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SETTLING RATE STUDY FOR MELTER FEED OF TANK 42 SLUDGE AND CST IN THE DEFENSE WASTE PROCESSING FACILITY (DWPF) (U)

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SAVANNAH RIVER SITE

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EXECUTIVE SUMMARY

One alternative ITP is looking into is the use of non-elutable CST resin to remove Cs-137 from the supernate of SRS High Level Waste. ITP requested ITS to investigate if the settling behavior of the CST resin differs from the current DWPF glass former (frit) used in the melter feed preparation (Issue 13, HLE-TAR-98060, Appendix C). The CST settling behavior is important in that it could impact the slurry and thus sampling homogeneity, which in turn affects the Waste Acceptance criteria and mixing requirements. The testing requested by this TAR was scoping in nature.

In order to conduct this study, melter feeds containing CST were obtained from a previous study of the maximum hydrogen generation rate for a CST augmented DWPF sludge (Issue 21, HLE-TAR-98060, Appendix C). In this previous study, CST was combined with a non-radioactive Tank 42 sludge simulant in a 1/10000th lab-scale SRAT vessel. The CST was to replace the PHA that is currently part of DWPF's coupled flowsheet. Frit 200 was added to a 1/10000th lab-scale DWPF SME vessel and a 10 wt% CST composition in the glass was targeted. From this hydrogen study four melter feeds were obtained: sludge-only, CST as received or coarse, CST intermediate ground, and CST finely ground. The settling behaviors of these four feeds were then examined.

The following major points can be drawn from the settling study:

- For the Coarse CST melter feed, no discernable settling pattern was evident between the frit and CST.
- The Intermediate CST (smallest sized particles) appears to settle slower than the frit.
- The Fine CST (intermediate sized particles) appears to settle at about the same rate as the frit.

FUTURE WORK

Due to the scale of this scoping study and the variability in the sampling and analytical techniques, it is suggested that a more precise study be performed to better quantify the settling differences between the CST and frit. One suggestion would be to perform a settling study on a DWPF scale using the full scale SRAT at TNX or on a 1/240th scale using the pilot system at 786-A. Using a larger scale would allow better matching on mixing characteristics, such as mixer speeds and impeller shapes. A larger scale would allow more consistent and easier sampling for both the settled solids and core samples.

This study does show that there are settling differences between the different sized CST and frit. Future experimentation should try to match DWPF's scale and mixing characteristics more closely, devise a more accurate method for extracting settling samples at more frequent times, and build a core sampler that is easy to use and properly segregates the layers at the bottom.

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BACKGROUND

The Defense Waste Processing Facility began processing radioactive Tank 51 Sludge in 1996. Due to delays in starting up the In-Tank Precipitation (ITP) process, DWPF began processing sludge-only feed instead of the planned coupled sludge and Precipitate Hydrolysis Aqueous (PHA) feed. Due to the problems with the ITP process, four replacement alternatives are being investigated, one of which is the use of CST. CST is a non-elutable resin to remove Cs-137 from the supernate fraction of SRS High Level Waste. The CST is combined with the sludge in the SRAT to replace the PHA that is currently part of DWPF's coupled flowsheet. Frit is then added to the SRAT product in a typical DWPF SME cycle.

The salt disposition team requested ITS to investigate the settling behavior of the CST resin in the DWPF melter feed (Issue 13, HLE-TAR-98060, Appendix C). The CST settling behavior is important in that it could impact the slurry and thus sampling homogeneity, which in turn affects the Waste Acceptance criteria and mixing requirements. The testing requested by this TAR was scoping in nature.

The melter feeds used in this test were prepared in a previous study of the maximum hydrogen generation rate for CST augmented sludge (Issue 21, HLE-TAR-98060, Appendix C). These melter feeds were prepared using a non-radioactive Tank 42 sludge simulant and a $1/10000^{th}$ scale SRAT/SME laboratory setup at TNX. A 10 wt% CST concentration was targeted in the glass that would be produced from the melter feeds. In all there were four melter feeds: sludge-only, CST as received or coarse, intermediate ground CST, and finely ground CST.

This document details the tests performed to quantify the settling behavior of the CST versus the frit in the four previously identified melter feeds.

INTRODUCTION

The objective of these scoping tests was to examine the settling behavior of the CST resin in the melter feeds for DWPF. Four melter feeds were investigated: Sludge-only to establish a base-line for normal sludge/frit settling behavior, CST as received or coarse, intermediate ground CST, and finely ground CST. The important parameters that were determined included:

- The Rheology model for yield stress as a function of weight percent solids of each melter feed
- The ratio of sludge, frit, and CST in the settled solids from each melter feed over time

DISCUSSION

Experimental Preparation

Rheology Curves

As part of the preparation for doing the experiments, the four melter feeds' yield stresses had to be adjusted to meet the low end of the DWPF design basis or 25 dynes/cm^2. This lower yield stress was chosen since it promotes segregation. In order to accomplish this task, a procedure for adjusting the melter feeds' yield stresses had to be developed and rheology tests were run on similar sludge-only melter feed from another SRAT/SME run (not these CST runs). The results were fitted to the following model:

$$\tau = \frac{e^{Ax}}{1 - \frac{x}{B}}$$

where τ is the yield stress in dynes/cm^2, x is the total weight percent solids, and A and B are fitted parameters where B represents the maximum weight percent solids of the slurry. This model was developed by James Marek and references are given in the run plan in Appendix B. The actual fitted parameters and curve for this preliminary feed are discussed in the first part of the attached run plan in Appendix B. The procedure for adjusting the Melter Feed Yield Stress is also shown in the attached run plan. The first part of the procedure gathers rheology data for fitting the appropriate yield stress model as a function of total weight percent solids of a particular SME product or melter feed. The weight percent solids is adjusted by adding different amounts of de-ionized water. Once this model is derived, it is used to adjust the yield stress of the remainder melter feed product in preparation for the settling runs.

This methodology was applied to each of the four SME products or melter feeds: 1) without CST, 2) with As-Received or Coarse CST, 3) with Intermediate ground CST, and 4) Finely ground CST. Complete listings of the procedures used to adjust these SME product's yield stresses are contained in Appendix B. The data used to develop these curves is shown in Table I. The rheology curves developed for the four feeds are shown in Figure 1 to Figure 4. Jim Marek's model shown above fit fairly well for all melter feeds (lowest R^2 was 0.89) but more rheology measurements would be needed to further validate the models since only four points were used in each model fit.

Based on the Rheology data and curves collected, the no CST or Sludge-Only with an initial yield stress of 24.4 dynes/cm^2 and the Fine CST melter feed with a yield stress of 25.1 dynes/cm^2 did not need adjustment. The coarse CST melter feed needed adjustment from initial weight percent of 44.5 wt% to 41.8 wt% solids to give a predicted yield stress of 25 dynes/cm^2. Due to variability in the slurry weights and solids measurements, the first dilution came back 42.7 wt% solids so another water dilution was done to try to reach 41.8 wt%. Unfortunately due to variability in the slurry weights and solids measurements, the second dilution came back 41.1 wt% for a predicted yield stress of 20.9 dynes/cm^2. Since there was greater variability in the yield stress curve for the coarse CST than for the other melter feeds and due to the time constraints of the experiment, it was decided to proceed with the Coarse CST melter feed.

The Intermediate CST melter feed also needed to be adjusted from its initial 43.6 wt% solids to 41.4 wt% to give a predicted yield stress of 25 dynes/cm^2. After the first addition of water the Intermediate CST melter feed had a 42.2 wt% solids so another dilution was made. Based on what was learned from the adjustment of the Coarse CST, this second dilution was cut in half. The resulting slurry had a 41.5 wt% solids slurry giving a predicted yield stress of 26.5 dynes/cm^2 which was acceptable for the experiment.

Table I. Rheology Data for Curve Fitting

Melter Feed Product	Sample Id	Total Solids Wt% (x)	Yield Stress (τ_{25}) [dynes/cm^2]
no CST	MFP-1	43.68	24.4
no CST	MFP-2	42.32	21.3
no CST	MFP-3	40.82	14.1
no CST	MFP-4	39.19	11.0
coarse CST	MFP-1-C	43.4	44.50
coarse CST	MFP-2-C	42.32	23.2
coarse CST	MFP-3-C	40.63	22.6
coarse CST	MFP-4-C	39.02	15.7
intermed. CST	MFP-1-I	43.62	40.60
intermed. CST	MFP-2-I	41.68	28.2
intermed. CST	MFP-3-I	40.12	21.1
intermed. CST	MFP-4-I	38.71	13.5
fine CST	MFP-1-F	41.07	25.1
fine CST	MFP-2-F	39.57	16.3
fine CST	MFP-3-F	37.57	11.7
fine CST	MFP-4-F	36.34	11.0

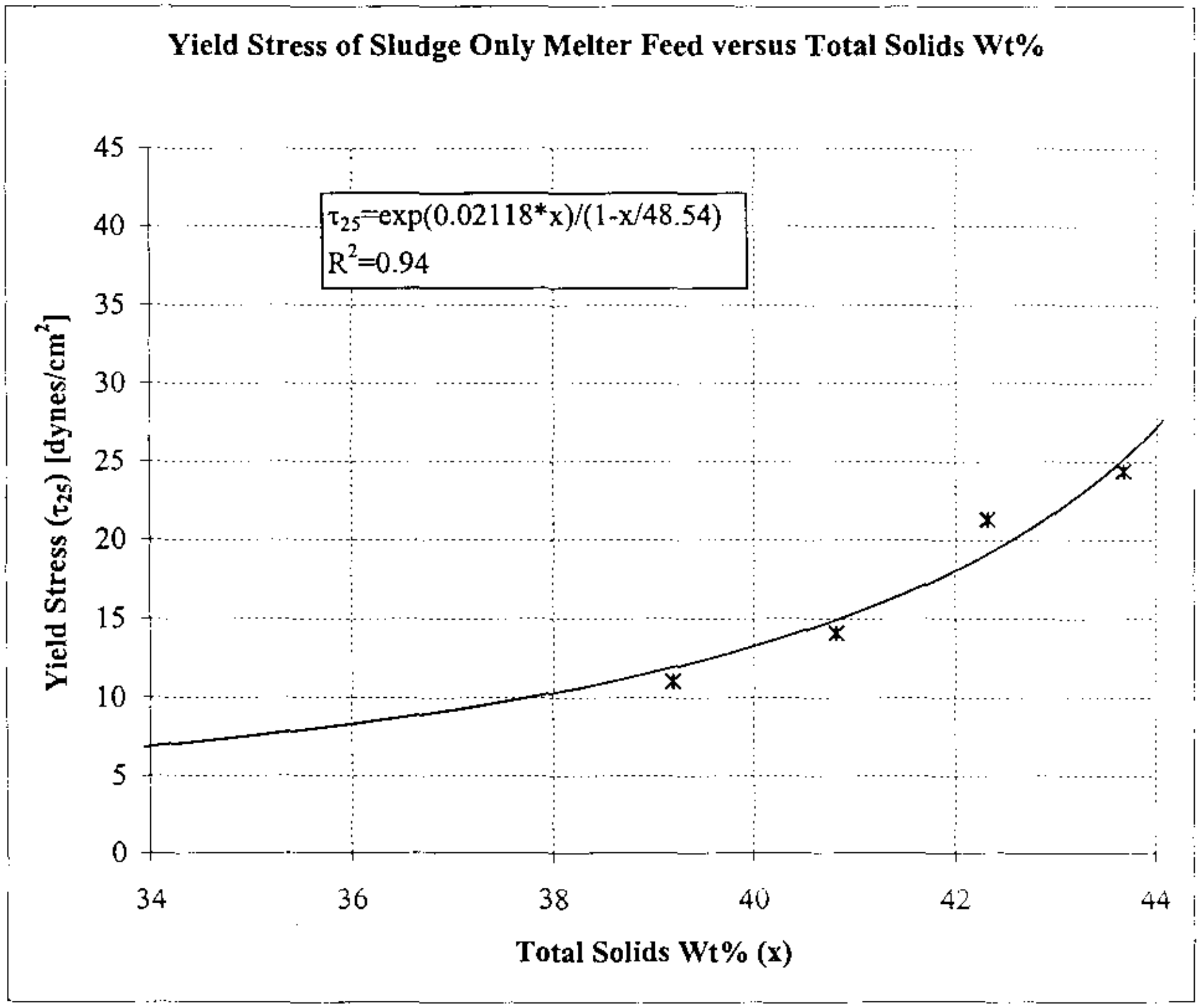

Figure 1. Yield Stress of Sludge Only Melter Feed versus Total Solids Wt%

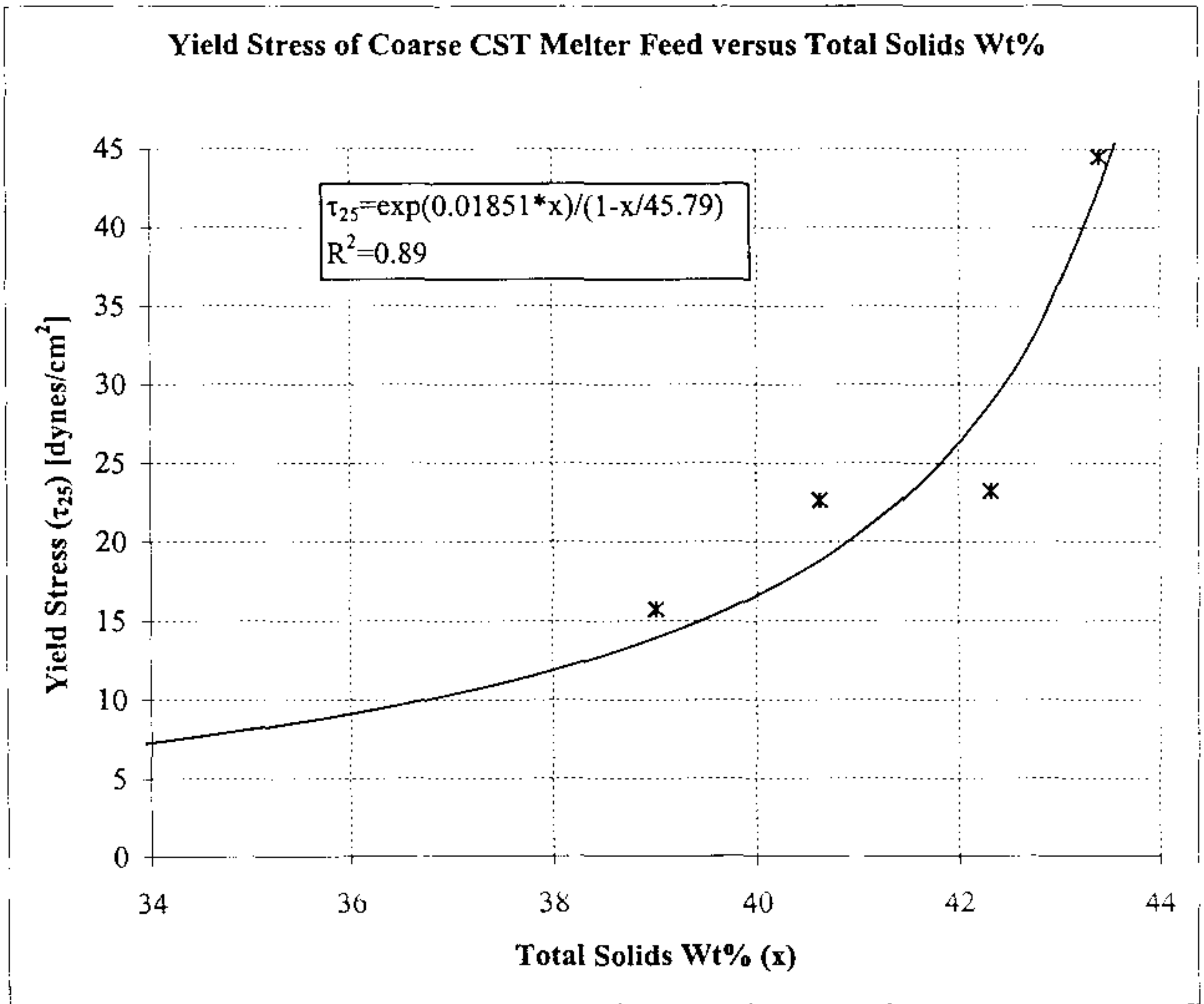

Figure 2. Yield Stress of ~~Coasre~~ Coarse CST Melter Feed versus Total Solids Wt%

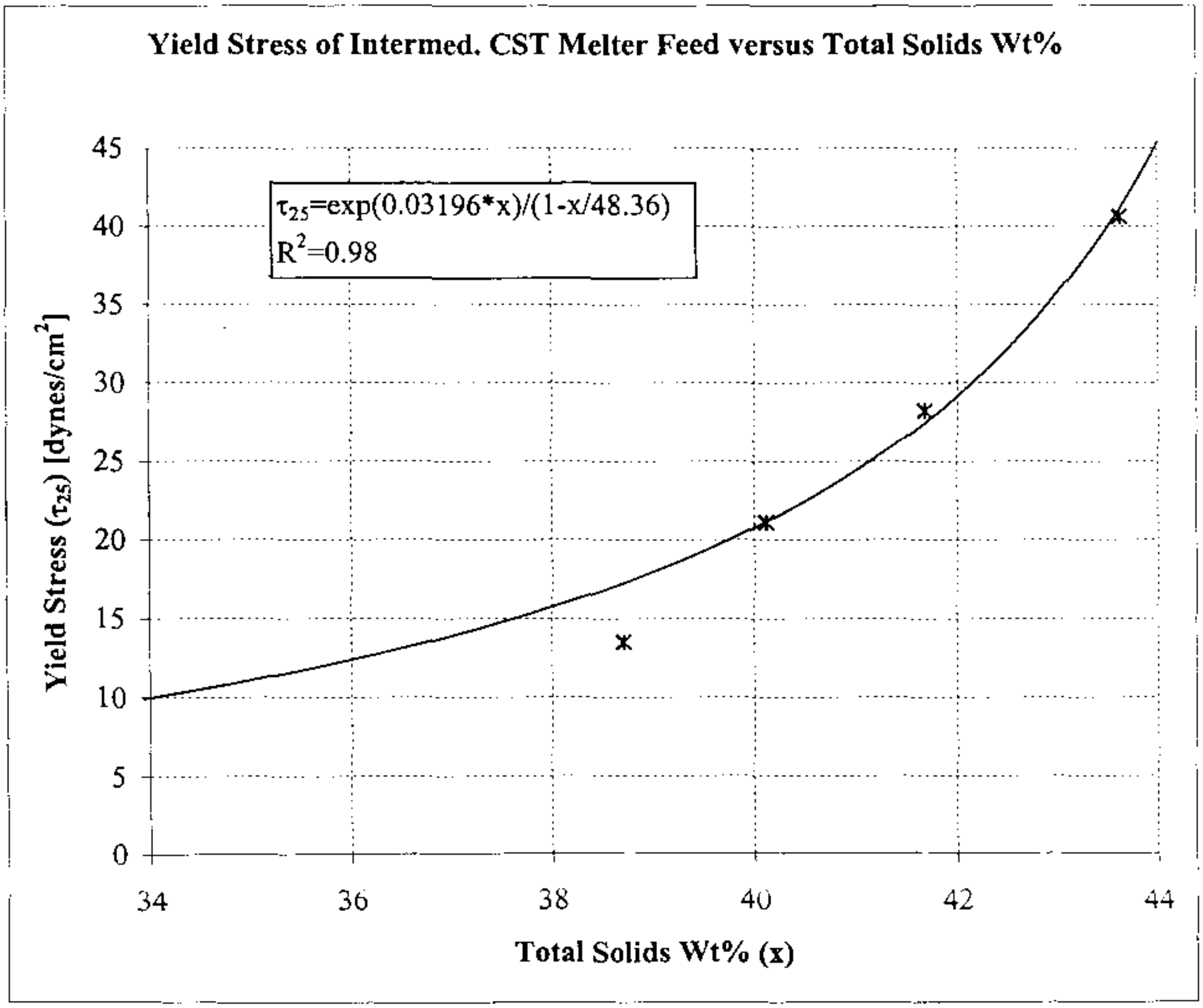

Figure 3. Yield Stress of Intermediate CST Melter Feed versus Total Solids Wt%

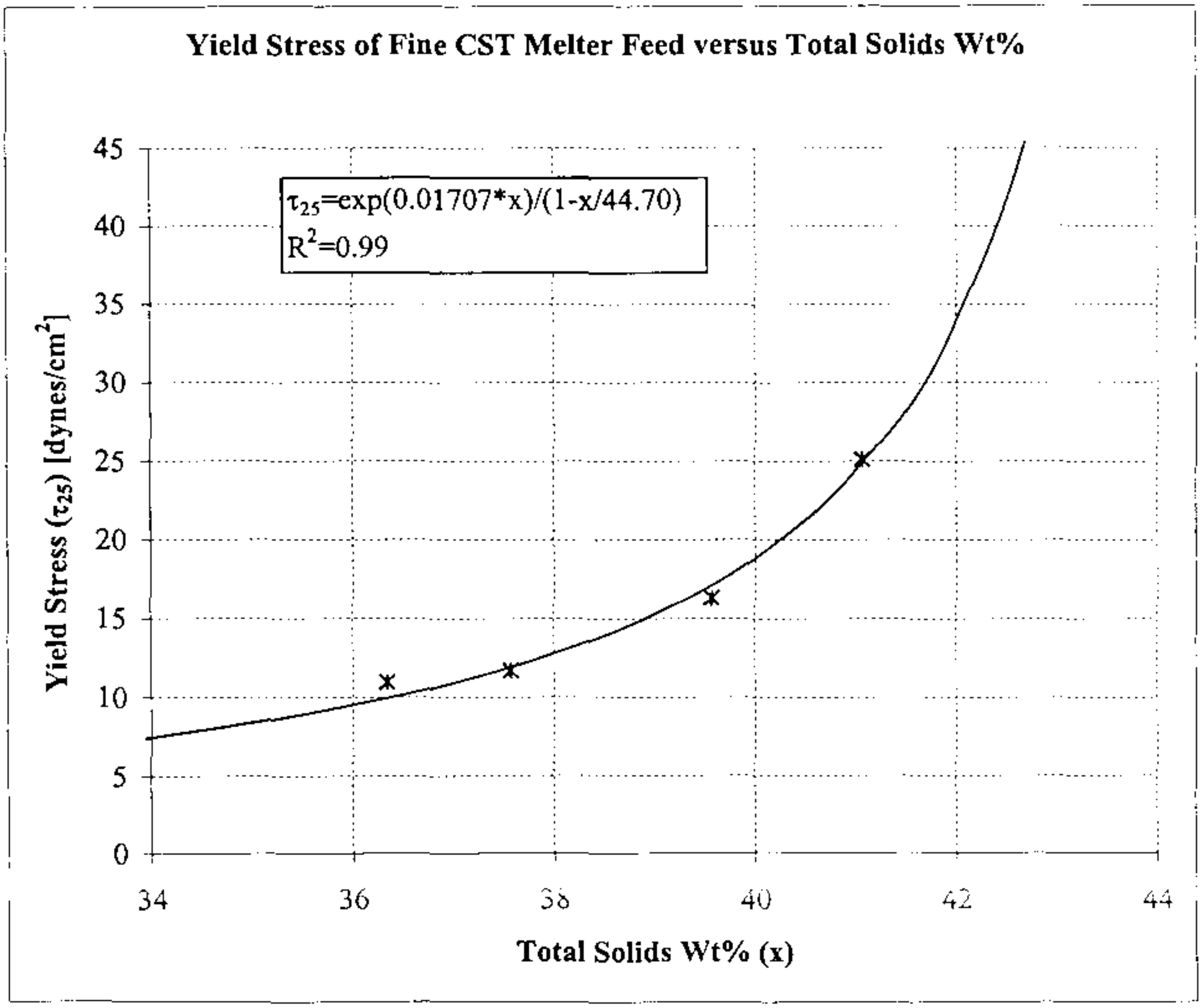

Figure 4. Yield Stress of Fine CST Melter Feed versus Total Solids Wt%

Rig Setup

Details of the experimental setup are contained in the attached run plan in Appendix B. In summary, a Servodyne Mixer with speed controller was used to stir the four melter feeds in a four-liter glass kettle at a constant speed. The impellers on the stirring shaft were spaced to match DWPF's volume only at a 1/13,000th scale or DWPF 8500 gallons set equivalent to TNX 2.5 liters. More details on the scaling are contained in the run plan in Appendix B. A 5-ml pipette was used to extract samples at specified intervals. A sketch of the experimental setup is contained in the run plan in Appendix B.

Mixer Speed Check

Before beginning the settling studies, the Servodyne mixer speed was checked to make sure the controller was maintaining the proper speeds. An attempt was made to use an IRD Mechanalysis Model 890 tachometer with optical strobe but the unit proved imprecise and inconsistent for the tests performed. Instead, visual checks of the mixer speed were performed and showed that the controller was operating correctly. A table of these speed checks is contained in the run plan in Appendix B.

Experimental Runs

Summarizing the experimental procedure, three settling runs were completed with various size CST as requested by the scoping document (HLE-TAR-98060, Appendix C). A fourth run was added to establish a baseline for comparison using sludge-only (without CST) melter feed. The four melter feeds used in the four settling runs are summarized Table II. The runs started with the melter feeds prepared in the hydrogen study (HLE-TAR-98060, Issue 21, Appendix C).

Table II. Settling Study Runs

Run	CST
Melter Feed without CST	None
Melter Feed with fine CST	Finely Ground CST treated with caustic
Melter Feed with mid-ground CST	Mid-ground CST treated with caustic
Melter Feed with unground CST	Unground CST treated with caustic

Four 2-liter scale melter feed settling runs were completed in the 772-T lab at TNX. In all of the runs, the same speed setting of 60 RPM was used on the Servodyne mixers. The Melter Feed contents were then allowed to settle over a period of 32 hours. Samples were taken at 1, 2, 4, 8, 25, 26, 29, and 32 hours from the initial start of mixing at 60 RPM. These samples were analyzed for weight percent solids to insure solids were settling. Other samples were taken at the same interval to be analyzed for elementals to determine the ratio of sludge, frit, and CST in the settled solids.

The first run consisted of a prototypic DWPF melter feed (Sludge-only) to validate the run procedure. As a result of this run, the procedures for all the settling studies were revised and are shown in the run plan in Appendix B. The sludge-only run showed that the mixer speed at which solids noticeably settled out was 60 RPM (higher speeds of 130 and 90 RPM showed no change in weight percent solids). Originally, only 5-ml samples were going to be pulled to do the weight percent solids analyses but the sludge-only run indicated this quantity was too small to provide consistent results so 10-ml samples were pulled.

The most information gained from the sludge-only run was on the Core sampling technique. As originally planned, the SRAT/SME glass sampler could not be used as the core sample would not stay in the sampler even with the upper end of the sampler closed. The SRAT/SME glass sampler did not work on the fine CST melter feed either. It was decided to go with a Coliwasa sampler as shown in the run plan in Appendix B. Even with the Coliwasa it was difficult to pull the core sample, i.e. the Coliwasa wanted to plug when the core sample was emptied into sample vials. Also, a Coliwasa core sample could not be pulled in back of the kettle (point 4) as the mixer clamp was in the way. Therefore, Coliwasa core samples were only taken at the right, front, and left sides of the kettle (points 1 to 3). Originally an additional sample point was to be the center of the vessel but due to the placement of the mixer and its impellers this point was not accessible. A closing comment on the core sampling is that the bottom samples pulled with the needle and syringe were difficult to obtain due to the thickness of the material. In many cases, several attempts had to be made to collect even a gram of material. In the future, a larger diameter needle is recommended.

After the procedure was validated for the sludge-only run, the other runs for the melter feeds with various sized CST resins were performed. Details of the experiments are contained in the run plan in Appendix B.

RESULTS

Particle Size Distribution

Three sizes of CST resin were used in preparing the melter feeds: Coarse or As-Received, Intermediate Ground, and Finely Ground. These different resins were sent to the lab for particle size analyses. Figure 5 shows the particle size distribution of the 3 CST resins in terms of volume % of resin. As can be seen from this figure, the Coarse CST contains the largest particles or 93 volume % of the Coarse CST particles are 352 microns or greater. The Fine CST particles are the next biggest with 62 volume % of the particles 352 microns or greater. Finally, the Intermediate CST particles are the smallest with 33 volume % of the particles 352 microns or greater. Another way to characterize the particle distributions is to average the three largest peaks for each CST group. The 3-peak average for the Coarse CST is 497 microns, the 3-peak average for the Fine CST is 398 microns, and the 3-peak average for the Intermediate CST is 266 microns. Again, the descending ordering of the CST groups by size are Coarse CST, Fine CST, and Intermediate CST. On the other hand if one looks only at particles less than 62 microns, the coarse has 0 volume %, the intermediate has 6 volume %, and the fine has 10 volume %. This observation shows that although the bulk of the fine CST particles are large, there are a greater number of small or fine particles in the fine CST. One explanation of what happened might be that the Fine CST was ground in a mortar and pestle where the material may have packed itself as it was being ground. The Intermediate CST was created by spreading the Coarse CST over a sieve (thus increasing the area of exposure) and pushing it through to the other side. In any case, the CST's used in this study do represent three distinctive sizes but their labeling may be misleading. This nomenclature will not be changed as all the run plans, lab notes, and lab analyses have used the existing labels. Please bear in mind that the Intermediate CST contains the smallest sized particles, the Fine CST contains the next largest particles, and the Coarse CST has the largest particles, based on average particle size.

Weight Percent Solids versus Time

As an indication that settling was taking place in the runs, the total weight percent solids for each of the four melter feeds was monitored over a 24 hour period. Samples were taken at 1, 2, 4, 8, 25, 26, 29, and 32 hours from the initial start of mixing at 60 RPM except for the first run or no CST melter feed. An initial sample used as a baseline was also taken before the experiment began and labeled as 0. The first run with the no CST melter feed was not required but done to validate the run procedure and provide a base-line set of data for comparison. This first run was sampled at 1, 2, 4, 19, 20, 22, and 26 hours due to the time constraints for completing the rest of the experiments.

For all feeds, total weight percent solids were run on each sample collected. The weight percent solids data for the no CST melter feed is shown in Table III while the data for the CST melter feeds is shown in Table IV. A plot of the weight percent solids of the melter feeds over time is shown in Figure 6. Notice that the weight percent solids generally rise during the first hours then drop off some point later. This behavior is explained by the same location that each sample was pulled (about center of the 2-liter slurry volume). Over time, as solids begin to settle, the weight percent solids for this center point will go up and then eventually fall as the solids go pass it. At some point later the solids level off or no more solids settle out as is evidenced by the last points in Figure 6.

An attempt was made to relate the different size CST feeds to the behavior of the weight percent solids. The weight percent solids of the Coarse CST with the largest particles rises the first hour but by the fourth hour has dropped and continues to drop until the 24 hour point where it essentially levels off. This response makes sense, as the largest particles should settle the quickest. Now the weight percent solids of the Fine CST or medium size particles rise slowly until the eighth hour and then drop off by the 24-hour point. The weight percent solids of the Intermediate CST with the smallest particles rise up to the fourth hour but drop off by the eighth hour. Based on the behavior of the weight percent solids, it appears that the Coarse CST feed contains the largest particles, followed by the Intermediate, then the Fine CST feed which takes the longest to settle. From the particle size analysis it would appear the Coarse CST feed has the largest particles followed by the Fine CST and then the Intermediate CST. What the data might be saying is that the settling depends more on the distribution or range of the particle sizes as the Fine CST feed has the broadest range of particle sizes. In any case, the three different sized CST feeds do have different settling properties based on the weight percent solids.

There are some interesting comparisons between the sludge-only feed and the CST feeds with regards to the weight percent solids. The Intermediate CST feed seems to settle very similar to the sludge-only feed especially for the first eight hours. The Coarse CST feed settles more quickly than the sludge-only feed while the Fine CST takes longer to settle. Since the sludge-only feed was sampled earlier than the CST feeds (19 hours versus 25 hours), the feed has essentially settled out at 19 hours. Since all the weight percent solids level out at the end, the sludge-only data suggests that the CST feeds also will settle out by 19 hours or earlier. If the

CST feeds weight percent solids for the last 4 sample periods (25, 26, 28, 32) are projected back to the sample periods for the sludge-only (19, 20, 22, 26) then one gets a chart like shown in Figure 7.

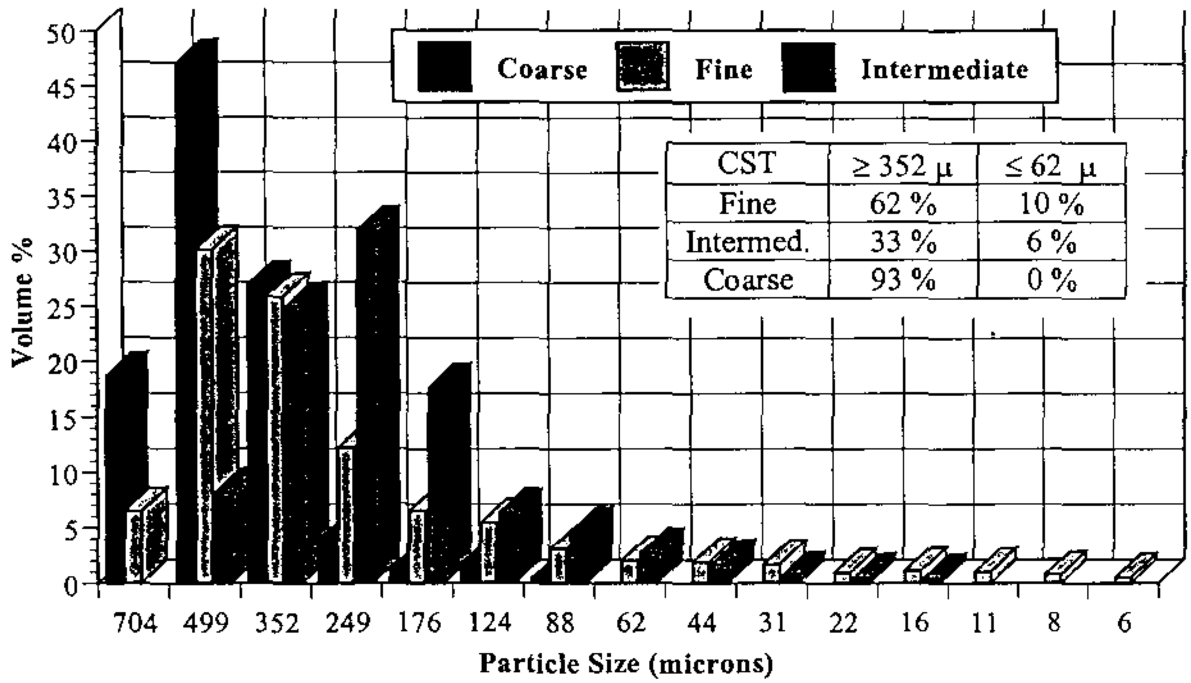

Figure 5. CST Particle Size Distribution

Table III. Weight Percent Solids of No CST Melter Feed over Time

Settling Time [hr]	No CST Total Solids Wt%
0	42.2
1	43.8
2	46.1
4	47.3
19	17.9
20	18.0
22	17.2
26	17.0

Table IV. Weight Percent Solids of CST Melter Feeds over Time

	Total Solids Weight %		
Settling Time [hr]	Coarse CST	Intermediate CST	Fine CST
0	41.9	42.5	39.4
1	43.2	43.6	41.9
2	43.9	46.0	42.6
4	40.3	48.5	42.4
8	36.6	34.9	43.4
25	15.7	16.0	15.3
26	16.0	15.2	15.2
28	15.0	16.4	15.0
32	14.7	14.9	14.9

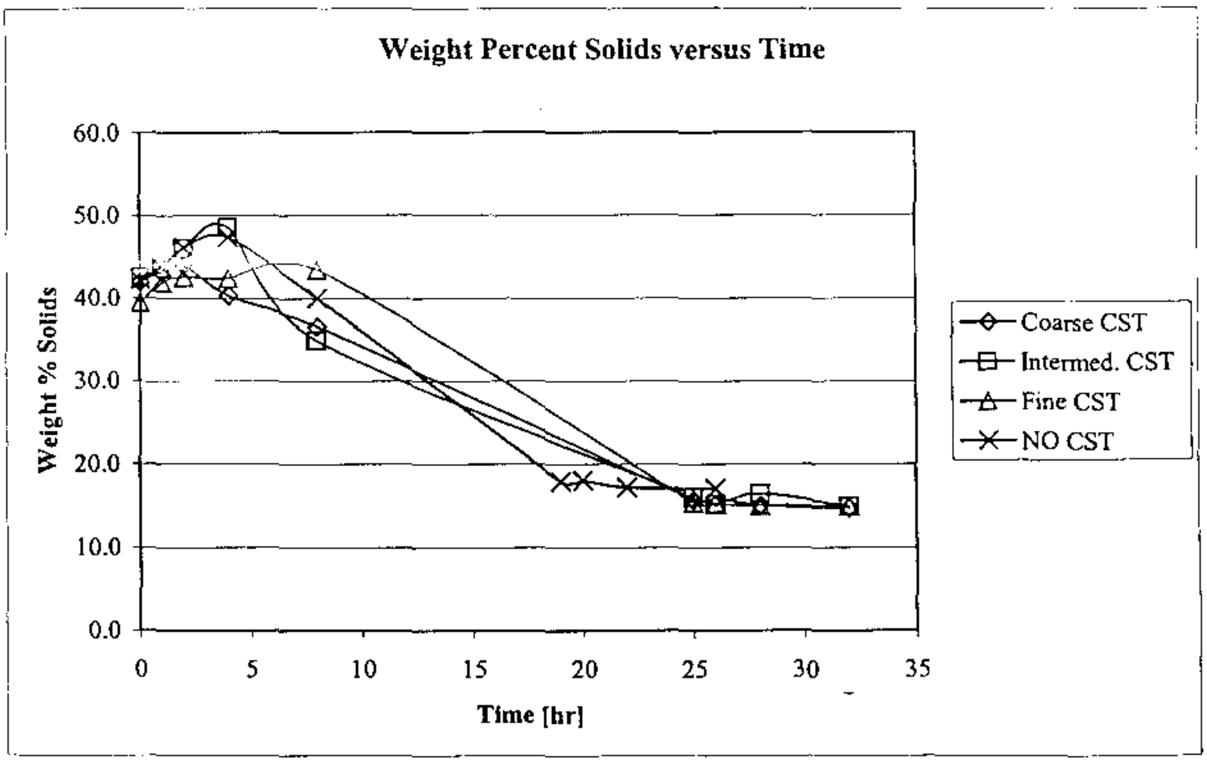

Figure 6. Weight Percent Solids versus Time for all Melter Feeds

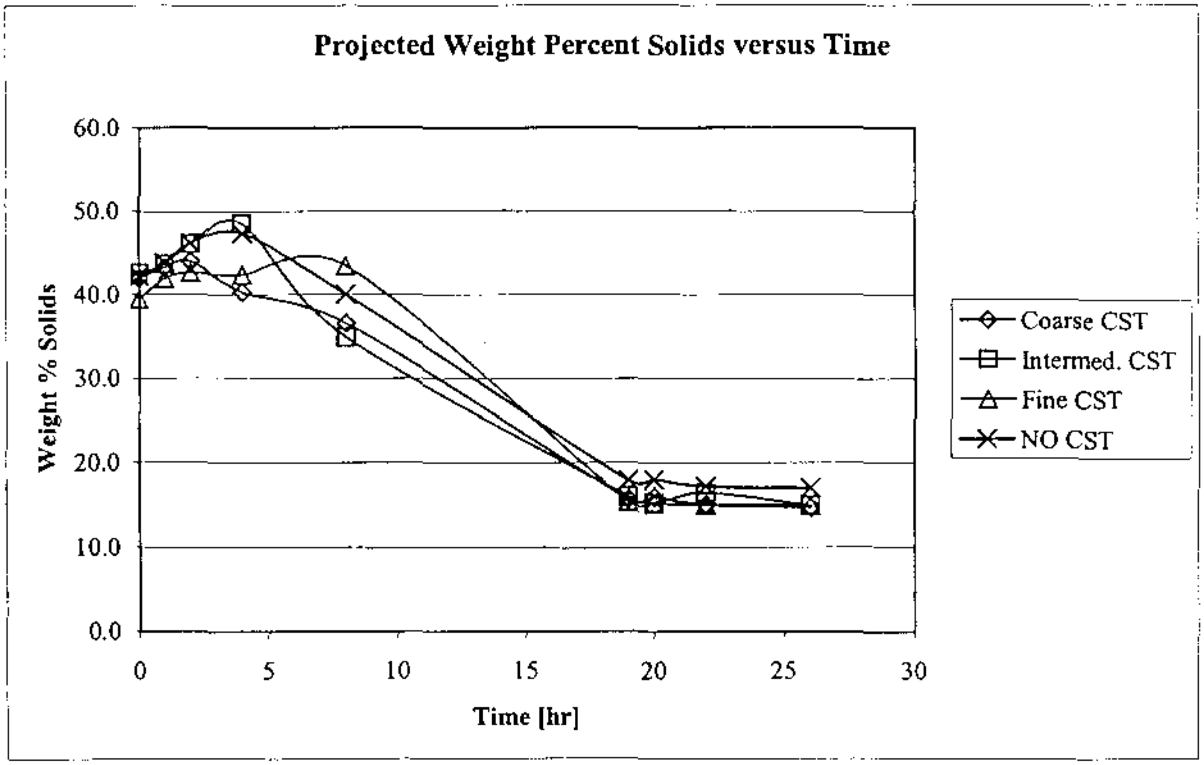

Figure 7. Projected Weight Percent Solids versus Time for all Melter Feeds

Settling Data

<u>*Samples taken at Set Intervals*</u>

To determine how the different components (sludge, frit, CST) in the melter feeds settled, unique elements were chosen to represent each component: Fe, Al, and Mn for sludge, Li for frit, and Ti for CST. Samples were then pulled from the same location (about center of the 2-liter vessel) at set intervals over a 32-hour period and then analyzed for the elements mentioned earlier. At the end of the 32-hour period, core samples were then taken and analyzed for the same elements. The elemental weight percent analyses for the settling samples for the No CST (Sludge-only), Coarse CST, Intermediate CST, and Fine CST melter feeds are listed in through Table I through Table IV, respectively, in Appendix A. Each table shows the analyses for the settling samples taken at the number of hours listed in the first column. Note that the zero point or first point is from an initial sample to establish a baseline for comparison.

In attempts to understand how the sampled elemental weight percents related to how solids settled, it was decided to relate the elemental weight percents to the amount of each primary component in the melter feeds. Using an oxide solids basis of 100 grams, each feed should consist of 64 grams of frit, 26 grams of sludge, and 10 grams of CST. The elemental iron (Fe) was associated with the sludge. The elemental titanium (Ti) was associated with the CST. The elemental lithium (Li) was associated with the frit. Due to the changing total weight percent solids, these elemental weight percents (Fe, Ti, Li) were adjusted by their corresponding total weight percent solids and then multiplied by their corresponding gravimetric factor (GF) to put them on an oxide basis. For example, for the Coarse CST melter feed, the zero point gets transformed as follows:

$$6.94 \text{ wt\% Fe}*41.9 \text{ wt\% solids}*1.43 \text{ GF of } Fe_2O_3 = 4.16 \text{ wt\% } Fe_2O_3$$

The elemental oxide weight percents were then ratioed to their corresponding component amounts to extract the amount of component still in solution based on the sample results. For example, for the Coarse CST feed the following relationship was used to deduce the amount of frit still in solution:

$$\frac{1.89wt\%Li_2O}{64gFrit} = \frac{0.80wt\%Li_2O}{x}$$

when solved for x gives 27 grams of frit are still in solution based on the sample taken. Since the desired goal is to see how CST settles with regards to frit, if 27 grams of frit are still in solution and initially there were 64 grams of frit, the amount of frit that has settled is calculated like:

$$64 \text{ g} - 27 \text{ g} = 37 \text{ g frit settled}$$

To put these settled quantities on a comparable basis, it was decided to look at what percentage of each component had settled at each sample point. For example, again looking at the Coarse CST feed, the amount of frit that had settled by the first hour can represented as:

$$\frac{37g}{64g}*100 = 57.8\%$$

Which means in the first hour, 57.8% of the frit has settled out as indicated by the samples. The settled masses and percents are shown in Table V through Table VIII for the No CST, Coarse CST, Intermediate CST, and Fine CST feeds, respectively. Plots of the percent settled values are shown in Figure 8 through Figure 11 for the No CST, Coarse CST, Intermediate CST, and Fine CST feeds, respectively.

For the No CST melter feed, before the 19^{th} hour sample, more sludge has settled (55%) than frit (44%) but after 19 hours more frit (100%) has settled than sludge (81%). Due to the sampling error these results most likely mean the sludge and frit settle at slightly different rates initially but as time passes, more of the frit falls out than the sludge.

For the coarse CST melter feed, the settling results are inconclusive. Based on the 1^{st} hour sample, more frit (58%) has settled than coarse CST (38%) but by the second hour, more CST (72%) has settled compared to the frit (59%). However, this condition reverses itself by the 8^{th} hour or more frit (73%) has settled than the coarse CST (66%). These swings may be in part due to the sampling error but also to the changes in the total weight percent solids. Remember that the total weight percent solids go up then down and level off sometime after the 8^{th} hour sample. These results may also be due to the range or distribution of particles in the coarse CST. The heavier or larger particles drop first then the smaller particles drop at such a rate that the coarse CST weight percent appears to go up as the particles pass through the sample zone.

Based on the Intermediate CST melter feed samples, the frit settles more quickly than the CST. Looking at the data in Figure 10 for the first four hours, the amount of CST that settles is always less than the amount of frit that has settled (59% frit versus 45% CST, 53% frit versus 34% CST, and 47% frit versus 30% CST). These results support the particle size analyses that the Intermediate CST actually has the greatest amount of smaller sized particles which take longer to settle out. Note that given enough time, greater than 8 hours in these experiments, all the frit and intermediate CST eventually settle out.

For the Fine CST melter feed, the frit and CST appear to settle at about the same rate. This result makes sense as the Fine CST really had the largest amount of intermediate sized particles. Although the data presented in Figure 11 may indicate that the Fine CST settles a little faster than the frit, the errors in sampling and analytical techniques prevent one from drawing this conclusion. Instead, the Fine CST appears to settle at least as fast as the frit.

Table V. Settled Components in No CST (Sludge-Only) Melter Feed

Sample Time	Settled Sludge [g]	Settled Frit [g]	% Sludge Settled	% Frit Settled
0	0.0	0.0	0.0	0.0
1	15.2	37.1	58.5	50.1
2	14.6	38.3	56.2	51.8
4	14.3	32.3	55.0	43.7
19	21.1	72.0	81.3	97.3
20	21.2	73.1	81.5	98.8
22	21.4	73.8	82.4	99.7
26	21.7	73.8	83.6	99.8

Table VI. Settled Components in Coarse CST Melter Feed

Sample Time	Settled Sludge [g]	Settled Frit [g]	Settled CST [g]	% Sludge Settled	% Frit Settled	% CST Settled
0	0.0	0.0	0.0	0.0	0.0	0.0
1	15.0	37.0	3.8	57.7	57.8	38.1
2	15.2	37.6	7.2	58.4	58.7	71.7
4	16.0	38.8	4.8	61.5	60.6	48.0
8	16.4	46.4	6.6	63.0	72.5	66.3
25	22.3	63.9	10.0	85.8	99.8	99.7
26	22.5	63.9	10.0	86.6	99.8	99.7
28	22.7	63.9	10.0	87.3	99.8	99.8
32	22.9	63.9	10.0	87.9	99.8	99.8

Table VII. Settled Components in Intermediate CST Melter Feed

Sample Time	Settled Sludge [g]	Settled Frit [g]	Settled CST [g]	% Sludge Settled	% Frit Settled	% CST Settled
0	0.0	0.0	0.0	0.0	0.0	0.0
1	14.1	38.0	4.5	54.2	59.4	45.3
2	13.8	33.8	3.4	53.0	52.8	34.4
4	13.3	30.3	3.0	51.2	47.4	30.2
8	14.9	58.6	8.6	57.2	91.5	86.3
25	22.0	62.7	9.7	84.6	98.0	96.7
26	22.1	63.9	9.9	85.1	99.8	98.7
28	22.0	62.9	9.7	84.8	98.3	97.0
32	22.6	63.7	9.9	87.0	99.6	98.6

Table VIII. Settled Components in Fine CST Melter Feed

Sample Time	Settled Sludge [g]	Settled Frit [g]	Settled CST [g]	% Sludge Settled	% Frit Settled	% CST Settled
0	0.0	0.0	0.0	0.0	0.0	0.0
1	15.7	34.0	5.6	60.4	53.1	55.8
2	15.4	34.6	6.3	59.4	54.1	62.5
4	15.2	36.4	6.9	58.3	56.9	69.3
8	14.9	33.4	5.8	57.3	52.2	58.4
25	22.4	63.9	9.7	86.0	99.8	97.5
26	22.4	63.9	9.7	86.3	99.8	97.4
28	22.6	63.9	9.8	86.8	99.8	97.6
32	22.7	63.9	9.8	87.1	99.8	97.8

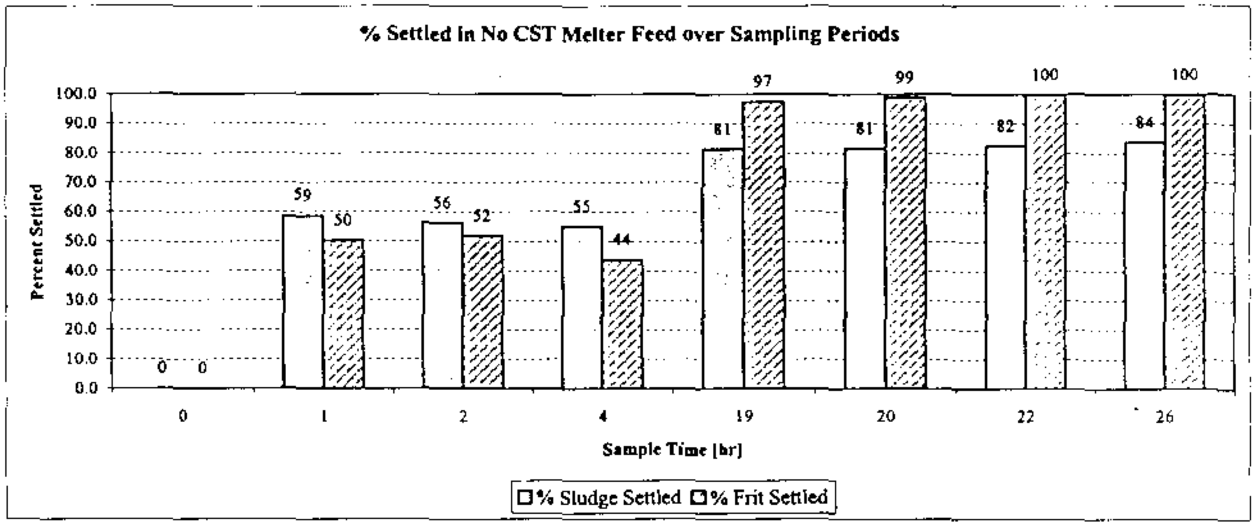

Figure 8. Percent Settled Components in No CST Melter Feed

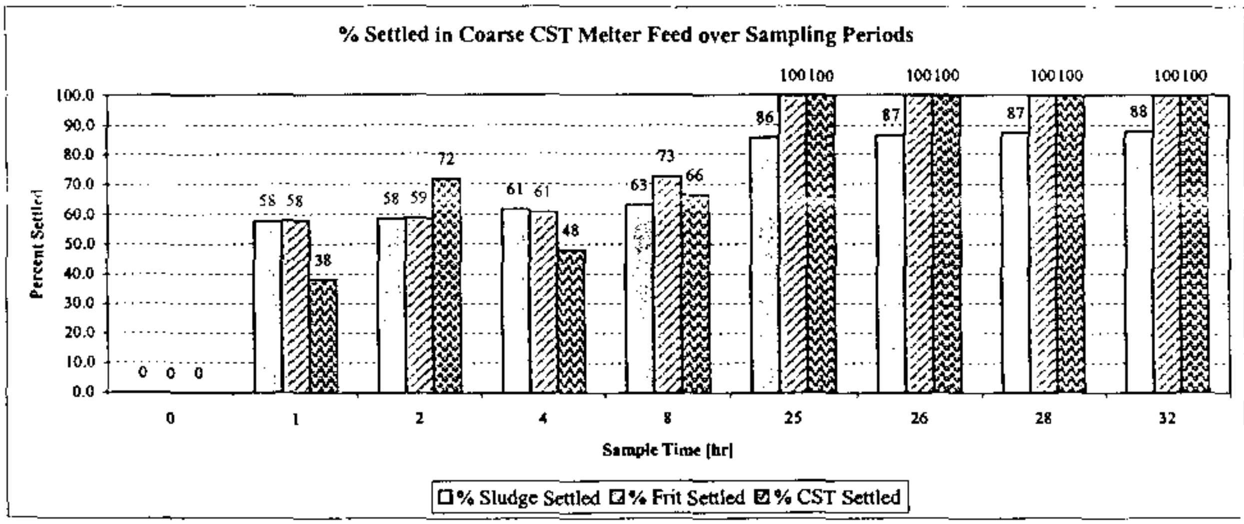

Figure 9. Percent Settled Components in Coarse CST Melter Feed

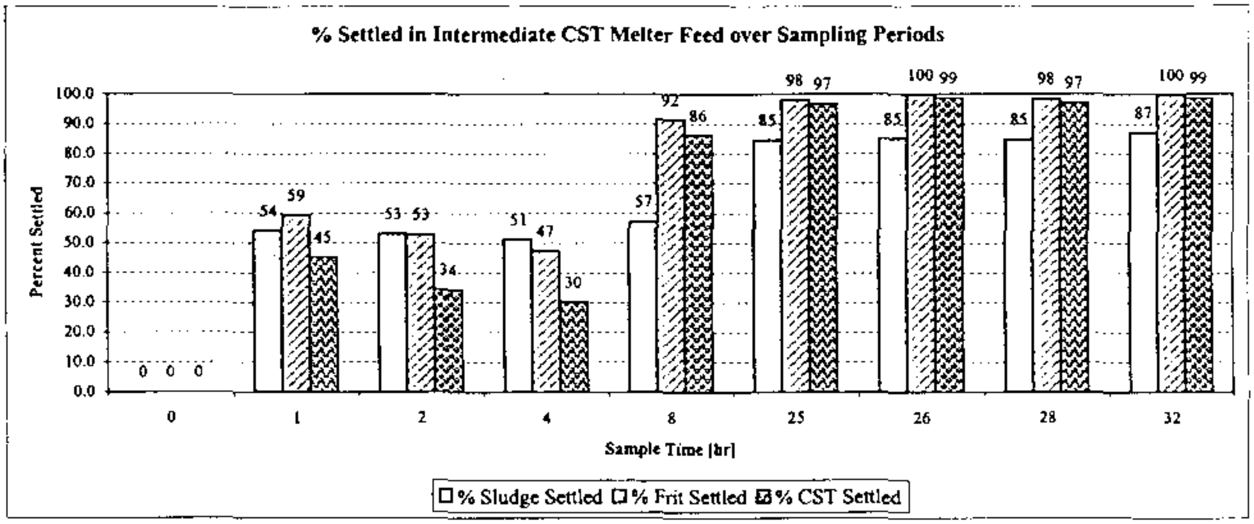

Figure 10. Percent Settled Components in Intermediate CST Melter Feed

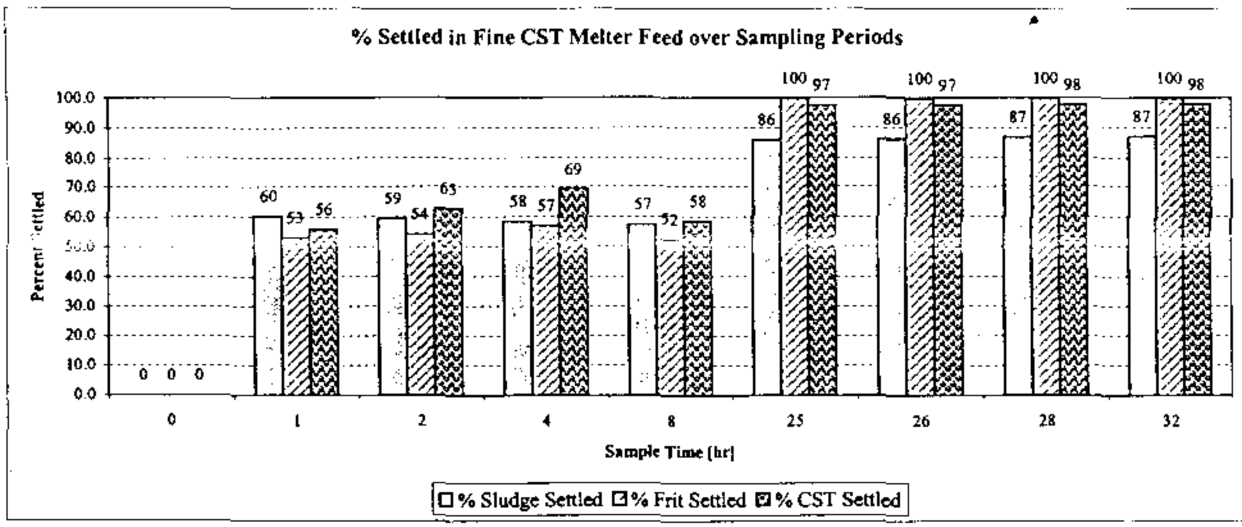

Figure 11. Percent Settled Components in Fine CST Melter Feed

<u>*Core Samples Taken at the End*</u>

To further check what settles in the melter feeds, an attempt was made to collect core samples at the end of each run directly from the bottom of the vessel with a syringe and needle (#-N) and with a Coliwasa (#-1, #-3, #-5). The first number in the core sample ids (1-#, 2-#, 3-#, 4-#) indicates where they were taken in the vessel. Point 1 was to the right, point 2 was in front, point 3 was to the left, and point 4 was in the back. An outline of the points is given in the run plan in Appendix B. More details are contained in the run plan in Appendix B. The elemental analyses for these samples are shown in Table IX. Note the analyses are ordered by the needle sample (#-

N) first or the very bottom sample, then the Coliwasa first inch sample (#-1), third inch sample (#-3), and fifth inch sample (#-5).

Due to the difficulty in obtaining the needle and Coliwasa samples, the numbers are presented more for completeness than analysis. The needle syringe may have biased the weight percent results more toward finer particles. The Coliwasa samples may be biased low or high in solids depending on if the end of the tube became partially blocked by the solids. The information does seem to indicate that for the Coarse, Intermediate, and Fine CST feeds, there are more solids at the bottom than at the upper levels. To draw more precise results would require a more accurate sampling technique.

Table IX. Core Sample Weight Percent Analyses

Sample Id	Sludge	Sludge	Sludge	Frit	CST
MFP	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
1-N	2.96	0.948	0.82	0.266	0.016
3-1	2.99	0.866	0.894	1.11	0.018
3-3	2.9	0.953	0.779	0.099	0.016
3-5	3.19	0.975	0.872	0.692	0.019
MFP-C	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
4-N	3.17	0.82	1.13	1.12	0.472
1-1	2.87	0.823	0.861	0.878	0.568
1-3	2.57	0.622	0.813	0.195	0.013
1-5	2.56	0.665	0.784	0.248	0.115
MFP-I	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
4-N	3.56	0.978	0.91	1.32	0.748
1-1	3.54	0.976	0.941	1.33	0.853
1-3	3.01	0.928	0.886	0.855	0.746
1-5	2.53	0.699	0.772	0.177	0.258
MFP-F	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
1-N	2.98	1.12	0.932	1.38	1.57
3-1	2.9	0.758	0.852	0.596	0.465
3-3	2.47	0.612	0.695	0.055	0.113
3-5	2.72	0.812	0.803	1.29	0.657
MFP-C	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
4-N	3.17	0.82	1.13	1.12	0.472
1-1	2.87	0.823	0.861	0.878	0.568
1-3	2.57	0.622	0.813	0.195	0.013
1-5	2.56	0.665	0.784	0.248	0.115

CONCLUSIONS

For the CST melter feeds, the Coarse CST consisted of the largest particles (93 volume % ≥ 352 μ) while the Intermediate CST had the smallest sized particles (33 volume % ≥ 352 μ). The Fine CST particles were somewhere in between (62 volume % ≥ 352 μ). The settling results showed that no discernable settling pattern could be found for the coarse CST. However, the Intermediate CST (smallest sized particles) appears to settle slower than the frit and the Fine CST (intermediate sized particles) settles at about the same rate as the frit.

Given the problems with the core sampling, no conclusions can be reached about the quantities of frit versus CST that reside on the bottom of the vessel. As seen from the settling samples taken at different time periods, the amount of CST that settles during a certain time can go up or down depending on the size of the CST. In turn, one would expect the amount of CST and frit on the bottom to change, i.e. sometimes more frit than CST rests on the bottom and sometimes more CST than frit exists on the bottom. Larger scaled experiments with more precise core sampling techniques need to be performed to characterize how CST and frit settle on the bottom of a vessel.

FUTURE WORK

Due to the scale of this scoping study and the variability in sampling and analytical techniques, a more precise study needs to be performed to better quantify the settling differences between CST and frit. One suggestion would be to perform a settling study on a DWPF scale using the full scale SRAT at TNX or on a 1/240th scale using the pilot system at 786-A. Using a larger scale would allow better matching on mixing characteristics, such as mixer speeds and impeller shapes. A larger scale would allow more consistent and easier sampling for both the settled solids and core samples.

This study does show that there are settling differences between the different sized CST and frit. Future experimentation should try to match DWPF's scale and mixing characteristics more closely, devise a more accurate method for extracting settling samples at more frequent times, and build a core sampler that is easy to use and properly segregates the layers at the bottom.

ACKNOWLEDGMENTS

Thanks to Roy Jacobs and Joe Carter for providing quick answers to our questions and direction as needed.

Thanks to Frances Williams, Sammie King, John Duvall, Mary Johnson, Vickie Williams for their help in preparing for and performing the experiments. Their work is very much appreciated. I appreciate the extra hours and long weekends the technicians worked to complete these experiments.

I would also like to thank the excellent technical support (as always) from Dan Lambert, Paul Monson, and Russ Eibling. Especially Russ Eibling contribution on deriving the rheology curves for the different melter feeds.

Thanks to William Ryan for getting the mixer controllers in and Nick Odom for assisting with the mixer controller speed check.

We appreciated the quick turnaround and accurate analyses that were reported by the Mobile lab.

Table I. Settling Elemental Weight Percent Analyses for No CST (Sludge-Only) Feed

Sample Id	Sludge	Sludge	Sludge	Frit	CST
MFP	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
0	7.16	1.88	1.96	2.31	0.023
1	2.86	0.843	0.798	1.11	0.018
2	2.87	0.832	0.834	1.02	0.017
4	2.87	0.826	0.838	1.16	0.019
19	3.16	0.965	0.822	0.147	0.018
20	3.11	0.999	0.853	0.067	0.017
22	3.1	0.98	0.815	0.015	0.017
26	2.92	0.948	0.791	0.014	0.017

Table II. Settling Elemental Weight Percent Analyses for Coarse CST Feed

	Sludge	Sludge	Sludge	Frit	CST
MFP-C	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
0	6.94	1.85	1.92	2.10	1.34
1	2.85	0.891	0.885	0.857	0.805
2	2.75	0.73	0.828	0.825	0.361
4	2.78	0.866	0.818	0.859	0.725
8	2.94	0.832	0.832	0.66	0.517
25	2.64	0.637	0.737	0.012	<0.010
26	2.44	0.608	0.826	0.01	<0.010
28	2.45	0.601	0.711	0.01	<0.010
32	2.39	0.597	0.75	0.011	<0.010

Table III. Settling Elemental Weight Percent Analyses for Intermediate CST Feed

Sample Id	Sludge	Sludge	Sludge	Frit	CST
MFP-I	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
0	6.41	1.87	1.89	2.08	1.50
1	2.86	0.901	0.858	0.822	0.797
2	2.78	0.911	0.767	0.905	0.906
4	2.74	0.91	0.747	0.957	0.914
8	3.34	0.842	0.86	0.214	0.25
25	2.62	0.674	0.754	0.11	0.131
26	2.66	0.66	0.766	0.012	0.056
28	2.52	0.678	0.738	0.089	0.117
32	2.37	0.626	0.766	0.025	0.061

Table IV. Settling Elemental Weight Percent Analyses for Fine CST Feed

Sample Id	Sludge	Sludge	Sludge	Frit	CST
MFP-F	Fe (S)	Mn (S)	Al (S)	Li (F)	Ti (C)
0	6.95	1.91	1.95	2.04	1.51
1	2.59	0.778	0.788	0.899	0.626
2	2.61	0.74	0.798	0.865	0.522
4	2.69	0.717	0.78	0.815	0.429
8	2.69	0.769	0.785	0.882	0.568
25	2.49	0.627	0.691	<0.010	0.098
26	2.48	0.618	0.692	0.011	0.101
28	2.4	0.622	0.664	0.01	0.095
32	2.36	0.608	0.668	<0.010	0.087

SRT-PTD-98-0036, Rev. 0

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August 21, 1998

TO: L. F. Landon, 704-1T

FROM: W. E. Daniel, 704-1T

Run Plan for ITP Alternatives Task 13
Examine Relative Settling Behavior of the CST Resin in DWPF Melter Feed (U)

One alternative ITS is looking into is the use of non-elutable CST resin to remove Cs-137 from the supernate of SRS High Level Waste. ITS has been tasked to find out if the settling behavior of the CST resin differs from the current DWPF glass former (frit) used in the melter feed preparation (HLE-TAR-98060, 7/20/98). If the settling behavior is different, it could impact the slurry homogeneity and in turn affect the Waste Acceptance criteria. This run plan is based on the scope of work and task assignment document SRT-PTD-98-032 that calls for completion of this work by September 21, 1998. The experimental work for this task will be controlled by the Laboratory Scale Chemical Process Cell Simulations (Manual L27, Procedure 2.02) and this run plan. This run plan includes details for conducting a series of settling experiments as well as the steps leading up to the experiments.

1) These experiments will use the melter feed products from earlier runs made for studying the impact of the CST resin on the hydrogen generation during the SRAT/SME cycles.[1] These hydrogen runs used a Tank 42 sludge simulant prepared from the Optima prepared Tank 51 simulant. This Tank 42 sludge simulant was prepared to be about 20 weight percent solids with H-modified (HM) purex levels of noble metals and mercury. From this sludge four SRAT/SME runs were made with no CST resin and three different CST resin sizes (as received, intermediate ground, finely ground). The CST resin was also treated with 1 M NaOH. These SRAT/SME runs were designed to produce a glass containing 10 weight percent CST, 26 weight percent calcined sludge solids, and 64 weight percent frit 202.
2) The next task was to establish a procedure for adjusting the melter feeds to a yield stress of 25 dynes/cm^2 or the lowest DWPF design basis yield stress at 25°C. This lower yield stress will promote

[1]Run Plan for ITP Alternatives Task 21: Measure Hydrogen Generation During Coupled Sludge/CST SRAT/SME Cycles (U), Dan Lambert, SRT-PTD-98-0033, Rev. 1, August 11, 1998.

segregation. In order to develop this procedure, preliminary rheology tests were run on similar sludge-only melter feed but without CST. These results were fitted to the following model:

$$\tau = \frac{e^{Ax}}{1 - \frac{x}{B}}$$

where τ is the yield stress in dynes/cm^2, x is the total weight percent solids, and A and B are fitted parameters where B represents the maximum weight percent solids of the slurry. This equation is well documented in previous reports.[2,3] For the preliminary sludge-only melter feed the fitted parameters were determined to be A = 0.02243 and B = 49.06. The measured and predicted results are shown in **Table I** and a plot of the values are shown in **Figure 1**. These initial tests led to the procedure "Melter Feed Yield Stress Adjustment" shown in Appendix A. The first part of the procedure gathers rheology data for fitting the appropriate yield stress model as a function of total weight percent solids of a particular SME product. The weight percent solids is adjusted by adding different amounts of de-ionized water. Once this model is derived, it is used to adjust the yield stress of the remainder melter feed product in preparation for the settling runs. There are four procedure listings for each of the four SME products: 1) without CST, 2) with As-Received or Coarse CST, 3) with Intermediate ground CST, and 4) Finely ground CST.

3) Perform the settling studies as outlined in the procedures shown in Appendix B. There are four procedure listings for each of the four SME products: 1) without CST, 2) with As-Received CST, 3) with Intermediate ground CST, and 4) Finely ground CST. Note that the placement of impellers on the mixing shaft was based on the scaling calculations shown in **Table II**. The TNX 4 liter vessel has an inside diameter of 5 inches and the simple relationship $\pi R^2 \bullet$ height = volume was used to derive the respective heights of the impellers. Due to the size of the scaling factor (13000), the bottom impeller was placed at the end of the shaft and the other impeller was placed 2.5 inches above it. The stirring shaft is then placed 0.25 inches off the bottom of the vessel to try to match the true scale but still leave a safe enough distance for the impeller and shaft.

4) The experiments are to be performed no later than the week of September 7, 1998. The report is planned to be issued by September 21, 1998.

Table I. Preliminary Rheology Tests on No-CST Melter Feed

Total Wt% Solids	Measured Yield Stress [dynes/cm^2]	Predicted Yield Stress [dynes/cm^2]
44.64	30.60	30.20
43.48	22.10	23.31
41.84	18.70	17.37
40.68	13.70	14.58
39.86	13.50	13.04

[2] Rheology Measurements of Simulated Slurry Mix Evaporator Material (U), James C. Marek, WSRC-TR-97-00343, October 17, 1997.
[3] Rheology of SME Product from Alternative Sludge-Only Flowsheet with Batch One Sludge Simulant (U), James C. Marek, WSRC-TR-96-0179, June 26, 1996.

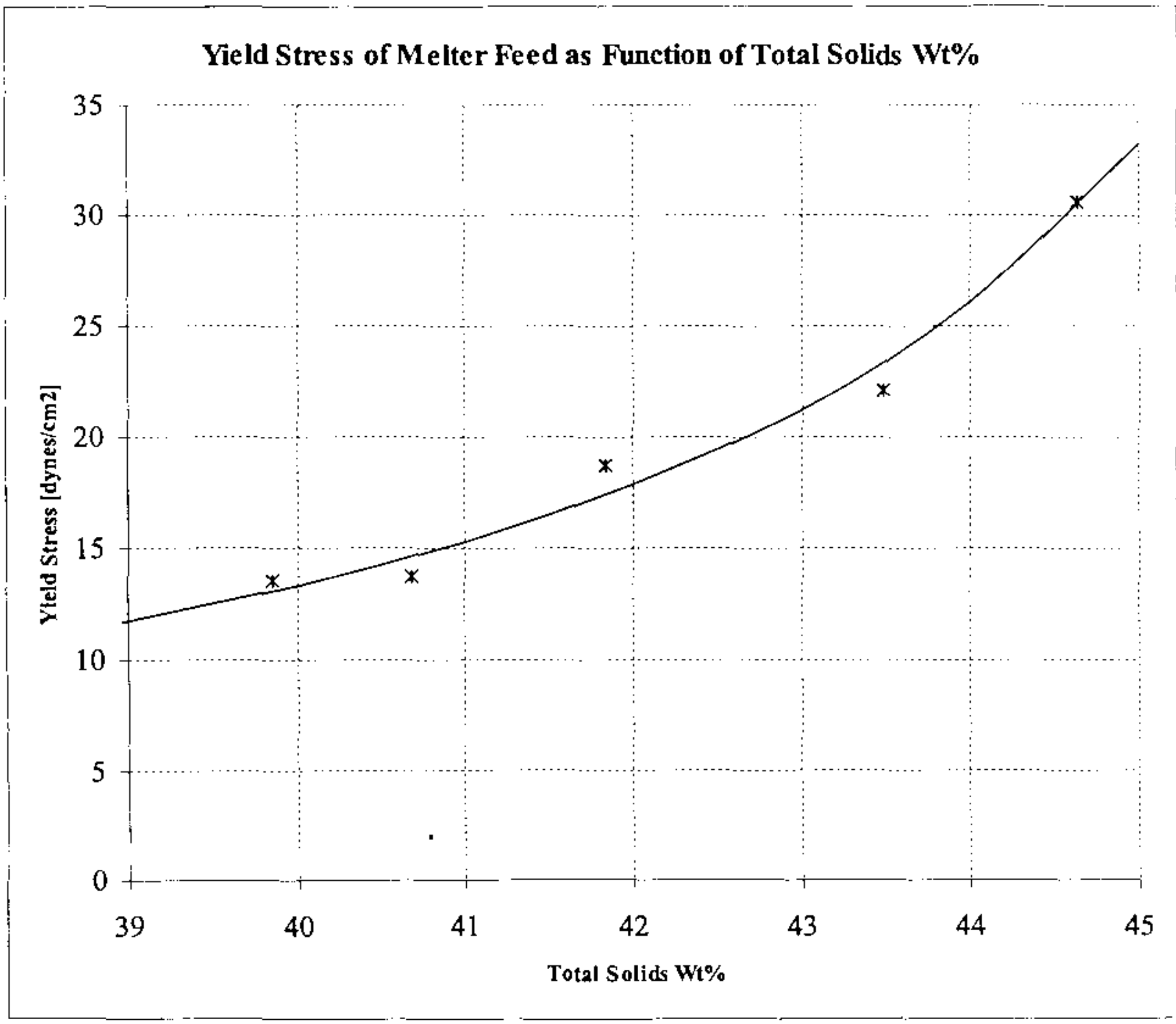


Figure 1. Yield Stress as a function of total weight percent solids for No-CST melter feed

Table II . Scaling Calculation For Impeller Placement

Scale: DWPF 8500 gal = TNX 2.5 L (0.66 gal)	SRAT/SME volume	TNX 4L volume	TNX 4L volume	TNX 4L height (desired)
1 foot above top of top impeller	4802 gal	0.3694 gal		
top of top impeller	3982 gal	0.3063 gal		
bottom of top impeller	3771 gal	0.2901 gal	67.013 in^3	3.413 in
1 foot above top of bottom impeller	1757 gal	0.1352 gal		
top of bottom impeller	~937 gal	0.0721 gal	16.655 in^3	0.848 in
bottom of bottom impeller	~271 gal	0.0209 gal	4.828 in^3	0.008 in

1. ☑ Take the Sludge-Only Melter Feed or SME product **without CST**, mix it well, and draw off four 120 ml samples into four tared 125 ml bottles. These samples will be referred to as MFP-1, MFP-2, MFP-3, and MFP-4.

2. ☑ Record the initial weights of the five MFP samples:

MF Product Sample	Initial MF Sample Weight [g]
MFP-1	not recorded
MFP-2	126.21
MFP-3	121.09
MFP-4	120.46

3. ☑ Weigh out the following amounts of water and add them to the appropriate MF product samples while recording their weights.

MF Product Sample	Amount of Water to Add [g]	Amount of Water Added [g]	Final Weight of MF Sample with Water [g]
MFP-2	5	5.01	131.22
MFP-3	10	10.00	131.09
MFP-4	15	15.00	135.46

4. ☑ Run total solids weight percent and density in duplicate on the first MF Product sample MFP-1.
5. ☑ Mix the MF Product Samples 1-4 well and run rheology or yield stress at 25°C on all of them and be sure to save the results on the Rheology computer.
6. ☑ Report the Rheology results to Gene Daniel and Russ Eibling for analysis and yield stress model development.
7. ☑ Using the yield stress model, predict the amount of water to add to achieve a yield stress of 25 dynes/cm^2 for the melter feed product of step 1.

   ✓ Predicted Amount of water to add __0__ g

8. ☑ Add the predicted amount of water from the previous step and record:

Initial MF Sample Weight [g]	Amount Of Water Added [g]	Final Weight of MF Sample [g]	Resulting Yield Stress [dynes/cm^2]
2623.2	0	2632.2	24.4[4]

9. ☑ If yield stress of the SME sample of the previous step is 25 dynes/cm^2 ± 2.5 dynes/cm^2 then proceed to the settling study. If the yield stress does not meet this spec then use the new data point to refine the yield stress model and repeat steps 6, 7, and 8 recording the new data until the desired yield stress is reached.

---

[4] **Initial Sludge-Only Melter Feed at 43.68 wt% total solids had a measured yield stress of 24.4 dynes/cm^2 which was close enough to the target so no water dilution was necessary.**

1. ☑ Take the Melter Feed or SME product with **Coarse CST (as-received)**, mix it well, and draw off four 120 ml samples into four tared 125 ml bottles. These samples will be referred to as MFP-1-C, MFP-2-C, MFP-3-C, and MFP-4-C.

2. ☑ Record the initial weights of the five MFP samples:

MF Product Sample	Initial MF Sample Weight [g]
MFP-1-C	129.9
MFP-2-C	120.5
MFP-3-C	123.5
MFP-4-C	131.2

3. ☑ Weigh out the following amounts of water and add them to the appropriate MF product samples while recording their weights.

MF Product Sample	Amount of Water to Add [g]	Amount of Water Added [g]	Final Weight of MF Sample with Water [g]
MFP-2-C	5	5.01	125.51
MFP-3-C	10	10.00	133.5
MFP-4-C	15	15.01	146.21

4. ☑ Run total solids weight percent and density in duplicate on the first MF Product sample MFP-1-C.
5. ☑ Mix the MF Product Samples 1-4 well and run rheology or yield stress at 25°C on all of them and be sure to save the results on the Rheology computer.
6. ☑ Report the Rheology results to Gene Daniel and Russ Eibling for analysis and yield stress model development.
7. ☑ Using the yield stress model, predict the amount of water to add to achieve a yield stress of 25 dynes/cm^2 at 25°C for the melter feed product of step 1.

   ✓ Predicted Amount of water to add 98.1 (55.7)[5] g

8. ☑ Add the predicted amount of water from the previous step and record:

Initial MF Sample Weight [g]	Amount Of Water Added [g]	Final Weight of MF Sample [g]	Resulting Yield Stress[6] [dynes/cm^2]
2549.7 (2635.1)[5]	98.1 (55.7)[5]	2647.8 (2690.8)[5]	32.4 (20.9)[5]

9. ☑ If yield stress of the SME sample of the previous step is 25 dynes/cm^2 ± 2.5 dynes/cm^2 then proceed to the settling study. If the yield stress does not meet this spec then use the new data point to refine the yield stress model and repeat steps 6, 7, and 8 recording the new data until the desired yield stress is reached.

[5] Did second water addition because targeting 41.8 wt% total solids for a predicted yield stress of 25 dynes/cm^2 and second dilution undershot due to variability in measurements and yield stress prediction curve.
[6] Predicted Yield Stress from Wt% Total Solids Curve developed in Step 6.

1. ☑ Take the Melter Feed or SME product with **Intermediate Ground CST**, mix it well, and draw off four 120 ml samples into four tared 125 ml bottles. These samples will be referred to as MFP-1-I, MFP-2-I, MFP-3-I, and MFP-4-I.

2. ☑ Record the initial weights of the five MFP samples:

MF Product Sample	Initial MF Sample Weight [g]
MFP-1-I	128.8
MFP-2-I	120.8
MFP-3-I	117.7
MFP-4-I	123.1

3. ☑ Weigh out the following amounts of water and add them to the appropriate MF product samples while recording their weights.

MF Product Sample	Amount of Water to Add [g]	Amount of Water Added [g]	Final Weight of MF Sample with Water [g]
MFP-2-I	5	5.00	125.8
MFP-3-I	10	10.00	127.7
MFP-4-I	15	15.01	138.11

4. ☑ Run total solids weight percent and density in duplicate on the first MF Product sample MFP-1-I.
5. ☑ Mix the MF Product Samples 1-4 well and run rheology or yield stress at 25°C on all of them and be sure to save the results on the Rheology computer.
6. ☑ Report the Rheology results to Gene Daniel and Russ Eibling for analysis and yield stress model development.
7. ☑ Using the yield stress model, predict the amount of water to add to achieve a yield stress of 25 dynes/cm^2 at 25°C for the melter feed product of step 1.

✓ Predicted Amount of water to add 140.9 (19.9)[7] g

8. ☑ Add the predicted amount of water from the previous step and record:

Initial MF Sample Weight [g]	Amount Of Water Added [g]	Final Weight of MF Sample [g]	Resulting Yield Stress [dynes/cm^2]
2652.1 (2753.5)[7]	140.9 (19.9)[7]	2793 (2773.4)[7]	29.1 (26.5)[7]

9. ☑ If yield stress of the SME sample of the previous step is 25 dynes/cm^2 ± 2.5 dynes/cm^2 then proceed to the settling study. If the yield stress does not meet this spec then use the new data point to refine the yield stress model and repeat steps 6, 7, and 8 recording the new data until the desired yield stress is reached.

[7] **Did second water addition because targeting 41.4 wt% total solids for a predicted yield stress of 25 dynes/cm^2 and due to variability seen in dilution of Coarse CST feed, cut second water dilution in half.**

1. ☑ Take the Melter Feed or SME product with **<u>Finely Ground CST</u>**, mix it well, and draw off four 120 ml samples into four tared 125 ml bottles. These samples will be referred to as MFP-1-F, MFP-2-F, MFP-3-F, and MFP-4-F.

2. ☑ Record the initial weights of the five MFP samples:

MF Product Sample	Initial MF Sample Weight [g]
MFP-1-F	117.9
MFP-2-F	118.7
MFP-3-F	119.0
MFP-4-F	117.1

3. ☑ Weigh out the following amounts of water and add them to the appropriate MF product samples while recording their weights.

MF Product Sample	Amount of Water to Add [g]	Amount of Water Added [g]	Final Weight of MF Sample with Water [g]
MFP-2-F	5	5.02	123.72
MFP-3-F	10	9.99	128.99
MFP-4-F	15	15.02	132.12

4. ☑ Run total solids weight percent and density in duplicate on the first MF Product sample MFP-1-F.
5. ☑ Mix the MF Product Samples 1-4 well and run rheology or yield stress at 25°C on all of them and be sure to save the results on the Rheology computer.
6. ☑ Report the Rheology results to Gene Daniel and Russ Eibling for analysis and yield stress model development.
7. ☑ Using the yield stress model, predict the amount of water to add to achieve a yield stress of 25 dynes/cm^2 at 25°C for the melter feed product of step 1.

✓ Predicted Amount of water to add ___0___ g

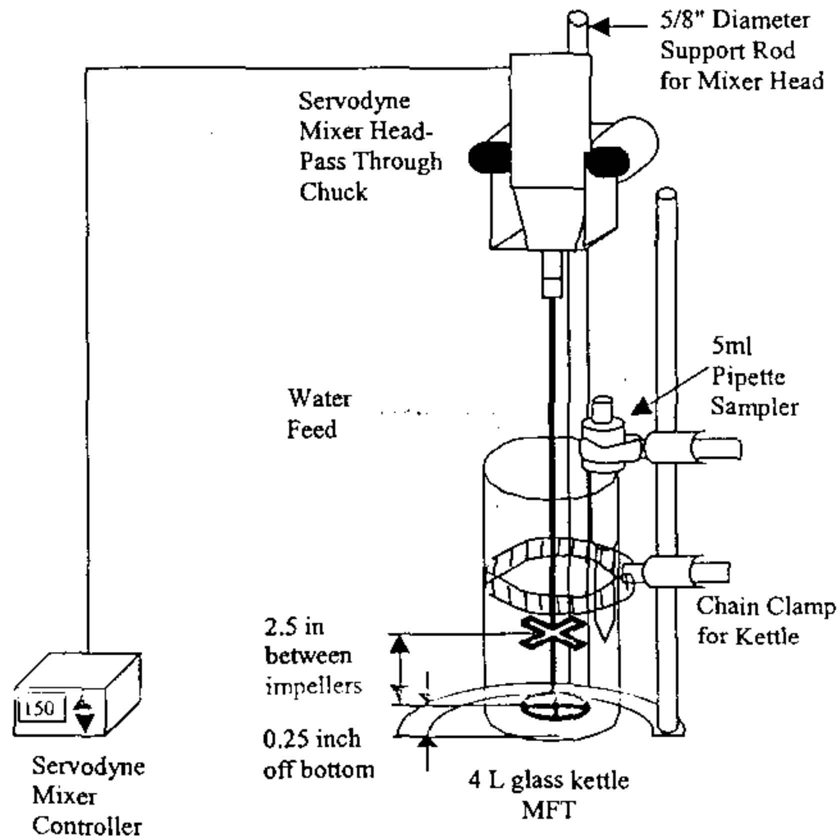
8. ☑ Add the predicted amount of water from the previous step and record:

Initial MF Sample Weight [g]	Amount Of Water Added [g]	Final Weight of MF Sample [g]	Resulting Yield Stress [dynes/cm^2]
2646.8	0	2646.8	24.8

9. ☑ If yield stress of the SME sample of the previous step is 25 dynes/cm^2 ± 2.5 dynes/cm^2 then proceed to the settling study. If the yield stress does not meet this spec then use the new data point to refine the yield stress model and repeat steps 6, 7, and 8 recording the new data until the desired yield stress is reached.

Prerequisites

1. ☑ Signed TAR requesting work.
2. ☑ Preliminary Rheology Testing for Melter Feed Yield Stress Adjustment.
3. ☑ Complete 4 SRAT/SME runs for Hydrogen CST studies
4. ☑ Setup experimental rig per sketch below. The rig will be operating at room temperature.



5. ☑ Check the speeds of the mixers and complete the following tables.

Mixer 1 Speed Reading	Mixer 1 Speed from Visual Inspection
60	62 (visual check)[8]
100	104 (visual check)[8]
125	skipped
150	164 (visual check)[8]

Mixer 2 Speed Reading	Mixer 2 Speed from Visual Inspection
60	62 (visual check)[8]
100	102 (visual check)[8]
125	skipped
150	157 (visual check)[8]

[8] Tried to use IRD Mechanalysis Model 890 Tachometer with optical strobe but gave imprecise and inconsistent readings so did a visual check that confirmed the mixer controllers were operating correctly.

Settling Studies for Melter Feed without CST

1. ☑ Put 2000 ml of the adjusted **Melter Feed Product Without CST** into a clean tared 4 L MFT kettle. Please record the following weights.

Clean 4 L Kettle Tare Weight	2029.4 g
4 L Kettle with Melter Feed Weight	4623.0 g
Initial Melter Feed Weight	2593.6 g

1:40 am 2. ☑ Set the Servodyne Mixer to 200 rpm for 15 minutes to well mix the melter feed.

1:55 am 3. ☑ Adjust Servodyne Mixer to 60 rpm and agitate the adjusted melter feed product. Please collect two 10-ml samples into two tared bottles at the following intervals while recording the following information.

9/1/98			Solids		Elementals			
Date/ Time	Sample Time	Mixer Speed Reading [RPM]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Sample Total Weight % Solids	Sample Id
12:55 pm	1 hour	60	14.3	21.1	14.4	27.9	43.8	MFP-1B
1:55 pm	2 hours	60	14.5	21.2	14.4	28.2	46.1	MFP-2B
3:55 pm	4 hours	60	14.4	28.1	14.4	29.9	47.34	MFP-4B
7:55 pm	8 hours	skip	skip	skip	skip	skip	skip	skip

Please analyze the first set of 10 ml samples for weight percent solids as soon as possible after taking them and hold the other 10 ml samples for later elemental analyses. **If the weight percent solids are not decreasing over time then add back any 10 ml samples pulled, lower the mixer speed, and go back to step 2.** If total solids decreased over time, agitation rate is slow enough or too slow. **If total solids decrease too quickly, add back any 10 ml samples pulled, raise the mixer speed, and go back to step 2.**

4. ☑ Leave the adjusted melter feed product mixing overnight.

5. ☑ The next day continue to mix the adjusted melter feed product and to take the 10 ml samples at the following intervals while recording the following information.

9/2/98			Solids		Elementals			
Date/ Time	Sample Time	Mixer Speed Reading [RPM]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Sample Total Weight % Solids	Sample Id
7:10 am	1 hour	60	14.3	25.8	14.5	26.3	17.88	MFP-19B
8:10 am	2 hours	60	14.3	26.1	14.5	26.1	17.98	MFP-20B
10:10 am	4 hours	60	14.5	26.0	14.4	26.1	17.18	MFP-22B
2:10 pm	8 hours	60	14.6	26.1	14.4	25.9	17.00	MFP-26B

Please analyze the first set of 10 ml samples for weight percent solids as soon as possible after taking them and hold the other 10 ml samples for later elemental analyses.

6. ☑ At the end of a successful run please send all the elemental 10-ml samples to the Mobile Lab for analyses of Fe, Al, or Mn for sludge, B or Li for frit, and Ti for CST.

7. ☑ After a successful run please attempt to pull a core sample from the kettle. The kettle will be divided into quadrants as shown in the sketch below and core samples will be extracted at the numbered points.

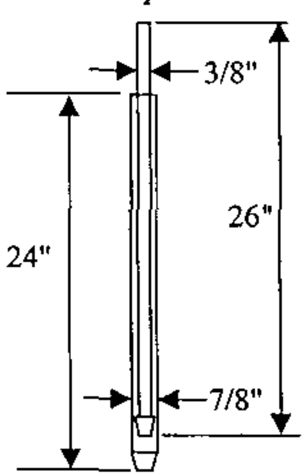
A. Collect and weigh 19 10-ml sample vials for the different core samples. The samples will be extracted with a Coliwasa that is normally used to extract core samples from 55-gallon drums and a stainless steel needle and syringe. These two devices are illustrated below.

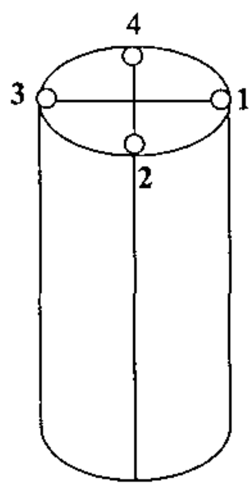
B. Hold the Coliwasa with its inner glass rod about 5 inches off its bottom and slowly insert it into the fluid at the first sample point as shown in the drawing below until it reaches the kettle bottom. Gently push down on the Coliwasa inner glass rod to capture the core and then pull it gently out of the kettle. Put the Coliwasa over the first sample vial and slowly pull up on the glass inner rod and let the slurry drain until the sample level drops about an inch. Push down on the Coliwasa inner glass rod and move to the next sample vial and pull back up on the inner glass rod and release about another inch of sample into the next vial. Continue to do so until the Coliwasa has been emptied. This should take no more than 5 sample vials. Now move to the next sampling point and repeat this step until all 4 core Coliwasa samples have been drawn. These samples will be labeled MFP-Sample Point-Sample Height, for example at sample point 1, the sample from the first inch in the Coliwasa will be labeled MFP-1-1 and the second inch sample will be labeled MFP-1-2.

C. Now use the stainless steel needle and syringe to pull samples off the bottom of the kettle at each of the 4 sample points shown below. Gently insert the needle with the syringe plunger down into the slurry at the first sampling point and pull a 10-cc sample directly off the bottom. Gently pull out the needle and syringe and empty its contents into a sample vial. Repeat this step for each of the 4 sample points. These samples will be labeled MFP-Sample Point-N, for example, MFP-1-N for the needle sample pulled from the first sample point.

D. Once all samples have been pulled submit them to the Mobile lab for the necessary analyses.

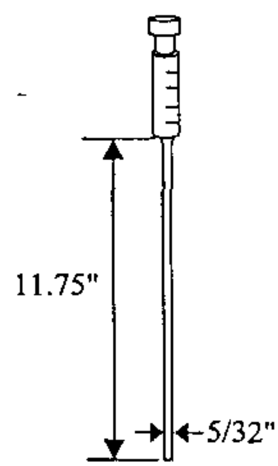
Coliwasa Sampler





4 L MFT Kettle

Stainless Steel Needle and Syringe Sampler



Settling Studies for Melter Feed with Coarse CST (as-received)

1. ✓ Put 2000 ml of the adjusted **Melter Feed Product with Coarse (as-received) CST** into a clean tared 4 L MFT kettle. Please record the following weights.

Clean 4 L Kettle Tare Weight	2029.3 g
4 L Kettle with Melter Feed Weight	4720.1 g
Initial Melter Feed Weight	2690.8 g

:30 am 2. ✓ Set the Servodyne Mixer to 200 rpm for 15 minutes to well mix the melter feed.

:45 am 3. ✓ Adjust Servodyne Mixer to 60 rpm and agitate the adjusted melter feed product. Please collect two 10-ml samples into two tared bottles at the following intervals while recording the following information.

9/3/98			Solids		Elementals			
Date/ Time	Sample Time	Mixer Speed Reading [RPM]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Sample Total Weight % Solids	Sample Id
8:45 am	1 hour	60	14.6	27.8	14.5	28.1	43.18	MFP-C-1
9:45 am	2 hours	60	14.4	27.9	14.4	27.7	43.94	MFP-C-2
11:45 am	4 hours	60	14.7	27.8	14.5	28.1	40.28	MFP-C-4
3:45 pm	8 hours	60	14.4	27.6	14.6	27.4	36.55	MFP-C-8

Please analyze the first set of 10 ml samples for weight percent solids as soon as possible after taking them and hold the other 10 ml samples for later elemental analyses. **If the weight percent solids are not decreasing over time then add back any 10 ml samples pulled, lower the mixer speed, and go back to step 2.** If total solids decreased over time, agitation rate is slow enough or too slow. **If total solids decrease too quickly, add back any 10 ml samples pulled, raise the mixer speed, and go back to step 2.**

4. ✓ Leave the adjusted melter feed product mixing overnight.
5. ✓ The next day continue to mix the adjusted melter feed product and to take the 10-ml samples at the following intervals while recording the following information.

9/4/98			Solids		Elementals			
Date/ Time	Sample Time	Mixer Speed Reading [RPM]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Sample Total Weight % Solids	Sample Id
8:45 am	1 hour	60	14.4	25.9	14.5	26.0	15.68	MFP-C-25
9:45 am	2 hours	60	14.3	25.9	14.4	25.9	15.95	MFP-C-26
11:45 am	4 hours	60	14.4	25.9	14.5	26.1	15.02	MFP-C-28
3:45 pm	8 hours	60	14.4	25.7	14.6	25.9	14.69	MFP-C-32

Please analyze the first set of 10 ml samples for weight percent solids as soon as possible after taking them and hold the other 10 ml samples for later elemental analyses.

6. ✓ At the end of a successful run please send all the elemental 10-ml samples to the Mobile Lab for analyses of Fe, Al, or Mn for sludge, B or Li for frit, and Ti for CST.

7. ✓ After a successful run please attempt to pull a core sample from the kettle. The kettle will be divided into quadrants as shown in the sketch below and core samples will be extracted at the numbered points.

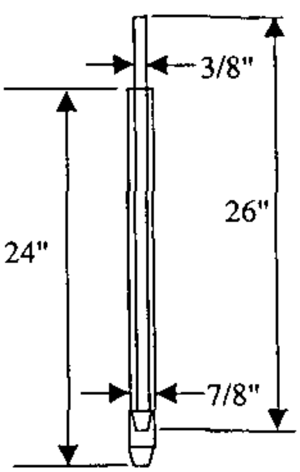
A. Collect and weigh 19 10-ml sample vials for the different core samples. The samples will be extracted with a Coliwasa that is normally used to extract core samples from 55-gallon drums and a stainless steel needle and syringe. These two devices are illustrated below.

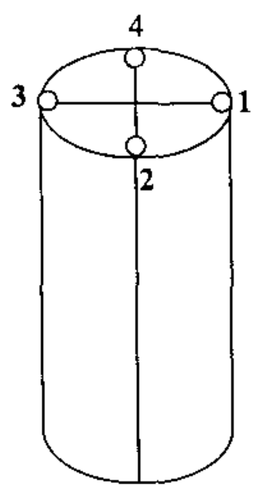
B. Hold the Coliwasa with its inner glass rod about 5 inches off its bottom and slowly insert it into the fluid at the first sample point as shown in the drawing below until it reaches the kettle bottom. Gently push down on the Coliwasa inner glass rod to capture the core and then pull it gently out of the kettle. Put the Coliwasa over the first sample vial and slowly pull up on the glass inner rod and let the slurry drain until the sample level drops about an inch. Push down on the Coliwasa inner glass rod and move to the next sample vial and pull back up on the inner glass rod and release about another inch of sample into the next vial. Continue to do so until the Coliwasa has been emptied. This should take no more than 5 sample vials. Now move to the next sampling point and repeat this step until all 4 core Coliwasa samples have been drawn. These samples will be labeled MFP-C-Sample Point-Sample Height, for example at sample point 1, the sample from the first inch in the Coliwasa will be labeled MFP-C-1-1 and the second inch sample will be labeled MFP-C-1-2.

C. Now use the stainless steel needle and syringe to pull samples off the bottom of the kettle at each of the 4 sample points shown below. Gently insert the needle with the syringe plunger down into the slurry at the first sampling point and pull a 10-cc sample directly off the bottom. Gently pull out the needle and syringe and empty its contents into a sample vial. Repeat this step for each of the 4 sample points. These samples will be labeled MFP-C-Sample Point-N, for example, MFP-C-1-N for the needle sample pulled from the first sample point.

D. Once all samples have been pulled submit them to the Mobile lab for the necessary analyses.

Coliwasa Sampler

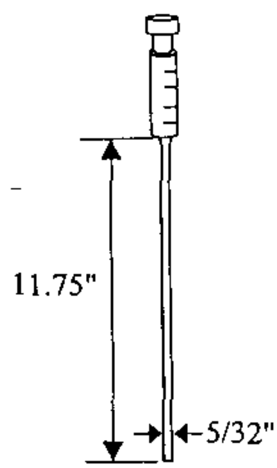




(front)

4 L MFT Kettle

Stainless Steel Needle and Syringe Sampler



Melter Feed with Intermediate Ground CST Settling Studies

1. ☑ Put 2000 ml of the adjusted **Melter Feed Product with Intermediate Ground CST** into a clean tared 4 L MFT kettle. Please record the following weights.

Clean 4 L Kettle Tare Weight	1950.2 g
4 L Kettle with Melter Feed Weight	4723.6 g
Initial Melter Feed Weight	2773.4 g

:00 am 2. ☑ Set the Servodyne Mixer to 200 rpm for 15 minutes to well mix the melter feed.

:15 am 3. ☑ Adjust Servodyne Mixer to 60 rpm and agitate the adjusted melter feed product. Please collect two 10-ml samples into two tared bottles at the following intervals while recording the following information.

9/4/98			Solids		Elementals			
Date/ Time	Sample Time	Mixer Speed Reading [RPM]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Sample Total Weight % Solids	Sample Id
9:15 am	1 hour	60	14.4	28.4	14.5	28.7	43.57	MFP-I-1
10:15 am	2 hours	60	14.5	28.1	14.5	28.5	46.04	MFP-I-2
12:15 am	4 hours	60	14.5	28.4	14.4	28.6	48.50	MFP-I-4
4:15 pm	8 hours	60	14.5	27.6	14.4	26.6	34.88	MFP-I-8

Please analyze the first set of 10 ml samples for weight percent solids as soon as possible after taking them and hold the other 10 ml samples for later elemental analyses. **If the weight percent solids are not decreasing over time then add back any 10 ml samples pulled, lower the mixer speed, and go back to step 2.** If total solids decreased over time, agitation rate is slow enough or too slow. **If total solids decrease too quickly, add back any 10 ml samples pulled, raise the mixer speed, and go back to step 2.**

4. ☑ Leave the adjusted melter feed product mixing overnight.
5. ☑ The next day continue to mix the adjusted melter feed product and to take the 10-ml samples at the following intervals while recording the following information.

9/5/98			Solids		Elementals			
Date/ Time	Sample Time	Mixer Speed Reading [RPM]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Sample Total Weight % Solids	Sample Id
9:15 am	1 hour	60	14.4	25.8	14.4	26.1	16.00	MFP-I-25
10:15 am	2 hours	60	14.4	25.9	14.4	25.8	15.22	MFP-I-26
12:15 am	4 hours	60	14.4	25.9	14.5	26.0	16.44	MFP-I-28
4:15 pm	8 hours	60	14.4	25.9	14.4	25.9	14.92	MFP-I-32

Please analyze the first set of 10 ml samples for weight percent solids as soon as possible after taking them and hold the other 10 ml samples for later elemental analyses.

6. ☑ At the end of a successful run please send all the collected 10 ml samples to the Mobile Lab for analyses of Fe, Al, or Mn for sludge, B or Li for frit, and Ti for CST.

7. ☑ After a successful run please attempt to pull a core sample from the kettle. The kettle will be divided into quadrants as shown in the sketch below and core samples will be extracted at the numbered points.

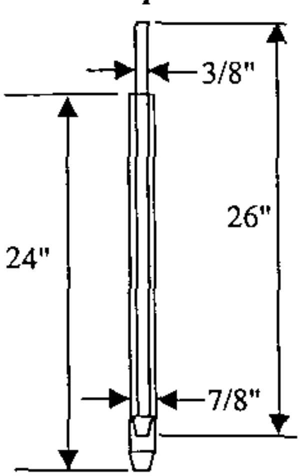
   A. Collect and weigh 19 10-ml sample vials for the different core samples. The samples will be extracted with a Coliwasa that is normally used to extract core samples from 55-gallon drums and a stainless steel needle and syringe. These two devices are illustrated below.

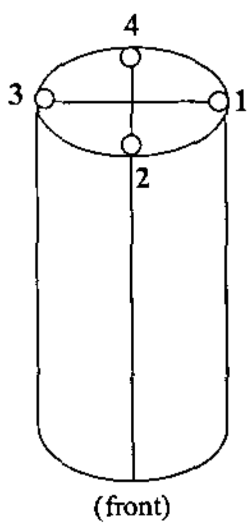
   B. Hold the Coliwasa with its inner glass rod about 5 inches off its bottom and slowly insert it into the fluid at the first sample point as shown in the drawing below until it reaches the kettle bottom. Gently push down on the Coliwasa inner glass rod to capture the core and then pull it gently out of the kettle. Put the Coliwasa over the first sample vial and slowly pull up on the glass inner rod and let the slurry drain until the sample level drops about an inch. Push down on the Coliwasa inner glass rod and move to the next sample vial and pull back up on the inner glass rod and release about another inch of sample into the next vial. Continue to do so until the Coliwasa has been emptied. This should take no more than 5 sample vials. Now move to the next sampling point and repeat this step until all 4 core Coliwasa samples have been drawn. These samples will be labeled MFP-C-Sample Point-Sample Height, for example at sample point 1, the sample from the first inch in the Coliwasa will be labeled MFP-C-1-1 and the second inch sample will be labeled MFP-C-1-2.

   C. Now use the stainless steel needle and syringe to pull samples off the bottom of the kettle at each of the 4 sample points shown below. Gently insert the needle with the syringe plunger down into the slurry at the first sampling point and pull a 10-cc sample directly off the bottom. Gently pull out the needle and syringe and empty its contents into a sample vial. Repeat this step for each of the 4 sample points. These samples will be labeled MFP-C-Sample Point-N, for example, MFP-C-1-N for the needle sample pulled from the first sample point.

   D. Once all samples have been pulled submit them to the Mobile lab for the necessary analyses.

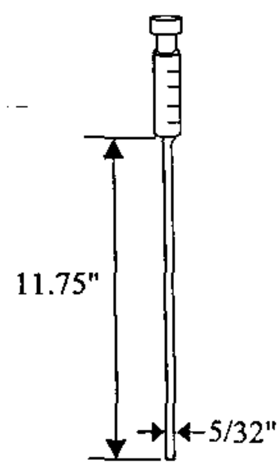
Coliwasa Sampler





4 L MFT Kettle

Stainless Steel Needle and Syringe Sampler



Melter Feed with Finely Ground CST Settling Studies

1. ☑ Put 2000 ml of the adjusted **Melter Feed Product with Finely Ground CST** into a clean tared 4 L MFT kettle. Please record the following weights.

Clean 4 L Kettle Tare Weight	1950.4 g
4 L Kettle with Melter Feed Weight	4597.2 g
Initial Melter Feed Weight	2646.8 g

:30 am 2. ☑ Set the Servodyne Mixer to 200 rpm for 15 minutes to well mix the melter feed.

:45 am 3. ☑ Adjust Servodyne Mixer to 60 rpm and agitate the adjusted melter feed product. Please collect two 10-ml samples into two tared bottles at the following intervals while recording the following information.

9/2/98			Solids		Elementals			
Date/ Time	Sample Time	Mixer Speed Reading [RPM]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Sample Total Weight % Solids	Sample Id
8:45 am	1 hour	60	14.5	28.0	14.5	28.0	41.87	MFP-F-1
9:45 am	2 hours	60	14.5	27.7	14.4	28.0	42.57	MFP-F-2
11:45 am	4 hours	60	14.5	28.4	14.5	28.0	42.38	MFP-F-4
3:45 pm	8 hours	60	14.4	27.7	14.4	27.6	43.42	MFP-F-8

Please analyze the first set of 10 ml samples for weight percent solids as soon as possible after taking them and hold the other 10 ml samples for later elemental analyses. **If the weight percent solids are not decreasing over time then add back any 10 ml samples pulled, lower the mixer speed, and go back to step 2.** If total solids decreased over time, agitation rate is slow enough or too slow. **If total solids decrease too quickly, add back any 10 ml samples pulled, raise the mixer speed, and go back to step 2.**

4. ☑ Leave the adjusted melter feed product mixing overnight.
5. ☑ The next day continue to mix the adjusted melter feed product and to take the 10-ml samples at the following intervals while recording the following information.

9/5/98			Solids		Elementals			
Date/ Time	Sample Time	Mixer Speed Reading [RPM]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Initial 10 ml Sample Bottle Weight [g]	Final 10 ml Sample Bottle Weight [g]	Sample Total Weight % Solids	Sample Id
9:15 am	1 hour	60	14.4	25.8	14.6	26.0	15.34	MFP-F-25
10:15 am	2 hours	60	14.4	25.7	14.5	26.0	15.16	MFP-F-26
12:15 am	4 hours	60	14.4	25.6	14.4	25.6	15.04	MFP-F-28
4:15 pm	8 hours	60	14.5	25.7	14.4	25.7	14.93	MFP-F-32

Please analyze the first set of 10 ml samples for weight percent solids as soon as possible after taking them and hold the other 10 ml samples for later elemental analyses.

6. ☑ At the end of a successful run please send all the collected 10 ml samples to the Mobile Lab for analyses of Fe, Al, or Mn for sludge, B or Li for frit, and Ti for CST.

7. ☑ After a successful run please attempt to pull a core sample from the kettle. The kettle will be divided into quadrants as shown in the sketch below and core samples will be extracted at the numbered points.

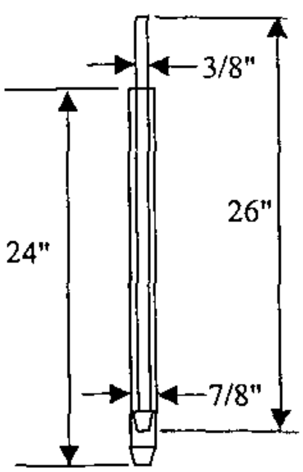
   A. Collect and weigh 19 10-ml sample vials for the different core samples. The samples will be extracted with a Coliwasa that is normally used to extract core samples from 55-gallon drums and a stainless steel needle and syringe. These two devices are illustrated below.

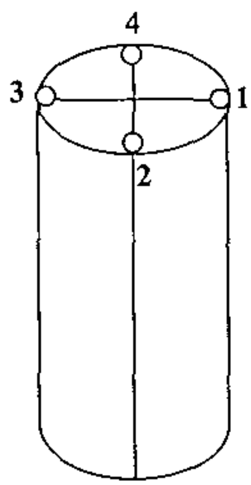
   B. Hold the Coliwasa with its inner glass rod about 5 inches off its bottom and slowly insert it into the fluid at the first sample point as shown in the drawing below until it reaches the kettle bottom. Gently push down on the Coliwasa inner glass rod to capture the core and then pull it gently out of the kettle. Put the Coliwasa over the first sample vial and slowly pull up on the glass inner rod and let the slurry drain until the sample level drops about an inch. Push down on the Coliwasa inner glass rod and move to the next sample vial and pull back up on the inner glass rod and release about another inch of sample into the next vial. Continue to do so until the Coliwasa has been emptied. This should take no more than 5 sample vials. Now move to the next sampling point and repeat this step until all 4 core Coliwasa samples have been drawn. These samples will be labeled MFP-C-Sample Point-Sample Height, for example at sample point 1, the sample from the first inch in the Coliwasa will be labeled MFP-C-1-1 and the second inch sample will be labeled MFP-C-1-2.

   C. Now use the stainless steel needle and syringe to pull samples off the bottom of the kettle at each of the 4 sample points shown below. Gently insert the needle with the syringe plunger down into the slurry at the first sampling point and pull a 10-cc sample directly off the bottom. Gently pull out the needle and syringe and empty its contents into a sample vial. Repeat this step for each of the 4 sample points. These samples will be labeled MFP-C-Sample Point-N, for example, MFP-C-1-N for the needle sample pulled from the first sample point.

   D. Once all samples have been pulled submit them to the Mobile lab for the necessary analyses.

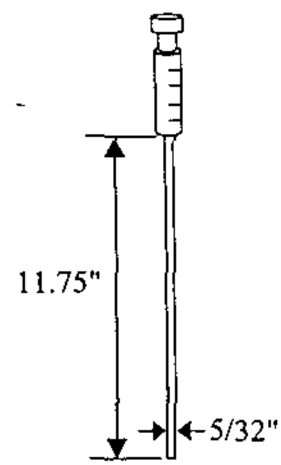
Coliwasa Sampler





4 L MFT Kettle

Stainless Steel Needle and Syringe Sampler



July 27, 1998

SRT-PTD-98-032

To: W. L. Tamosaitis, 773-A

From: L. F. Landon, 704-1T
C. T. Randall, 704-T

SCOPE OF TESTS REQUESTED BY WPT IN SUPPORT OF SELECTION PROCESS FOR IN-TANK PRECIPITATION ALTERNATIVE PROCESSES

Reference: HLW Technical Request HLE-TAR-98060, CST Ion Exchange, Salt Team Phase 3 Evaluation, 7/14/98.

ISSUE 13: Quantify the relative settling behavior of the CST resin in sludge as compared to the current DWPF reference Frit 200.

Objective and Scope:

One of the four process options to the current In-Tank Precipitation process is the use of non-elutable CST resin to remove the Cs-137 from the supernate fraction of SRS High Level Waste. The loaded resin would be blended with the insoluble solids fraction (sludge) of this waste that is currently being processed in DWPF. It is important to determine if the settling behavior of the CTS resin differs from that of the current DWPF glass former (Frit 200) in DWPF sludge slurries produced throughout the melter feed preparation processes. If the CST resin were to settle in these sludge slurries faster than the Frit 200, slurry homogeneity required for Waste Acceptance, both sampling and melter feeding, may not be met. These tests are "scoping activities" and accordingly do not require the preparation and approval of Technical Task and QA Plans. The quantity of CST resin added to a sludge batch will be based on a CST loading in the glass product of 10-wt%.

Researchers:

Dan Lambert and Gene Daniel

Experimental Method:

Experiments will be performed using simulated DWPF melter feed containing CST in three particle size distributions. The experiments will determine if, under conditions that promote particle segregation, the CST particles settle more readily or less readily than the reference Frit 200. If the frit separates more readily than the CST, then the adequacy of existing DWPF agitation for the CTS process will be confirmed. If any of the three CST particle size distributions tested separate more readily than frit, additional tests beyond the scope of this task will be required to better define CST particle size limits, DWPF design modifications, and/or DWPF operating parameters.

The three size distributions to be tested are qualitatively defined as,

- As received (engineered form)
- Extensively crushed resin (to be defined)
- Moderately crushed resin

The size distribution of the CST feed material will be measured for each test.

The CST-bearing melter feed will be produced by carrying out DWPF flowsheet operations at bench scale as outlined in Issue 21 below. The relative settling characteristics of DWPF frit and CST will be evaluated by measuring the ratio of CST to frit in either the solids that settle out under conditions which promote particle segregation or in the overlying depleted slurry.

The rheological properties of the melter feed will be measured with a Haake viscometer and the rheology will be adjusted by adding or removing water to simulate the lowest DWPF design basis yield stress for sludge process slurries (25 dynes/cm^2). The lower yield stress will promote segregation.

Each batch of melter feed (SME product) will be agitated in a beaker with an agitator that is similar to the DWPF SME agitator so it will maximize bulk fluid motion and minimize local shear gradients. To promote segregation, only enough agitation (shear) will be developed to overcome hindered settling. Samples of the settled insolubles and/or the depleted slurry will be obtained as a function of time. The ratio of frit solids to CST solids will be determined by analyzing the samples for titanium and a cation specific to Frit 200.

Preparation and testing will occur in the following order:

- Extensively crushed resin
- Moderately crushed resin
- Resin as received

Slurries produced for this study shall be retained for possible further experiments until released by the Salt Disposition Flowsheet team.

Deliverables:

- The Frit 200 to CST ratio in settled solids or depleted slurry as function of settling time for each particle size distribution tested.

- Technical report

Pre-Requisites:

- A run plan will be prepared by ITS for each experiment.

- HLWE to confirm with DWPF-E that these tasks have priority over the current bench-scale R & D currently in progress or planned in support of DWPF in FY98.

- HLWE to specify whether or not the CST is to be pre-treated with caustic as would be the procedure in preparing the CST for actual use. If the CST is to be pre-treated, HLWE to specify the procedure.

- HLWE to specify whether the CST resin is added to the sludge slurry dry or as a water slurry. If to be added a water slurry, HLWE to specify the wt% CST in the slurry.

- HLWE to provide ITS any information on the density of the engineered form of the CST.

- HLWE to provide ITS any data on the particle size distribution of the engineered form of the CST.

Data Applicability:

The tests focus on determining the relative settling characteristics of CST resin as compared to the DWPF glass former (Frit 200) in the melter feed. If there is not a discernable difference in settling characteristics for any of the CST size distributions tested, it would suggest the introduction of CST resin to the SRAT (equivalent to a 10 wt% concentration in glass) will not adversely impact homogeneity and slurry sampling due to segregation.

However, if it is determined that the CST resin, in any of the tested size distributions, were to settle faster than DWPF Frit 200 it would suggest that additional testing is needed. This testing would not only entail process vessel mixing studies but would also include an assessment of the DWPF sampler to confirm it will obtain a representative sample. These tests could be performed in the full-scale SRAT located at TNX, which contains prototypic DWPF SRAT and SME mixing and sampling systems.

Schedule:

Technical report issued by 9/21/98.

ISSUE 21: Assess the impact of blending CST resin with sludge in the DWPF Sludge Receipt and Adjustment Tank (SRAT) on the maximum hydrogen generation rate produced during the DWPF melter feed preparation processes.

Objective and Scope:

One of the four process options to the current In-Tank Precipitation process is the use of non-elutable CST resin to remove the Cs-137 from the supernate fraction of SRS High Level Waste. The resin would be blended with the insoluble solids fraction (sludge) of this waste that is currently being processed in DWPF. It is important to determine if the presence of the CST resin in the DWPF melter feed preparation processes will affect the rate of hydrogen generation from formic acid decomposition. These tests are "scoping activities" and accordingly do not require the preparation and approval of Technical Task and QA Plans. No monosodium titanate solids will be included in the batch. The quantity of CST resin added to a sludge batch will be based on a CST loading in the glass product of 10-wt%.

Researchers:

Dan Lambert and Paul Monson

Experimental Method:

Hydrogen generation rates in bench scale versions of the DWPF Slurry Receipt and Adjustment Tank (SRAT) and Slurry Mix Evaporator (SME) will be measured in real time during a SRAT cycle simulation and during a SME cycle simulation. Three experiments will be conducted using the proposed more reducing sludge-only flowsheet and each of the three CST particle sizes provided for Issue 13. The SME product produced in this task will be used as the starting material for each of the three experiments in Issue 13.

A Tank 42 sludge slurry surrogate, containing HM levels of noble metals and mercury, will be used in the tests. The CST will be blended with the SRAT sludge batch prior to beginning the SRAT cycle. Each test will use approximately 2-Liters of the Tank 42 sludge surrogate. SRAT cycle and SME cycle process simulations will be performed in each test. Samples of the SRAT and SME products will be analyzed for titanium and these results used to validate that the CST content of the product from vitrification of the SME product would be as targeted (10 wt%). The rheology of the SME product will be measured to determine if the solids concentration needs to be adjusted to obtain the yield stress required for Issue 13 (25 dynes/cm^2).

Deliverables:

- Quantitative data on hydrogen generation rates (including the peak rate) from the SRAT and SME for each of the three CST size distributions.
- Results will be compared with existing hydrogen data from previous DWPF process simulations performed under identical processing conditions.
- The calculated CST content of a glass product if the SME product had been verified.
- Technical report

Pre-Requisites:

- A run plan will be prepared by ITS for each experiment.
- HLWE will confirm with DWPF-E that these tasks have priority over the current bench-scale R & D currently in progress or planned in support of DWPF in FY98

Data Applicability:

As currently defined, these tests will only assess the impact that unused CST resin has on the peak hydrogen generation rates previously determined for DWPF for the proposed more reducing sludge-only flowsheet using Tank 42 sludge surrogate at HM levels of noble metals. The impact of loaded CST resin, containing the level of noble metals projected to be absorbed on the CST resin during the life expectancy of a CST column, would also need assessed for this option.

Schedule:

Technical report issued by 9/21/98

Approvals:

J. T. Carter 7/28/98

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M. F. Williams, 704-1T
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