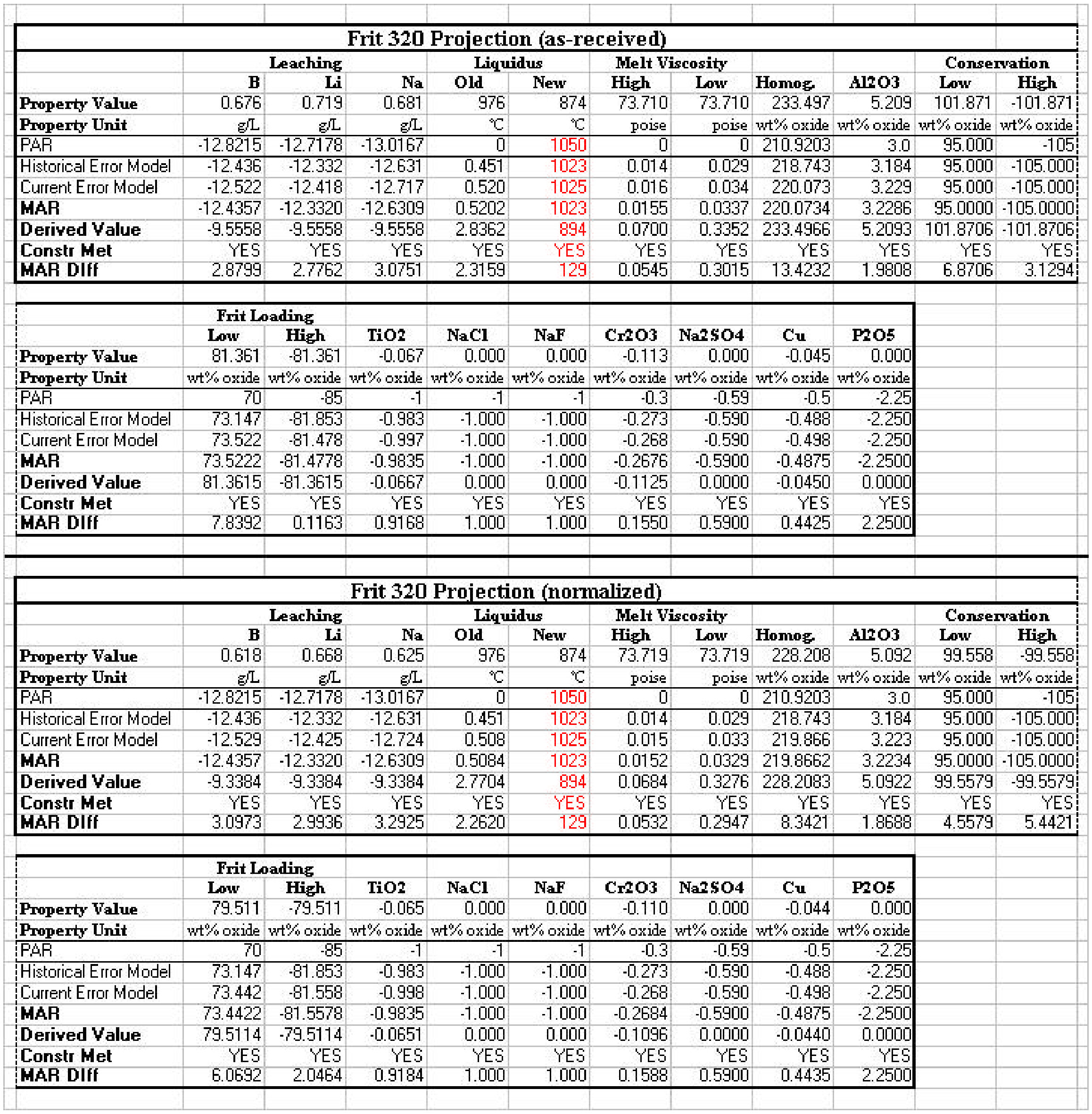
The following conclusions were made based on the rheological measurements that accompanied the Frit 320 GFPS campaign:

- Frit 320 is comparable to Frit 200 in its impact on the rheology of SME product.
- Better analysis was complicated by the presence of long-lived bubbles.

- 47 wt. % total solids gives a fairly viscous slurry.
- More work is needed with the parallel plate geometry before it can be considered ready for routine use on slurry samples.
- Two factors were affecting rheology changes during acid addition. These were pH and wt. % insoluble solids.
- Acid addition produces a maximum in yield stress near pH 7.5. One good way to evaluate how simulant chemistry compares to real waste chemistry might be to see if a washed radioactive waste slurry produces similar rheological changes during acid addition.

APPENDIX A: GLASS ACCEPTABILITY RESULTS

Performed by Kevin G. Brown of the Immobilization Technology Section.



Frit 320 Projection (as-received)

	Leaching			Liquidus		Melt Viscosity				Conservation	
	B	**Li**	**Na**	**Old**	**New**	**High**	**Low**	**Homog.**	**Al2O3**	**Low**	**High**
Property Value	0.676	0.719	0.681	976	874	73.710	73.710	233.497	5.209	101.871	-101.871
Property Unit	g/L	g/L	g/L	℃	℃	poise	poise	wt% oxide	wt% oxide	wt% oxide	wt% oxide
PAR	-12.8215	-12.7178	-13.0167	0	1050	0	0	210.9203	3.0	95.000	-105
Historical Error Model	-12.436	-12.332	-12.631	0.451	1023	0.014	0.029	218.743	3.184	95.000	-105.000
Current Error Model	-12.522	-12.418	-12.717	0.520	1025	0.016	0.034	220.073	3.229	95.000	-105.000
MAR	-12.4357	-12.3320	-12.6309	0.5202	1023	0.0155	0.0337	220.0734	3.2286	95.0000	-105.0000
Derived Value	-9.5558	-9.5558	-9.5558	2.8362	894	0.0700	0.3352	233.4966	5.2093	101.8706	-101.8706
Constr Met	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES
MAR Diff	2.8799	2.7762	3.0751	2.3159	129	0.0545	0.3015	13.4232	1.9808	6.8706	3.1294

	Frit Loading								
	Low	**High**	**TiO2**	**NaCl**	**NaF**	**Cr2O3**	**Na2SO4**	**Cu**	**P2O5**
Property Value	81.361	-81.361	-0.067	0.000	0.000	-0.113	0.000	-0.045	0.000
Property Unit	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide
PAR	70	-85	-1	-1	-1	-0.3	-0.59	-0.5	-2.25
Historical Error Model	73.147	-81.853	-0.983	-1.000	-1.000	-0.273	-0.590	-0.488	-2.250
Current Error Model	73.522	-81.478	-0.997	-1.000	-1.000	-0.268	-0.590	-0.498	-2.250
MAR	73.5222	-81.4778	-0.9835	-1.000	-1.000	-0.2676	-0.5900	-0.4875	-2.2500
Derived Value	81.3615	-81.3615	-0.0667	0.000	0.000	-0.1125	0.0000	-0.0450	0.0000
Constr Met	YES	YES	YES	YES	YES	YES	YES	YES	YES
MAR Diff	7.8392	0.1163	0.9168	1.000	1.000	0.1550	0.5900	0.4425	2.2500

Frit 320 Projection (normalized)

	Leaching			Liquidus		Melt Viscosity				Conservation	
	B	**Li**	**Na**	**Old**	**New**	**High**	**Low**	**Homog.**	**Al2O3**	**Low**	**High**
Property Value	0.618	0.668	0.625	976	874	73.719	73.719	228.208	5.092	99.558	-99.558
Property Unit	g/L	g/L	g/L	℃	℃	poise	poise	wt% oxide	wt% oxide	wt% oxide	wt% oxide
PAR	-12.8215	-12.7178	-13.0167	0	1050	0	0	210.9203	3.0	95.000	-105
Historical Error Model	-12.436	-12.332	-12.631	0.451	1023	0.014	0.029	218.743	3.184	95.000	-105.000
Current Error Model	-12.529	-12.425	-12.724	0.508	1025	0.015	0.033	219.866	3.223	95.000	-105.000
MAR	-12.4357	-12.3320	-12.6309	0.5084	1023	0.0152	0.0329	219.8662	3.2234	95.0000	-105.0000
Derived Value	-9.3384	-9.3384	-9.3384	2.7704	894	0.0684	0.3276	228.2083	5.0922	99.5579	-99.5579
Constr Met	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES	YES
MAR Diff	3.0973	2.9936	3.2925	2.2620	129	0.0532	0.2947	8.3421	1.8688	4.5579	5.4421

	Frit Loading								
	Low	**High**	**TiO2**	**NaCl**	**NaF**	**Cr2O3**	**Na2SO4**	**Cu**	**P2O5**
Property Value	79.511	-79.511	-0.065	0.000	0.000	-0.110	0.000	-0.044	0.000
Property Unit	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide	wt% oxide
PAR	70	-85	-1	-1	-1	-0.3	-0.59	-0.5	-2.25
Historical Error Model	73.147	-81.853	-0.983	-1.000	-1.000	-0.273	-0.590	-0.488	-2.250
Current Error Model	73.442	-81.558	-0.998	-1.000	-1.000	-0.268	-0.590	-0.498	-2.250
MAR	73.4422	-81.5578	-0.9835	-1.000	-1.000	-0.2684	-0.5900	-0.4875	-2.2500
Derived Value	79.5114	-79.5114	-0.0651	0.000	0.000	-0.1096	0.0000	-0.0440	0.0000
Constr Met	YES	YES	YES	YES	YES	YES	YES	YES	YES
MAR Diff	6.0692	2.0464	0.9184	1.000	1.000	0.1588	0.5900	0.4435	2.2500

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