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**Hanford Waste Vitrification Program Process
Development: Melt Testing Subtask, Pilot-Scale
Ceramic Melter Experiment, Run Summary**

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**Prepared for
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830**

**Pacific Northwest National Laboratory
Richland, Washington 99352**

MASTER

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SUMMARY

The PSCM-22 experiment was designed to evaluate the behavior of the HW-39 glass composition under long-term, pilot-scale conditions. The test consisted of 427 hours of operation and had an online efficiency above 95%.

Major results and conclusions of PSCM-22 are listed below.

- HWVP melter feed exhibited excellent processing characteristics for the majority of the test.
- The forming process used in feed preparation added sufficient Total Organic Carbon (TOC) to the melter feed to maintain a reducing feed without the addition of sugar.
- Redox potentials established during each segment of the run were sufficient to maintain stable processing. No melt foaming was observed during the experiment.
- Feed and glass samples tested for redox potential following the run were well within the acceptable ranges except for the initial batch of Auxiliary feed.
- Feed rates for the more reduced feed segment (sugar added as a reductant) of the test matched predicted rates from HWVP-11, which processed a similar feed (sugar was added as a reductant).
- A feed without added reductant processed at higher rates than the more reduced feed.
- Due to unexpected electrode current shorting through the crystal sample boat, local heating probably occurred at times during the run. The resulting wide swings in measured bulk glass temperature made temperature control difficult.
- The melter feed system using an ADSP II feed pump operated with minimal interruptions.
- Concentration of waste components in feed samples varied, suggesting some segregation had occurred in the waste slurry holding tank.

- The PSCM pour stream was determined to be stable and successfully filled half-scale canisters (6.4 cm, 2.5 in. I.D. neck opening).
- Melter gas phase losses to the off-gas system are only significant for B, C, N, S and the halogens, which all readily form volatile acidic gases.
- Melter DFs for Cs were similar to previous values obtained during the processing of formatted SRL feeds in the PSCM.
- Ruthenium volatility was minimal due to the reducing conditions of the glass throughout the test.
- The combined overall DF across the melter, EVS and SAS was essentially even though significant melter and EVS performance changes did occur. This demonstrates the ability of the scrubbing equipment to respond to higher aerosol loadings that periodically occur.
- HEME and SAS DFs were a factor of 3 and 10, respectively, below design expectations.
- Laboratory melt rate tests failed to yield quantitative results due to the thin cold cap generated by HWVP melter feed.
- The crystalline phase observed in the PSCM-22 melter run was the same basic composition, $(\text{Ni,Fe})(\text{Fe,Cr})_2\text{O}_4$, that has been observed in laboratory and previous melter testing. Mass balance, melter core sample, and settling collection boat evaluation indicate that a measurable amount of settling of the crystalline material did occur in the melter during the run. A layer of at least 1.5 cm thickness and approximately 20 volume % settled crystalline material was produced during the PSCM-22 melter run. It is not known if the settled layer is produced at a continuing steady rate or if the layer approaches an equilibrium thickness due to factors such as melter convection conditions.

Additional data collected during PSCM-22 that is not presented in this summary includes:

- results of temperature profiles obtained from the filling of a 61-cm-diameter instrumented canister
- evaluation of step and pulse tracer studies performed during PSCM-22.

This work was done in support of the NWTP. Data analysis will be completed during FY 86 and reported by the NWTP under separate cover.

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INTRODUCTION

Hanford Waste Vitrification Program (HWVP) activities for FY 1985 have included engineering and pilot-scale melter experiments HWVP-11/HBCM-85-1 and HWVP-12/PSCM-22. Major objectives designated by HWVP for these tests were to evaluate the processing characteristics of the current HWVP melter feed during actual melter operation and establish the product quality of HW-39 borosilicate glass. The current melter feed, defined during FY 85, consists of reference feed (HWVP-RF) and glass-forming chemicals added as frit.

HWVP-11, completed in April 1985, was performed to evaluate two options for the addition of glass-forming chemicals to melter feed. The results of this test show that a melter feed having glass-formers added solely as premade frit (100% frit feed) processed at a higher rate with superior cold cap characteristics in actual melter conditions than a feed that consisted of glass-forming chemicals added as one third frit and two thirds unreacted bulk chemicals (1/3 frit - 2/3 unreacted chemical feed).

PSCM-22, which consisted of 427 hours of melter operation, was completed on August 2, 1985. During the run, approximately 17,100 liters of feed were processed and 6,800 kilograms of glass were produced. The eighteen day melter experiment was designed to evaluate the processing characteristics of the current HWVP melter feed (100% frit feed) on a pilot-scale. An important objective of this test was to investigate the effects of redox level on stability of the glass melt and overall melter operation. Melter feed was processed with and without added reductant (sugar) during different segments of the test.

Secondary objectives in support of HWVP during PSCM-22 are identified below.

- evaluation of the settling potential for spinel crystals formed in the current glass (high Cr concentration)
- generation of additional data to be used in ongoing efforts to correlate results of laboratory scale melting behavior with the PSCM
- characterization of off-gas equipment performance

- collection of preliminary information on the compatibility of the proposed HWVP melter/turntable design with the HWVP reference canister
- obtain additional experience using melter support equipment such as the ADSP II feed pump and the Hewlett-Packard Data Acquisition System.

Data analyses and discussion of the methods used in satisfying HWVP primary and secondary objectives of PSCM-22 are presented in this summary report.

Also accomplished during PSCM-22 were several objectives in support of the Nuclear Waste Treatment Program (NWTP). They included:

- the filling of a 61 cm diameter instrumented canister to obtain additional glass temperature profiles to be used in verification of the TEMPEST computer code
- further evaluation of a constant glass temperature feedback control algorithm
- the addition of tracers to the melter feed to generate additional data for use in verification of a process mass balance model.

Finally, a total of ten half-scale test canisters were filled in support of the Transportation Technology Center (TTC) located at Sandia National Laboratory (SNL) under a Materials Characterization Center (MCC) task. Six of these canisters will be used in impact testing for shipping casks to be done during the first quarter of FY 86 at SNL. The remaining four canisters will be used in future testing at PNL. The modifications and techniques used to fill the half scale canisters will be discussed in this summary. The methods and results of the impact testing will be reported under separate cover.

FEED PREPARATION

PSCM-22 utilized the current HWVP reference feed designated during the first quarter of FY 85.^(a) The reference feed, HWVP-RF, is a simulation of a NCAW (neutralized current acid waste) and a CRW (cladding removal waste) blend. Table 1 shows the target composition of the reference feed. Included in the table are the concentration of bulk chemicals used in feed make-up and the assumed oxide concentrations in the glass. The basis for the HWVP simulation is 400 grams total oxides per liter of melter feed with the reference feed supplying 25 percent of the oxide total. Remaining oxides in the melter feed are the glass-forming chemicals in the form of a pre-melted glass frit (see Table 1). The target composition and oxide loading (400 g/L) are based on laboratory evaluations of melter feed rheological properties performed during FY 84-85.^(b) Most of the reference feed components are added as hydroxides, with very few nitrate compounds being present.

The preparation of HWVP melter feed involves the treatment of HWVP-RF with formic acid. Formic acid reacts with the mono- and di-valent waste cations to form formate salts. This step, which is part of the Defense Waste Processing Facility (DWPF) flowsheet, has been shown to improve the rheology of the slurry while also providing additional total organic carbon (TOC).^(c) Preparation of PSCM-12 melter feed began with the batching of technical grade bulk chemicals (2300 L batches), per Table 1, in Tank 311 of the Chemical Make-up Room. Because the feed needed to be homogenized before being fed to the melter, no SiO_2 was added to the reference feed in Tk 311. Previous experience with the Tekmar PSD-LT60/4 single stage rotor/stator homogenizer showed that SiO_2 caused excessive wear on the rotor/stator and should be avoided.

-
- (a) Nankani, F. D., T. D. Kirkpatrick. 1984. "HWVP Feed Composition Variability." Letter No. 65612-84-148, Rockwell International, Richland, Washington.
 - (b) Blair, H. T., B. A. Pulsipher. 1985. "Laboratory Evaluation of the Maximum Concentration of Melter Feed for the Hanford Waste Vittrification Plant." Internal Letter Report HWVP-85-020203B. Pacific Northwest Laboratory, Richland, Washington.
 - (c) Blair, H. T., C. O. Harvey, D. McCarthy. 1985. "Status of Melter Feed Rehology Evaluation for Melter Feed Selection." Internal Letter Report HWVP-85-020203A, Pacific Northwest Laboratory, Richland, Washington.

TABLE 1. Composition of Simulated Melter Feed Slurry⁽¹⁾

Compound	(2) Concentration g/L	Assumed Oxide	Concentration g/L	Glass wt%
Fe(OH) ₃	59.5	Fe ₂ O ₃	44.4	11.10
Al(OH) ₃	26.3	Al ₂ O ₃	17.2	4.30
NaNO ₃	13.4	Na ₂ O	10.7	2.68
Cr(OH) ₃	7.21	Cr ₂ O ₃	5.3	1.33
NaOH	3.58	ZrO ₂	2.4	0.60
Na ₂ C ₂ O ₄ (3)	3.37	SiO ₂	3.0	0.75
Zr(OH) ₄	3.11	NiO	2.4	0.60
SiO ₂	3.00	SO ₃	1.5	0.38
Ni(OH) ₂	3.00	La ₂ O ₃	2.2	0.55
Na ₂ SO ₄	2.32	MoO ₃	1.2	0.30
LaF ₃	2.16	Nd ₂ O ₃	2.1	0.53
Na ₂ MoO ₄ • 2H ₂ O	2.00	Cs ₂ O	1.0	0.25
Nd(OH) ₃	1.84	CeO ₂	0.7	0.18
CsOH	1.05	CuO	0.6	0.15
Ce(OH) ₃	0.79	MnO ₂	0.7	0.18
Cu(OH) ₂	0.74	BaO	0.4	0.10
Mn(OH) ₂	0.74	F ⁻	1.2	0.30
BaSO ₄	0.63	SrO	0.4	0.10
NdF ₃	0.58	Pr ₆ O ₁₁	0.4	0.10
Sr(OH) ₂	0.47	MgO	0.3	0.08
Pr(OH) ₃	0.47	CaO	0.3	0.08
La(OH) ₃	0.47	Y ₂ O ₃	0.2	0.05
NaF	0.47	Sm ₂ O ₃	0.2	0.05
Hg(OH) ₂	0.42	TOC	0.6	0.15
CaF ₂	0.42	I ⁻	0.04	0.01
Y(OH) ₃	0.26			
Sm(OH) ₃	0.21			
NaI	0.05			
SUBTOTALS	138.56 g/L		99.4 g/L	24.9%
HW39 Frit (4)	300.0	SiO ₂	201.8	50.44
		B ₂ O ₃	38.2	9.56
		Na ₂ O	30.8	7.69
		Li ₂ O	15.0	3.75
		CaO	11.2	2.81
		MgO	3.0	0.75
SUBTOTALS:	300.0 g/L		300.0 g/L	75.0 wt%
TOTALS:	438.5 g/L		400.0 g/L	100 wt%

Reductants: 12.2 g/L formic acid, 3.5 to 0 g/L sugar

(1) Basis: 400 g oxide/L slurry, 25 wt% waste oxide loading.

(2) Prior to Reaction with Formic Acid

(3) TOC substitute

(4) HW39 frit size distribution as follows: 1/3 to be -80 +200 mesh
2/3 to be -200 mesh

The waste was then heated to between 90 and 95°C and reacted with 10.2 g/L of 90 wt% formic acid. Based on previous experience, the acid was added at a rate of 20 L/h initially and was lowered to 10 L/h after approximately 60% of the total acid had been added. The reduced addition rate successfully avoided any excessive foam accumulation during PSCM-22 feed make-up. This phenomenon had been observed in laboratory experiments and during feed preparation for HWVP-11/HBCM-85-1.

Following acid addition, the waste was heated to around 100°C and held there for two hours. This step was incorporated into feed make-up to ensure that all reactions occurring in the slurry had gone to equilibrium and to correlate more closely with laboratory experiments and SRL procedures. Finally, the reference feed was diluted to original levels and allowed to cool before transfer to TK 69 in the High-Bay Extension. It should be noted that minor problems were encountered with the transfer of feed from TK 311 to TK 69. Accumulations of solids, combined with ferric hydroxide sludge tended to plug a strainer (0.25 cm openings) in the outlet line from TK 311. The strainer had to be cleaned 2 to 3 times during each feed transfer. Because of the limited solubility of some of the components of the reference feed the accumulation of a small amount of solids in the bottom of the make-up tank is not unusual.

To provide the necessary amount of melter feed for PSCM-22 five batches (2300 L each) of reference feed were prepared in the manner described above and combined in TK 69. Approximately 11,000 L of reference feed were homogenized for almost 12 hours in an effort to break up some of the solid agglomerations. Initially, the waste had a tendency to plug a strainer (0.25 cm openings) in the exit line of TK 69, upstream of the homogenizer. The plugs occurred approximately every 30 to 40 minutes and had a composition that was visually similar to those experienced during feed transfer. Homogenation eventually solved the problem and subsequent feed transfers were accomplished without plugging. Once the waste was adequately homogenized a sample was analyzed using induction coupled plasma (ICP) spectroscopy. Adjustments to the reference feed were made based on these results (see Table 2). Major deficiencies included Fe, La, Si, Zr, and Cs.

TABLE 2. Comparison of Reference Batches

<u>Compound</u>	<u>Target Wt%</u>	<u>Reference Wt%(a)</u>	<u>Auxiliary Wt%(a)</u>
Al ₂ O ₃	4.30	4.45	3.71
B ₂ O ₃	--	0.16	0.12
BaO	0.10	0.10	0.10
CaO	0.08	0.13	0.13
CeO ₂	0.18	0.15	0.16
Cr ₂ O ₃	1.33	1.35	1.36
CuO	0.15	0.17	0.15
Fe ₂ O ₃	11.10	10.82	10.13
La ₂ O ₃	0.55	0.46	0.31
MgO	0.08	0.13	0.09
MnO ₂	0.18	0.20	0.19
MoO ₃	0.30	0.27	0.27
Na ₂ O	2.68	2.78	2.63
Nd ₂ O ₃	0.53	0.52	0.31
NiO	0.60	0.56	0.51
SiO ₂	0.75	0.51	1.61
ZrO ₂	0.60	0.37	0.54
Cs ₂ O	0.25	0.13	0.09
I ⁻	0.01	0.01	--
F ⁻	0.30	0.19	0.18
Cl ⁻	--	0.10	3.42
NO ₃ ⁻	--	2.25	2.40
SO ₄ ²⁻	0.38	0.37	0.07

(a) Prior to Adjustment.

Higher than expected feed rates were encountered during the portion of the run when no sugar was added, and losses were incurred in transferring feed between process tanks. The original batch of reference feed was exhausted during the final week of the run. Sufficient quantities of bulk chemicals were on hand for 1500 L of additional melter feed. Reference feed for the Auxiliary feed batch was prepared in the same manner as the initial reference feed and used many of the same bulk chemicals. The major difference between the two reference feed batches was that the $\text{Fe}(\text{OH})_3$ used in the second batch was from a chemical order that had previously been rejected due to an excessive chloride contamination. An overall comparison of reference feed compositions showed that the Auxiliary feed was well matched with the initial reference feed except for the chloride concentration (see Table 2). Adjustments to the batch were made to increase Al, Fe, Nd, and Cs concentrations. Problems in processing the auxiliary feed resulting from the chloride contamination are discussed in the Feed Processability section.

The final step of feed preparation occurred in Tk 11 of the High-Bay Extension. Reference feed was mixed with 100 g coarse frit/liter of melter feed (coarse frit: -80 +200 mesh), 200 g fine frit/liter melter feed (fine frit: -200 mesh), 1 weight percent formic acid (1% x total weight of frit), and water to reach the desired volume. The additional acid was used to stabilize the pH of the melter feed, which had a tendency to drift upward due to alkali leaching of the frit. Coarse frit, which makes up 1/3 of the total frit, will come from the canister frit blasting step in the current HWVP flowsheet. The remaining frit was added in the -200 mesh size range to enhance the rheology of the melter feed. The finer particles increased the feed viscosity and improved particle suspendibility in laboratory testing.^(a)

(a) Blair, H. T., C. O. Harvey, D. McCarthy. 1985. "Status of Melter Feed Rheology Evaluation for Melter Feed Selection." Internal Letter Report HWVP-85-020203A, Pacific Northwest Laboratory, Richland, Washington.

MELTER OPERATIONS

CHRONOLOGY

PSCM-22 was an eighteen day pilot-scale melter experiment that was conducted between July 15 and August 2, 1985. Total run time was 427 hours with approximately 5 hours of unplanned equipment downtime. The majority of downtime was due to problems with plugging in the feed system and no major equipment failures were encountered during the run. Seven and a half hours of scheduled downtime were incurred on Day 8 of the run (July 23) to change over from the initial feed, which had a small amount of reductant added in the form of commercial sugar, to a feed with no additional reductant. During this time, the feed was shut off and the cold cap was sampled as it melted off. Also, an Inconel lid was placed on the spinel crystal sampler (a collection boat that had been placed on the melter floor, prior to PSCM-22 start-up, to collect material that had settled out of the melt.

Numerous activities in support of HWVP and NWTP were carried out during PSCM-22. Figure 1 is a chronology chart which outlines the time sequence of a majority of the stated objectives fulfilled during this experiment. Table 3 summarizes the major segments of the test. The 61 cm (24 in) diameter instrumented canister was filled during the period from July 19 to July 22 or hours 81 to 163. Similarly, the 10 half-scale canisters for SNL were filled from July 26 to August 1 (hours 247 to 398) with occasional breaks due to canister shipping delays.

EQUIPMENT PERFORMANCE

Feed System

The primary feed system for PSCM-22 utilized a second generation air-displacement slurry pump (ADSP II), a magnetic flow meter mounted above the feed tank flange, feedline, and a water-cooled feed nozzle. Figure 2 is a graphical representation of the ADSP II. The control schematic used for operating the pump is shown in Figure 3. The feed line from the pump chamber through the magnetic flow meter was 0.97 cm O.D. x 0.09 cm wall thickness (0.38 in. x 0.04 in.) stainless steel tubing. The feed line from the flow

FIGURE 1. PSCM-22 Chronology

TABLE 3. Summary of PSCM-22

Test Duration:	427.0 hours
Scheduled Downtime:	7.5 hours
Unscheduled Downtime:	5.0 hours
Process Efficiency: ^(a)	98.8%
ADSP II Efficiency: ^(b)	85.9%

	<u>Added Sugar</u>	<u>No Added Sugar</u>	<u>Auxiliary</u>
Duration	186	198	30.5
Total Feed (L)	6737	9213	1123
Nominal Feed Rate (L/h) ^(c)	35.9	39.1	39.3 ^(d)
Maximum Feed Rate (L/h) ^(c)	39.1	50.9	
Feed Redox	0.104	0.002	0.42 ^(e)
Glass Redox	0.160	0.015	0.147 ^(f)

(a) $\text{Process Efficiency} = 100 - \frac{[\text{Unscheduled Downtime}] \times 100}{(\text{Duration} - \text{Scheduled Downtime})}$

(b) $\text{ADSP II Efficiency} = 100 - \frac{[\text{ADSP Downtime}] \times 100}{(\text{Duration} - \text{Total Downtime})}$

ADSP Downtime = Total time ADSP was not in service.

Total Downtime = Scheduled + Unscheduled Downtime.

(c) Feed rate values are averaged rates (see Figure 18).

(d) Due to the short duration of the Auxiliary segment, no distinction was made between nominal and maximum feed rates.

(e) The initial batch of Auxiliary feed had a ferrous-to-ferric of 1.2. Addition of NaNO_3 lowered the value to 0.028 for the remainder of the segment.

(f) Due to the high redox level in the initial batch of Auxiliary feed, the glass ferrous-to-ferric ratio never reached equilibrium.

FIGURE 2. Air Displacement Slurry Pump II

meter to the melter was 0.64 cm O.D. x 0.09 wall thickness (0.25 in. x 0.04 in.) stainless steel tubing for a majority of the run. During the portion of the run when no sugar was added to the feed the feed line was changed to 0.97 cm O.D. tubing in an effort to decrease the problems with degradation of pump output at higher feedrates. It was obvious after only

FIGURE 3. Control Configuration for ADSP II

a short time that the 0.97 cm O.D. feed line did not generate sufficient back pressure to allow for acceptable control of feed rates. A 1 psig increase in the initial pressure resulted in a 23 L/h increase in feed rate. Therefore, the feed line was switched back to the 0.64 cm O.D. tubing after only 3 hours.

For all but 60 hours of the test the ADSP II was utilized with only minor problems. The value for ADSP II Efficiency (85.9%) shown in Table 3 does not reflect the portion of the run when the ADSP was operational but not in service (see below). While the ADSP II was out of service, a Moyno progressive cavity pump was used to maintain feed to the melter. Problems

incurred with the feed system were mostly due to the accumulation of solids in the feedline from the pump chamber. The first significant pumping problem occurred after 227 h of operation. It was suspected that a plug had developed in the pump chamber when routine flushing procedures were unsuccessful in restoring feed. The ADSP was stopped, the feedline was switched to the Moyno, and feeding resumed. The ADSP was removed from the feed tank and disassembled. Visual inspection failed to identify any large insoluble material that could have plugged the 0.97 cm chamber outlet line. The pump was cleaned, reassembled and placed back in operation. Subsequently, the feed system plugged again after only 12 hours of operation. A solid plug was dislodged, using manual techniques, from the 0.97 cm tubing between the pump chamber and the flow meter. Only a small portion of the ADSP downtime initiated by the second plug was associated with pump failure. During the clearing of the pump chamber, the continuous filling of a SNL half-scale canister was started. It was decided that melter feed should not be interrupted until the canister fill was complete. Feed was maintained using the Moyno even though the ADSP was operational.

During PSCM-22 the melter feed system frequently showed signs of solids accumulation. While maintaining a constant air pressure, the pump output would decline over time (see Figure 4 - top graph). A water flush was generally used to clear the line and restore the output. An alternate solution was to increase the air pressure and boost the output back up to the original level (see Figure 4 - bottom graph). Many times, the increased pressure would maintain output for a period of time and then it would begin to increase. Solids were being worked through the feedline, the feed rate would increase, and the air pressure would have to be reduced.

Overall, the feed system mechanically performed very well. The consistency of output over a wide range of feed rates, 25 L/h to 65 L/h was most acceptable while the 0.64 cm O.D. feedline was used. Pump output control needed to be adjusted frequently in order to maintain a desired feed rate. The amount of control adjustment required during the

FIGURE 4. ADSP II Signal Degradation

experiment was felt to be excessive. Additional development of the control system, possibly making use of a flowrate feedback or characteristic pressure response curve signal, should be investigated to improve pump operation.

The feed nozzle performed very well. Feed buildup around the tip of the feed nozzle was noticed only during the start-up sequence of the experiment when the plenum was at approximately 800°C. A water flush successfully removed the solids accumulation.

Melter System

The pilot-scale ceramic melter (PSCM) system used during this experiment is located in Module 17 of the Experimental Development Laboratory, 324 Building (see Figure 5). The PSCM is a joule-heated melter and is used in the production of simulated high-level waste borosilicate glass.

The pilot-scale melter has an exposed glass surface of 0.73 m² (7.85 ft²). During operating conditions, the melter maintains a glass inventory of approximately 290 L (725 kg) of glass based on a glass density of 2.5 kg/L at 1150°C. The surface area to volume ratio of PSCM is 2.5 m⁻¹ and the maximum depth of the melt volume is 42 cm.

Major operating parameters monitored during a melter test are glass temperatures, electrode power, melter tank electrical resistance, plenum temperature, and vacuum. Glass temperatures are monitored using seven calibrated Type K thermocouples placed in a 2.54 cm O.D., 0.64 cm wall thick, Inconel-690 thermowell. Figure 6 shows the location of the thermowell, along with additional support equipment, in the melter lid. Individual thermocouples were located at 11.0, 20.3, 27.9, 35.6, 38.1, 40.6, and 43.2 cm from the melter floor. Average glass temperature was calculated using an average of the temperatures read by the 11.0, 20.3, and 27.9 cm thermocouples. Plenum temperature was measured by a Type K thermocouple located in the plenum space approximately 61 cm (24 in.) from the top of the melt surface. Melter power (kW) and the melter electrical resistance (ohms) were electronically calculated from the electrode voltage and current values indicated by the primary RMS voltage and current meters used to monitor the electrode system.

FIGURE 5. Pilot-Scale Ceramic Melter (PSCM)

FIGURE 6. Melter Lid Arrangement for PSCM-22

Operational parameters for PSCM-22 were monitored using a Hewlett-Packard 9836 desk top computer with a 3497A Data Acquisition System (see Figure 7) and four calibrated Leeds and Northrup chart recorders. Data accumulated during the run was stored on the chart recorders and magnetic tape from the HP system. Key melter parameters identified above are plotted in Figures 8 and 9 as a function of run time for the three separate segments of the run; reductant added to the feed, no additional reductant, and melter feed from a second batch of reference feed (Auxiliary segment).

The operational control parameters for PSCM-22 were electrode power, average glass temperature, and plenum temperature. Constant power and constant average glass temperature control were used alternately throughout the run to maintain glass temperatures. This run was marked by wide variations in average glass temperature which made melter control difficult. Temperature swings were more pronounced during the segment of the run when no additional reductant was added to the feed (see Figures 8 and 9). Causes for the variations in glass temperature are not fully understood at this time. Because level bubblers were in use during portions of the run and convection bubblers were installed during the final stages, slow or erratic convection currents are not thought to have been a major contributor to the problem.

Following the run, the melter was drained and the lid was removed. Visual inspection of the melt cavity revealed damage to both the North and South plate electrodes. The North electrode had shifted toward the melter wall (east side) and two cracks were present along the face of the electrode. The South electrode was pulled approximately 6.4 cm (2.5 in.) from the melter side wall (west side) and the electrode had slumped. Most of the welds between the bus connection and electrode plate on both electrodes had been broken. Also, the lid refractory (Alfrax 66) on the east side of the melter above the tank lid flange had crumbled and fallen into the cavity.

The damaged state of the melter observed following PSCM-22 was symptomatic of the age of the melter and was not caused by this run. Electrode damage may have contributed to distortions in current densities within the melter during the run. The Alfrax 66 that had fallen into the tank had not fully dissolved and may have distorted the current path. The position of the settling sample

FIGURE 7. Hewlett-Packard Computer/Data Acquisition System

FIGURE 8. Operating Parameters (Slurry with Added Sugar)

FIGURE 9. Operating Parameters (Slurry without Added Sugar and Auxiliary Feed)

boat on the melter floor could also have contributed to the electrical problems within the melt. The boat was approximately 2.5 cm from the foot of the South electrode. The creation of a shorting path through the boat is quite possible. Especially when considering that the frequency of temperature fluctuations increased when the first boat lid was placed on the boat making the shorting path even longer. The close proximity of the boat and the thermowell could have resulted in the thermowell indicating localized heating caused by any shorting that occurred. The large thermal mass of the melt prevents the entire tank from fluctuating in temperature as frequently as was recorded by the glass thermowell thermocouples. A deficiency of the thermowell concept is the fact that its measurement is highly localized. It is interesting to note that upon initial installation the boat did not noticeably affect the electrode current flow. Apparently, the electrode voltage had to increase above the idling value of ~70 volts before any possible current shorting would occur. In any case, a single cause for the temperature fluctuations during PSCM-22 cannot be stated. The glass itself is not considered to have caused the glass melt temperature problems. HW-39 glass properties are very similar to other glasses tested in the past and no problems were encountered during HBCM-85-1 using essentially the same glass.

The plenum temperature was used to monitor the feed rate and cold cap coverage. For nominal feed rates the plenum was maintained within a range of $275 \pm 25^\circ\text{C}$. Maximum rates generated plenum temperatures of around $225 \pm 25^\circ\text{C}$ (see Figures 8 and 9). An excessive cold cap tends to be a good insulator and leads to higher glass temperatures while the plenum temperature decreases. Thus, by monitoring the plenum and average glass temperatures the operator obtains valuable information to assist in maintaining an acceptable feed rate.

Melter Instrumentation

The glass thermowell, pneumatic level detection system (bubbler, and the infrared camera were tested during PSCM-22. The time domain reflectrometer level detection system and the acoustic monitor were not used during the run due to problems with the equipment.

Glass Thermowell

The glass thermowell is used to monitor the glass temperatures within the melter. Seven Type K thermocouples were positioned 11, 20.3, 27.9, 35.6, 38.1, 40.6, and 43.2 cm above the melter floor in the 2.5 cm diameter, 0.64 cm thick Inconel 690 thermowell. The thermowell was located in the south rectangular port on the melter lid, just south of the film cooler (see Figure 6).

The lowest three thermocouples were averaged to define the bulk glass temperature. This average temperature was displayed on the Hewlett-Packard data acquisition system and was used by the temperature control algorithm, also on the HP (model 9836). The upper four thermocouples were located in the region of the glass/cold-cap interface as a possible detection method for foaming. No foaming occurred during the run so this feature was not tested. The 35.6 cm thermocouple burned out early in the run. Since it was located in the glass/cold-cap region, its failure did not significantly impact the current testing and evaluation.

Level Bubbler Probe

The pneumatic level detection system or level bubbler is used to determine the glass specific gravity and level in the melter and to monitor the stability of the cold cap including the occurrence of foaming. A new three-tube design bubbler was installed just prior to PSCM-22 because of corrosion failure of the original level bubbler probe (see Figure 10). The probe was installed in the north rectangular port on the melter.

Level detection using the probe appeared to be impacted by the pressure and flow rate of the bubbler air flow. Calibration of the bubble had not been possible due to the short time between its installation and the start of the run. The original bubbler had 0.64 cm I.D. tubes compared to 1.3 cm I.D. for the bubbler used during PSCM-22. Therefore, qualitative measurements such as stability were useful, however, level measurements were not reliable. Foam detection was not evaluated because no foaming incidents were generated during the test.

FIGURE 10. Glass Pneumatic Level Detection System Schematic

Infrared Camera

The infrared camera was not installed until the final week of PSCM-22. Before the run started, the nose piece protecting the lenses and mirrors extending into the melter plenum was found to have corroded through such that cooling to the lenses was lost. This necessitated refabricating several parts of the camera housing which were constructed of 304 L stainless steel.

Installation was further delayed by a short circuit in the electronics of the camera itself. A buildup of glass and corrosion products in the melter lid nozzle sleeve through which the camera is installed to look into the melter had to be removed before the camera could finally be installed. Picture quality was not as good as with previous melter runs. This is probably a result of changes in critical distances within the optical path caused by the refabrication of the camera housing.

Constant Temperature Controller

A temperature control algorithm has been developed to control electrical power to the melter using glass temperatures as feedback to the controller. A cascade control scheme is employed to achieve the desired control. The Hewlett-Packard computer/data acquisition system serves as the primary control loop and calculates the power setpoint based upon the average glass temperatures and the glass temperature setpoint. A 0-10 volt signal is sent from the HP to a Research Incorporated power controller which then provides PID control of the power at the specified setpoint.

The PSCM has been operated using constant power, resistance or current as the method to maintain glass temperatures within the desired range. Each of the control methods has its drawbacks, however. Since a constant temperature, or at least a temperature within a specific range, is required a controller using the glass temperatures as the feedback should provide the necessary control. Temperature fluctuations associated with the convective currents in the melter have precluded employing this scheme in the past. The addition of the Hewlett-Packard computer to the PSCM instrumentation/data acquisition system now permits more sophisticated control schemes.

Figure 11 shows the flow diagram for the temperature control algorithm written for the HP computer. The algorithm is a subroutine of the data acquisition and display program currently in use with the PSCM. The subroutine is called each time a data scan is performed by the computer although the algorithm can be implemented at any integral multiple of the scan rate. Even if the algorithm is not implemented on a particular scan, the controller parameters are stored with the other run data.

FIGURE 11. Constant Temperature Control Algorithm

The algorithm starts by reading the glass temperatures, checking for foaming, and determining if a thermocouple has burned out. A thermocouple is assumed to have burned out if the indicated temperature is outside an 800-1250°C range. The average glass temperature is then calculated from those temperatures within the acceptable range. A digital filter (Equation 1) is then used to smooth the average glass temperature.

$$T_{\text{filter}}(n) = (1-a)T_{\text{filter}}(n-1) + aT_{\text{ave}} \quad (1)$$

The smaller the value of the filtering parameter, a , the more smoothing. A value of 0.3 for the filtering parameter produces about the same smoothing as a one-hour time average of data taken every five minutes. The digital filter does introduce a time delay between the measured and filtered glass temperatures.

The error between the setpoint temperature and the filtered average glass temperature is then calculated. If the error is less than an acceptable deviation from the setpoint (i.e., the melter has frequently been operated at 1150 + 25°C) then no further changes are made and the controller parameters are stored. The algorithm does not automatically adjust the power setpoint if the glass temperature error is outside the given range. Significant lag time is associated with changes in glass temperature due to changes in power setpoint. Thus, the algorithm checks the time elapsed between setpoint changes. If no power change has occurred within a defined period of time the power setpoint is adjusted. If a power change has occurred, the algorithm is designed to disregard the glass temperature error and allow the system time to respond to the previous setpoint change. Also, when the algorithm checks the glass temperature error it compares the current value with the error from the previous scan. If the temperature error for the present scan is significantly larger than the previous value, the algorithm will lower the power setpoint. This feature is designed to combat the rapid increase of glass temperature associated with melt foaming.

The control algorithm can function as a proportional (P), proportional-integral (PI), proportional-derivative (PD), or proportional-integral-derivative (PID) controller. The controller calculates the necessary setpoint change which the HP sends as a 0-10 volt signal to the Research Incorporated (RI) power controller. The RI controller then provides PID control of the power at the specified setpoint. This dual controller scheme is called cascade control with power as the feedback in the secondary control loop and temperature in the primary control loop. SRL also plans to use cascade control with their melter system.

For PSCM-22, a foam detection scheme was added to the control algorithm. During foaming incidents, glass temperatures increase rapidly throughout the melter and temperatures measured by thermocouples in the cold cap region increase as the foam rises. The foam detection scheme looks for this characteristic increase in temperature in the region of the cold cap. An alarm would sound and the power setpoint would be reduced to zero if the glass temperatures at the 40.6 and the 43.2 cm (16 and 17 in.) levels exceeded 1050°C and 1100°C, respectively. These temperatures are higher than those measured during idling so that the alarm will not sound when the cold cap has burned off or when the melter is idling.

PSCM-22 was characterized by severe temperature fluctuations which made control of temperature and power very difficult. As an example, late in the run, indicated glass temperatures dropped 100 degrees in a five minute period. On another occasion, electrode power levels greater than 90 kW could not maintain glass temperatures above 1120°C when temperatures 50-100°C higher would have been expected.

Several changes in the control parameters were made to try to improve temperature and power control under these difficult conditions. While the changes were not successful in achieving control, additional information was gained regarding the response of the temperature control algorithm. The PID control mode appeared to work well during the initial startup of feeding to the melter. Power levels were adjusted appropriately by the controller to account for the increased load as the feed rate increased and the cold cap grew. Decreasing the effect of the digital filter used to smooth the average glass

temperatures over time resulted in a quicker response to the temperature changes. The algorithm was designed to not make any power setpoint changes as long as the average glass temperature was within an acceptable range around the setpoint (i.e., $\pm 10^{\circ}\text{C}$). Nor were power setpoint changes to be made on a frequency greater than 5 to 10 minutes. The acceptable temperature deviation and the frequency were both selected by the operators based on PSCM operating history. During PSCM-22, the algorithm was actually operated for periods of the test with a zero acceptable temperature deviation (i.e., $\pm 0^{\circ}\text{C}$) and an update frequency shorter than the one-minute scan rate of the HP data acquisition system. In this mode, the power changes were smooth and gradual rather than the large step changes observed with former control parameters. Additional experience is needed to verify that these changes result in better control of glass temperatures and melter power.

Off-Gas System

The configuration of the off-gas equipment for the PSCM is shown in Figure 12. In this experiment, the off-gas train included (in order) the film cooler, the ejector-venturi scrubber (EVS), the NO_x absorption column, the Hydrosonic[®] scrubber (HSS) or steam atomized scrubber (SAS), and the high efficiency mist eliminator (HEME). The gas routing was maintained throughout the experiment, however the HSS was operated with injection of water and motive air during only a part of the run. During the remainder of the test, the off-gas passing through the HSS was not treated.

Film Cooler Performance

The stainless steel film cooler (see Figure 13) used in previous PSCM experiments was badly corroded following PSCM-21. No repair was possible so a new film cooler was built for this experiment. The new film cooler was built of 304L stainless steel, and the inlet section was electroplated with platinum to reduce the corrosive attack by acid gases (primarily the nitrogen oxides). The pitting type of corrosion seen following PSCM-21 was not present on the film cooler following PSCM-22. Because the concentration of nitrates in PSCM-22 feed was much lower than previous runs the desirability of platinum coating melter equipment has not been established.

FIGURE 12. Off-Gas Treatment System

Control of air to the film cooler was changed from earlier experiments. In PSCM-19, the film cooler air flowrate was modulated to control the vacuum in the melter. The fluctuating air flowrate frequently exceeded the velocity limit for laminar flow and a pattern of solids accumulated on the trailing edge of the injection slots. In this experiment, the air flow to the film cooler was maintained between 35 and 40 cfm, and was independent of the melter vacuum. An additional stream of air was introduced into the off-gas line downstream of the EVS to the NO_x absorption column to control melter vacuum. The

FIGURE 13. Film Cooler Cross-Section Details

inside surface of the film cooler was notably cleaner than following PSCM-19 and the coating of solids which did collect were quite easily removed.

Figure 14 shows the melter leakage as a function of melter vacuum. The data points shown were calculated from a heat and material balance across the film cooler during periods in which there was no feed to the melter and the cold cap had burned off. The curve shown can be described by Equation 2:

$$\text{PSCM leakage (cfm)} = 30 \times [\text{melter vacuum, inches H}_2\text{O}]^{1/2} \quad (2)$$

FIGURE 14. Inleakage vs Melter Vacuum.

This relationship is of the form for pressure drop across an orifice, which is consistent with the nature of inleakage flow. The relationship is useful in estimating the contribution inleakage makes to the total off-gas flow at various melter vacuum conditions.

The nominal 7.6 cm (3 in.) diameter jumper from the film cooler to the EVS was quite clean following the experiment. Off-gas velocity in the line was nominally 60 ft/s during periods of feeding to the melter. The flow was approximately 70 cfm inleakage, 35 cfm film cooler air, and 15 cfm steam from evaporating feed. The injection air used for pressure control was not directly measured, but was estimated to be about 40 cfm. This rate was determined by noting the change in total off-gas flow when the off-gas valve was throttled back until the melter vacuum control injection air automatically reduced to zero. Hence the total off-gas flow entering the NO_x absorber was about 150 cfm, since most of the steam from the melter condensed in the EVS.

NOx Absorber Performance

The NOx absorber was operated with a recirculating stream of water which was introduced through a distributor at the top of the column. The gas flow entered through a 10 cm (4 in.) inlet pipe near the bottom of the column and about 30.5 cm (12 in.) below the bottom of the packing support. The absorber was a 30.5 cm diameter column packed with 195 cm (6 ft 4 in.) of Gempak 4A structured packing. All parts of the column were 304 or 304 L stainless steel. The aqueous scrubbing solution was cooled in a jacketed recirculation tank and the nominal solution temperature entering the column was 26°C. The water flow to the column was maintained at 19.7 LPM (5.2 gpm) throughout the experiment. Based on the online NO/NOx analyzer, the column removed only a negligible percentage of NOx from the gas stream.

The efficiency predicted for the column would be similar to the efficiency for one or more trays of a bubble cap column, depending on the relative efficiency of the packing to the tray. Figure 15 shows the relationship of the NOx concentration in the inlet gas to the overall tray collection efficiency. For partial pressures (or mole fractions) lower than 0.01, the collection efficiency falls drastically. The inlet mole fraction of NOx flow to the packed column absorber during PSCM-22 was nominally 0.002. At this concentration, the extrapolated absorption efficiency would be less than 20%. Pressure drop across the column was nominally 5.0 inches H₂O and is shown in Figure 16.

Hydrosonic Scrubber

The Hydrosonic Scrubber (HSS), also called the Steam Atomized Scrubber (SAS), was tested for the last three days of the experiment with motive air and steam. The air supply was cleaned through a coalescing type filter to remove solid or liquid material. The motive air was maintained at 20 cfm, which corresponded to an inlet pressure of 50 psig. The scrubbing water was recirculated from Tk-21, and maintained at 3.8 gpm throughout the three days of operation. The pressure drop across the HSS was 10 inches (water column) when no motive air or water was injected, and nominally 23 inches WC with the air and water injection.

Source: M. S. Peters, University of Illinois Engineering Experiment Report No. 14, USAEC-COO-1015 (1955).

FIGURE 15. Effect of NO_2^* Concentration in Entering Gases on Plate Efficiency With a Three-Plate Bubble-Cap Column Obtained by Peters

High Efficiency Mist Eliminator

The HEME was operated with the gas flow entering the top and flowing from the inside to the outside of the cartridge element. The bottom of the element was blanked off to prevent gases or liquids from bypassing the element. This arrangement is shown in Figure 17. An atomizing spray nozzle was installed

FIGURE 16. Pressure Drop vs. Run Time (NO_x column)

FIGURE 17. High Efficiency Mist Eliminator

near the top of the element and on the centerline, so that the spray pattern would cover the entire element. A new polypropylene fiber element was installed for this experiment.

During the first ten days of the run the HEME was operated without the spray. The pressure drop in this period was stable at about 10 inches. After sampling for this period was completed, the off-gas was bypassed around the HEME and a water spray was turned on to flush the element for two hours. The HEME was put back online with a water spray rate of 16 ml/min. Figure 18 shows that the pressure drop across the element started out at a higher value than during dry operation (14 in. vs 10 in.). The pressure drop steadily increased during this period, indicating an accumulation of material in the element. The combination of high gas flow velocities and water seem to promote penetration

FIGURE 13. Pressure Drop vs. Run Time (HEME)

of the particulate material deep into the element. The gas face velocity at the inside surface of the element was 0.7 ft/s, which is at the high end of the normal design range recommended by the manufacturer.

The HEME collection efficiency was correlated to total gas flowrate and showed dramatically different effects for aerosols and larger sized particles. Figure 19 shows that the Ru DF decreased as the gas flow increased, and the Fe DF increased with total gas flow. This is consistent with the mechanisms governing particle collection. Past experiments have shown that Ru exists as an aerosol in the melter off-gas, and Fe exists predominantly as larger ($>10\mu$) particles. Larger particles are collected by inertial means, so a higher gas velocity should improve collection. Aerosols are collected by Brownian motion, hence a higher velocity will decrease the time particles spend within the fiber element and correspondingly decrease the probability of collection.

FIGURE 19. HEME DF vs Off-Gas Flow

FEED PROCESSABILITY

Processing Parameters

Acceptable processability of a melter feed is based on how well the feed behaves in the overall melter system. Some of the significant parameters used to define an acceptable feed are outlined below.

Feed Rate

The rate at which feed is processed in the melter is a critical characteristic of a feed. The higher the feed rate the more desirable the specific feed. A nominal feed rate is usually defined as one that generates not more than 80% cold cap coverage while not allowing excessive buildup of feed or cooling of the plenum (plenum temperatures around $275 \pm 25^{\circ}\text{C}$ for this experiment). A maximum feed rate occurs when the cold cap approaches 100% and the plenum temperature has been reduced to approximately $225 \pm 25^{\circ}\text{C}$ during PSCM-22.

Cold Cap Characteristics

The behavior of the cold cap is qualitatively determined by the amount of glass surface covered, cap thickness, venting, and flexibility.

Coverage. Coverage should be maximized to optimize feed rate. Actual coverage sought will depend on venting ability, thickness, and flexibility of the cold cap. Excessive cold cap coverage can cause "bridging," a situation where the cold cap attaches to the interior refractory walls of the melter tank and separates from the glass surface. Bridging results in an insulating layer of gases between the cold cap and the molten glass and causes a drop in feed rate due to a reduction in heat transfer between the melt and the slurry pool.

Thickness. Eventual thickness of a cold cap is dependent on meltability, feed rate, and spreading tendency of the feed. An unacceptably thick cold cap serves as an insulating blanket and can result in higher than normal glass temperatures and low plenum temperatures. A thick cold cap limits heat transfer from the melt to the slurry pool on the cold cap.

Venting. Venting is a measure of a flexible cold cap and indicates good contact between glass surface and cold cap. Decomposition gas is released from beneath and within the cold cap to the plenum through the vents, allowing a

high surface coverage and, hence, higher feed rates. Vents are usually characterized by bubbles of molten glass extending through the cold cap.

Flexibility. Cold cap flexibility and thickness are usually directly related. A thick cold cap is generally very rigid and unyielding while a thin cap flexes and cracks occasionally allowing for direct contact between the melt surface and the slurry pool. A thin flexible cold cap is preferred.

Rheology

Feed rheology refers to the viscosity, yield stress, and solids suspendability of the melter feed. In general terms, it is a measure of how well a slurry pumps and flows through the system without dropping an excessive amount of solid particles. Although not characterized, how well a slurry will flow at plenum temperature conditions is very important to processability. A slurry that will spread over the entire cap is preferred over one that quickly thickens forming a feed pile directly beneath the feed inlet nozzle. Factors which are believed to affect this slurry property are concentration of dissolved solids, viscosity, sugar concentration, and concentration of easily evolved volatiles such as nitrates.

Glass Properties

A melter feed must also yield a glass with properties within an acceptable range of values. The more important glass parameters from an operational standpoint are listed below.

Viscosity

The glass viscosity should be stable with respect to time and temperature. The temperature at which the glass has a viscosity of 100 poise (T100P) should be between 1070°C and 1150°C. A higher viscosity may lead to a decrease in the interaction between the glass melt and the cold cap and lower processing rates. A lower viscosity can allow the glass to penetrate between the melter bricks and also cause an increase in brick and electrode corrosion due to high mixing currents.

Electrical Conductivity

In joule-heated melters of the PSCM design, the electrical conductivity of the glass melt should be between 0.18 and 0.5 (ohm cm)⁻¹ at 1150°C. If the electrical conductivity of the glass is too high, the current required to heat the glass will exceed the recommended maximum current density for the melter electrodes of 1 amp/cm². If the electrical conductivity is too low, the resulting voltage potential of the electrodes would be such that conduction could occur within the melter refractories.

Absence of Excessive Phase Separation

Phase separation can occur with the formation of crystals within the melt or as a layer of immiscible second glass phase or molten salts. Crystal settling on the melter floor (sludge) can reduce the operating lifetime of the melter and in extreme cases lead to an electrical short between the electrodes. An immiscible layer impedes the flux that occurs between the cold cap and the melt surface and leads to lower feed rates. Accumulation of a molten salt layer is undesirable because it would require periodic feed interruption for removal. In extreme circumstances there is the potential for violent salt-water interactions.^(a)

Tendency to Reboil

Reboil or melt foaming tendency occurs when multivalent metals in the melt change oxidation state (become more reduced) and in the process release oxygen. The stable valence state of the metals is a function of temperature. Thus, as the melt experiences a rapid local temperature increase, usually caused by mixing convection cells, oxygen is released and foam results. The tendency to and severity of foaming is based on the concentration of multivalent metal species such as Fe, Mn, and Cr. The feed chemistry is normally adjusted to maintain the melt in a reduced condition to prevent spontaneous reboil.

(a) Hutchenson, M. N., R. E. Henry, D. J. Pellarin. 1984. Annual Meeting of the American Nuclear Society - Transactions.

Leachability Characteristics

Glass quality is evaluated based on leaching behavior or glass dissolution rates using standard Materials Characterization Center (MCC) tests.

Feeds Utilized During PSCM-22

PSCM-22 was split into three separate segments based on the type of feed being processed at the time. The three feeds utilized during the experiment were:

Slurry with Added Sugar - with a concentration of 3.5 grams sugar per liter of melter feed. The target ferrous-to-ferric ratio was 0.10 (see the section on Melter Feed and Glass Analysis).

Slurry without Added Sugar - was an attempt to generate a slightly oxidizing glass melt in order to test the foaming tendencies of HW-39. Because this feed turned out to be slightly reducing because of the formates, it contained an oxidizing melt was not obtained and did not foam.

Auxiliary - Because the auxiliary melter feed was made with a different batch of reference feed, this segment of the run was treated separately. No sugar was added to this feed; but due to chloride contamination from the ferric hydroxide the redox level of the melter feed was overly reducing initially (see the section on Melter Feed and Glass Analysis).

Average feed rate vs run time is plotted in Figure 20. The single points that appear in the graph are actually six hour average values and the value reported as the average feed rate over the identified segment is an average of all the actual hourly feed rates during that portion of the run. Start up refers to a change over period where the melter achieves stability and glass in the melter is replaced by glass that has the same composition as the melter feed. Nominal feed rates are rates that can be maintained for indefinite periods of time because operational parameters all fall within accepted ranges as outlined above. On the other hand, a maximum feed rate for a particular melter feed pushes the system to the limit. One or more of the standard parameters

FIGURE 20. Average Feed Rate vs Run Time (Based on Totalizer)

are on the verge of being exceeded. Feed surges may then lead to instability because little, if any excess capacity remains.

Slurry with Added Sugar

During the initial 187 hours (approximately 8 days) of PSCM-22 the melter feed being processed contained added sugar. Sugar has been shown to raise the redox potential of the borosilicate glass (as measured by the ferrous-to-ferric ratio) which in turn increases the stability of the glass. The amount of reductant (sugar) added to the feed was determined to be 3.5 g/L and was based on laboratory experiments (see the section on Melter Feed and Glass Analysis). Start up of the feed lasted three days with the feed rate being maintained at an average value of 32.3 L/h. The cold cap ranged from 60 to 75% and good flexibility and venting was observed. Cold cap thickness ranged from 2.5 to 5 cm based on visual observations. Plenum temperatures remained high ($>300^{\circ}\text{C}$). Following start up, a nominal feed rate was established at 36.9 L/h and maintained for approximately 120 hours. The feed rate was pushed up to a maximum average value of 40.9 L/h for a total of 18 hours just prior to switching over to a feed without added sugar. Cold cap thickness ranged from 4.0 cm to 6.0 cm for the nominal rate and from 5.0 cm to 6.5 cm for the maximum feed rate.

Based on predictions from the April High-Bay melter run (HWVP-11), these feed rates met or exceeded expectations. As stated in the Run Plan for PSCM-22, the nominal rate for the feed containing sugar was targeted at 32 L/h and the maximum rate was 38 L/h. These numbers were based on the rates determined during the 100% frit feed portion of HWVP-11. Melter feed from the two different experiments were similar in oxide composition and oxide loading. Ferrous-to-ferric ratios established during HWVP-11 (100% frit feed) were between 0.04 and 0.004 whereas for PSCM-22 the melter feed was maintained very close to 0.10.

During both nominal and maximum processing the cold cap generated by the feed was very thin and flexible. Vents were numerous and the glass surface was active, even vigorous at times. Large amounts of "hair" formed during this segment of the run. "Hair" refers to strands of glass that are ejected from vents in the cold cap and attach and cool on solid surfaces above the glass

surface. Major buildup was experienced on the feed nozzle, the view port and the thermowell. Towards the end of this segment, when the six hour average feed rates were up to around 50 L/h, the cold cap thickness grew to approximately 7.5 cm and the plenum temperature dropped to around 210°C. The characteristics of the melt were judged acceptable.

Slurry without Added Sugar

After 186 hours of operation, feed to the melter was shut down and the cold cap was allowed to melt off. Remaining feed with added sugar (almost 1000 L) was transferred from the feed tank and stored in 55 gallon drums. The feed tank was filled with a melter feed that did not contain added sugar. This feed was fed to the melter for approximately 198 hours and essentially utilized the remaining reference feed from the original Tk-69 batch.

The start up portion of this segment of the run was shorter than the initial startup (18 hours as opposed to 66 hours) because the oxide composition of the glass was not being altered; only the redox potential of the glass was changing and plenum and refractory temperatures were already in the desired range. Tank turnover is much quicker in terms of redox potential as opposed to oxide composition. Average feed rate for the start up was 35.6 L/h, the nominal rate was 44.5 L/h (96 hours), and the maximum rate established and maintained was 52.5 L/h (78 hours). Processability for the two feeds was similar in all aspects except feed rate where the feed without sugar was clearly superior. It is hypothesized that the difference in processing rates is based on the condition of the cold cap/melt interface. As redox potential decreases, secondary crystal phases form and influence fluxing of the cold cap into the melt. The crystals dissolve more slowly and become rate limiting. It is still not clear if the nature of the reductant is important.

Even though no sugar was added to the feed during this segment of the run the ferrous-to-ferric ratio of the glass remained greater than zero (see the section on Melter Feed and Glass Analysis). For this reason the glass melt remained stable throughout feeding even though several rapid temperature spikes within the glass occurred, providing the potential for a foaming incident.

Auxiliary Feed

The Auxiliary segment of PSCM-22 lasted for the final 30 hours of the test. This segment was separated from the second feeding period by the reference feed batch used to make up the melter feed. Both of the previous melter feeds utilized reference feed from the original batch. The Auxiliary feed was made from a second batch that was made up during the last week of the run. The reference and auxiliary feed batches were closely matched compositionally except for the concentration of chloride (see Table 2).

The processing rate for the Auxiliary feed started out at around 40 L/h but dropped rapidly. This was probably due to the minimized redox potential (initially a ferrous-to-ferric ratio of 1.2) generated by the chloride present in the feed (see the section on Melter Feed and Glass Analysis). In an effort to lower the ferrous-to-ferric ratio, NaNO_3 was added as an oxidant. Following the NaNO_3 addition, the feed $\text{Fe}^{+2}/\text{Fe}^{+3}$ level dropped to 0.028. Correspondingly, the feed rate was increased steadily to the end of the run. The average feed rate for the entire Auxiliary segment of the test was 39.5 L/h.

During the initial portion of the Auxiliary segment, the cold cap generated by the highly reducing feed was very thick and plenum temperatures were in the low section of the control range. These characteristics were most probably due to overfeeding the melter as opposed to traits of the feed. Once the redox potential was adjusted and the feed rate increased, the cold cap behavior resembled the earlier segments of the run.

MELTER FEED AND GLASS ANALYSIS

RESULTS OF FEED ANALYSIS

As outlined in the Run Plan for PSCM-22, daily 0600 h feed and glass samples were sent for compositional analysis. Standard methods of analysis included induction coupled plasma (ICP) spectroscopy, ion chromatography (IC), flame atomic absorption (AA), X-ray fluorescence, and specific ion electrode. Table 4 shows the results of these analyses. Based on standard deviation values from target concentrations, the feed composition was consistent for the overall test (see Figure 21). However, as is apparent from the individual data, there were fluctuations greater than the percent deviation. Also, as stated earlier, the Auxiliary feed matched up well with the previous feeds. Major deviations from target values were confined to minor constituents like CeO_2 , MnO_2 , and ZrO_2 (BaO , TiO_2 , and Co_2O_3 were used in tracer studies; fluctuations in concentration and deviations from target values were expected). The Run Plan also stated that major components, such as SiO_2 , Na_2O , B_2O_3 , and Fe_2O_3 , would be maintained within 5% of target values. Table 4 shows that the mean concentration of SiO_2 and Na_2O exceeded this range, but only marginally. Overall, the composition of melter feed utilized during PSCM-22 was acceptable (390 ± 19 g total oxide/liter of feed).

It is apparent from Table 4 that the concentration of the majority of the waste constituents (HWVP-RF stored in Tk-69) dropped during the last third of the experiment. Tk-69 waste fraction samples, additional feed samples, and the original feed analyses were evaluated to try and explain the drop in concentration. Additional feed samples taken during the section of the run when the drop in concentration was most apparent were analyzed. Ferric oxide and silica dioxide were studied to estimate the variation in sample analysis. Variation in the ferric analysis was found to be ± 0.65 wt% of the reported value. The silica values were found to be ± 0.9 wt% of the reported value. These values are comparable to the standard deviations reported.

The additional feed samples that were analyzed only verified the original results. Tk-69 waste fraction sample analysis did show results that indicate an inadequately mixed batch. Waste samples from Tk-69 were routinely taken

TABLE 4. Compositional Analyses - Feed and Glass Samples

Date	Time hr:min	Run Time hr.	Sample #	Al2O	B2O3	BaO	CaO	CaO2	Co2O3	Cr2O3	Cs2O	Fe2O3	Height Percent La2O3	Li2O	MgO	MnO2	MoO3	Na2O	Nd2O3	NiO	RuO2	SiO2	SrO	TiO2	ZrO2	Total Oxides g/l	Density g/ml	
FEED																												
7/16	600	13	54	5.01	9.31	0.13	2.85	0.15		1.39	0.37	11.90	0.67	4.00	0.95	0.22	0.32	11.80	0.53	0.60		48.57	0.10	0.08	0.75	374.9		
7/17	600	37	103	4.90	9.28	0.14	2.82	0.19		1.35	0.33	11.59	0.65	3.94	0.90	0.22	0.29	10.87	0.52	0.58		49.60	0.10	0.08	0.61	383.5	1.213	
7/18	600	61	144	4.83	9.23	0.14	2.80	0.19		1.31	0.32	11.24	0.69	3.96	0.87	0.21	0.30	10.65	0.51	0.56		48.34	0.10	0.08	1.11	388.9	1.227	
7/19	600	85	200	4.61	9.07	0.28	2.78	0.19	0.16	1.29	0.32	10.82	0.80	3.87	0.88	0.21	0.26	10.64	0.53	0.56		47.62	0.09	0.29	0.62	392.7	1.225	
7/20	600	109	262	4.81	9.62	0.30	2.88	0.11	0.16	1.31	0.31	11.17	0.79	4.05	0.88	0.21	0.28	10.97	0.54	0.57		49.36	0.10	0.30	0.66	393.0	1.227	
7/21	600	133	306	4.66	9.47	0.29	2.85	0.13	0.07	1.34	0.31	11.15	0.71	4.02	0.87	0.21	0.29	11.02	0.51	0.58		48.61	0.10	0.29	0.68	392.9	1.232	
7/22	600	157	365	4.78	9.68	0.30	2.90	0.10	0.03	1.33	0.31	11.30	0.63	4.12	0.86	0.21	0.29	11.12	0.50	0.57		49.33	0.10	0.30	0.69	383.1	1.239	
7/23	600	181	420	4.68	9.32	0.30	2.79	0.13	0.01	1.40	0.33	11.48	0.62	3.95	0.84	0.22	0.29	10.90	0.51	0.61		47.08	0.10	0.29	0.63	397.2	1.244	
7/23	600	196	449	4.62	9.03	0.31	2.74	0.14		1.35	0.21	11.31	0.58	3.87	0.86	0.21	0.29	10.92	0.51	0.59		47.18	0.10	0.31	0.64	396.0	1.224	
7/24	600	205	470	4.65	9.09	0.31	2.75	0.12		1.36	0.21	11.32	0.54	3.92	0.85	0.21	0.29	10.94	0.50	0.59		47.01	0.10	0.31	0.64	385.0	1.219	
7/25	600	229	518	4.65	9.09	0.31	2.75	0.12		1.41	0.31	11.56	0.54	3.84	0.86	0.21	0.29	10.75	0.50	0.61		45.97	0.10	0.32	0.65	385.0	1.220	
7/26	600	253	567	4.64	9.27	0.32	2.82	0.12		1.39	0.38	11.59	0.52	3.90	0.89	0.21	0.30	11.56	0.49	0.60	0.07	47.95	0.10	0.33	0.67	379.6	1.207	
7/27	600	277	618	4.38	9.16	0.30	2.81	0.16		1.30	0.17	10.68	0.51	3.88	0.88	0.20	0.28	10.31	0.49	0.58	0.07	48.03	0.09	0.34	0.61	381.0	1.221	
7/28	600	301	664	4.26	9.39	0.31	2.92	0.17		1.29	0.18	10.26	0.50	3.97	0.90	0.20	0.27	10.39	0.47	0.58	0.05	49.21	0.09	0.33	0.61	380.0	1.225	
7/29	600	325	716	4.11	9.39	0.29	2.89	0.15		1.26	0.16	10.08	0.50	3.98	0.89	0.19	0.27	10.42	0.46	0.57		49.34	0.08	0.33	0.58	377.0	1.221	
7/30	600	349	733	3.89	9.52	0.34	2.95	0.12		1.11	0.21	9.34	0.43	4.02	0.89	0.17	0.24	10.69	0.45	0.50		49.80	0.08	0.33	0.56	382.5	1.216	
7/31	600	373	788	3.97	9.40	0.39	2.88	0.10	0.52	1.14	0.14	9.48	0.43	3.97	0.87	0.17	0.24	10.57	0.42	0.50		49.53	0.08	0.33	0.57	375.5	1.205	
8/01	600	397	829	4.35	9.23	0.32	2.84	0.12	0.07	1.28	0.15	9.64	0.50	3.90	0.85	0.18	0.25	10.68	0.41	0.51		48.73	0.09	0.14	0.53	461.7	1.242	
8/02	600	421	873	4.42	9.21	0.32	2.81	0.14	0.06	1.30	0.18	9.80	0.52	3.88	0.88	0.19	0.25	12.61	0.43	0.52		49.23	0.10	0.15	0.53	426.4	1.264	
Target				4.30	9.36	0.10	2.89	0.18	NR	1.33	0.25	11.10	0.55	3.75	0.83	0.18	0.30	10.37	0.53	0.60	NR	51.19	0.10	NR	0.60	400.0	NR	
Mean				4.54	9.30	0.28	2.83	0.14	0.12	1.31	0.26	10.83	0.59	3.95	0.88	0.20	0.28	10.94	0.49	0.57	NR	48.42	0.09	0.26	0.65	390.8	1.226	
Std dev				0.30	0.18	0.07	0.06	0.03	0.15	0.08	0.08	0.79	0.11	0.07	0.02	0.02	0.02	0.53	0.04	0.03	NR	1.02	0.01	0.09	0.12	19.48	0.014	
GLASS																												
Total Ht %																												
6/24	2240	NR	36	3.96	10.80	0.08	1.39	NR	NR	0.06	NR	12.40	0.14	4.33	1.60	3.62	0.03	12.90	0.05	NR		41.70	0.01	0.70	0.59	94.70		
7/16	615	13.3	60	5.47	10.40	0.09	1.92	0.13		0.58	0.08	12.14	0.18	4.26	1.53	2.74	0.06	12.50	0.14	1.04		45.00	0.04	0.55	0.60	99.20		
7/17	302	34.0	93	5.29	9.66	0.11	2.23	0.15		0.77	0.14	11.80	0.44	4.10	1.29	1.73	0.07	11.90	0.34	0.88		45.40	0.06	0.37	0.62	97.50		
7/18	534	60.6	140	5.14	9.40	0.12	2.46	0.22		0.96	0.15	11.40	0.54	4.00	1.14	1.11	0.15	11.00	0.45	0.77		47.10	0.08	0.25	0.57	97.20		
7/19	326	82.5	191	5.14	9.28	0.16	2.61	0.27	0.13	1.03	0.15	11.30	0.59	3.87	1.11	0.73	0.16	10.70	0.53	0.72		47.60	0.08	0.28	0.59	97.20		
7/20	539	108.7	258	4.82	9.31	0.24	2.67	0.14	0.12	1.08	0.16	11.20	0.64	3.89	0.82	0.49	0.26	11.00	0.46	0.63		48.40	0.09	0.26	0.58	97.40		
7/21	441	131.7	310	4.75	9.04	0.25	2.71	0.17	0.10	1.17	0.14	11.00	0.65	3.85	0.81	0.38	0.27	10.70	0.47	0.59		48.40	0.09	0.28	0.64	96.60		
7/22	600	157.0	365	4.75	9.15	0.27	2.76	0.16	0.08	1.15	0.17	10.90	0.62	3.93	0.81	0.50	0.26	10.70	0.46	0.58		48.50	0.10	0.28	0.63	96.70		
7/23	545	180.8	417	4.82	9.13	0.29	2.76	0.17	0.05	1.22	0.19	11.20	0.60	3.94	0.82	0.28	0.26	10.90	0.47	0.58		48.70	0.10	0.30	0.63	97.60		
7/23	1157	187.0	441	4.83	9.10	0.28	2.79	0.19	0.05	1.21	0.20	11.20	0.60	3.87	0.84	0.26	0.26	10.70	0.49	0.59		48.60	0.10	0.29	0.62	96.50		
7/24	139	200.7	456	4.81	8.89	0.29	2.75	0.19	0.04	1.22	0.23	11.10	0.59	3.91	0.85	0.25	0.25	10.80	0.50	0.58		48.10	0.10	0.29	0.64	97.20		
7/25	555	225.0	516	4.79	8.95	0.30	2.83	0.14		1.20	0.23	11.10	0.57	3.88	0.81	0.24	0.26	10.80	0.47	0.56		48.00	0.10	0.29	0.62	96.50		
7/26	444	251.8	563	4.91	9.26	0.32	2.86	0.15		1.15	0.18	11.40	0.53	3.88	0.84	0.22	0.30	11.20	0.47	0.53		49.00	0.10	0.32	0.67	98.60		
7/27	620	277.4	623	4.77	9.14	0.32	2.80	0.17		1.25	0.21	11.30	0.54	3.97	0.90	0.22	0.25	11.10	0.50	0.59	0.05	49.00	0.10	0.33	0.67	98.50		
7/28	612	301.3	668	4.65	9.17	0.32	2.85	0.17		1.15	0.20	10.90	0.52	3.84	0.92	0.22	0.16	11.10	0.48	0.56	0.05	49.10	0.10	0.32	0.61	97.50		
7/29	600	325.1	713	4.60	9.12	0.32	2.84	0.19		1.19	0.19	10.60	0.50	3.87	0.94	0.22	0.26	10.90	0.49	0.59	0.05	49.40	0.10	0.33	0.60	96.80		
7/30	550	348.9	751	4.42	9.20	0.32	2.84	0.18		1.18	0.16	10.20	0.48	4.00	0.93	0.20	0.27	10.50	0.46	0.53		49.40	0.09	0.34	0.63	96.50		
7/31	520	372.4	792	4.34	9.37	0.34	2.97	0.22	0.04	1.09	0.23	9.73	0.45	3.84	1.02	0.20	0.13	10.80	0.48	0.53		50.40	0.09	0.34	0.56	97.30		
8/01	650	397.9	833	4.10	9.22	0.36	2.83	0.10	0.18	1.13	0.17	9.77	0.42	3.90	0.80	0.19	0.10	10.50	0.36	0.53		49.70	0.09	0.32	0.54	95.40		
8/02	603	421.2	878	4.47	9.53	0.37	2.96	0.15	0.18	1.18	0.19	10.30	0.46	4.06	0.94	0.20	0.12	11.00	0.44	0.53		51.40	0.09	0.30	0.60	99.60		
8/02	1200	427.0	890	4.55	9.10	0.37	2.90	0.11	0.11	1.11	0.14	9.98	0.47	3.82	0.89	0.19	0.20	11.00	0.41	0.57		47.30	0.09	0.23	0.52	94.32		
Target				4.30	9.36	0.10	2.89	0.18	NR	1.33	0.25	11.10	0.55	3.75	0.83	0.18	0.30	10.37	0.53	0.60	NR	51.19	0.10	NR	0.60	100.00

FIGURE 21. Feed Composition vs Run Time

at a slurry depth <1 m from the top of the slurry level. These samples have reported values very close to those reported during the second half of the experiment. This information strongly points to inadequate mixing in Tk-69. The design of Tk-69 includes a three impeller mixer. Although mixing action was very evident on the top surface of the slurry, it is very possible that each impeller developed separate mixing cells. It appears that the lower impellers (one or both) did not apply sufficient energy to move slurry into the upper cell. As a result, over time, a density or solids gradient developed. Since the feed transfer is from the bottom of the tank, the "enriched" slurry was used in the first half of the test leading to the concentration loss towards the end of the run.

Another feed component that showed inconsistencies between feed and glass concentrations was sulfur. The average feed concentration over the duration of the run of S0 was 0.27 weight percent based on IC analyses. Following the run, three glass samples from the later part of the test were analyzed for S0 concentration using ICP. The average concentration was 0.35 wt%. Information on off-gas losses for sulfur indicated that significant sulfur was evolved from the melter during operation (average DF was 1.8). The inconsistency of this data is attributed to the difference in analytical techniques used (detection limits for S0 using ICP were approached at these concentrations). The accuracy of the off-gas analysis is more reliable because of the consistency of the results. In addition, sulfur is a major component of the off-gas stream, and calibration replicates were used during sampling to assure accuracy. Further study of the mechanism of S0 release in the off-gas is needed.

RESULTS OF GLASS PRODUCT ANALYSIS

Three glass samples were taken each time a glass pour was made and at four hour intervals during periods when the melter was continuously pouring (SNL canister fills). Analyses were performed on samples from the final pour of graveyard shift, and results are shown in Table 4. Comparing the oxide concentrations in samples taken during the run with the glass in the melter at the beginning of the run (pre-glass), the melter glass composition steadily converged toward the target composition. By the end of the fifth day of testing, the tank glass composition of major components was essentially on target. The pre-glass resulted from a flush of the melter following PSCM-21. Approximately 1500 kg (3300 lb) of SRL 131 frit/calcline was used as a dry flush prior to the start of PSCM-22.

Figures 22 and 23 are plots of melter feed and glass composition (specific components) as a function of run time. These plots show that concentration of selected oxides in the glass samples follow that of the feed samples. A time delay, associated with tank turnover, is also clearly evident. The SiO_2 and Cr_2O_3 plots show how the concentration of these oxides in the glass approached target values. On the other hand, because the concentrations of Fe_2O_3 and Al_2O_3 were not consistent in the feed the last half of the run, their

FIGURE 22. Wt% vs Run Time (Feed and Glass)

FIGURE 23. Wt% vs Run Time (Feed and Glass)

concentrations in the glass did not reach an equilibrium and instead continued to track the concentration variations in the feed. The variation of waste concentration is explained above and is evident in Figures 22 and 23. Glass-forming chemicals did not exhibit the fluctuations of the waste components as additions were not made in Tk-69.

Convection bubblers were installed on Day 15, per discussions with Program Management, in an effort to increase mixing in the melt tank. The results presented in Figure 22 for Cr_2O_3 do not indicate an increase in the amount of Cr in the overflow glass samples. This suggests that the bubblers did not effectively increase spinel suspension.

RESULTS OF COLD CAP ANALYSIS

Cold-cap samples were taken during PSCM-22 for analysis. Samples of the cold cap were powdered and sent for ICP analysis. Thin sections were also cut and polished for optical and SEM/EDX analysis. The elemental compositions of the cold-cap samples were similar to the glass target compositions. No excessive concentration of any specific elements was observed. The crystalline material within the cold cap was limited to the spinel $((\text{Fe},\text{Ni})\text{Cr}_2\text{O}_4)$ phase observed in the bulk glass and a very few Zr-silicate crystals. The crystals appear to agglomerate in or near the cold cap. These results are identical to the HBCM cold-cap sample results.

REDOX POTENTIAL

Target $\text{Fe}^{+2}/\text{Fe}^{+3}$ for the two types of feed (with and without added sugar) tested during the run were outlined in the Run Plan to be 0.10 and 0.0, respectively. As in past experiments, a sample of melter feed was sent for laboratory analysis prior to the start of each segment.

Bottom-heated crucible techniques were used to determine the concentration of sugar needed to achieve the desired ferrous-to-ferric ratio for the first segment of the test. Figure 24 shows the curve that was used in choosing the value of 3.5 g sugar/L feed for this segment of the test. The laboratory curve was well defined by the number of experimental points, and 3.5 g/L predicted a

FIGURE 24. Laboratory Redox Curve (Slurry with Added Sugar)

ferrous-to-ferric ratio of 0.116. Figure 25, which is a plot of ferrous-to-ferric ratio vs run time for feed and glass samples, shows that the ratio for the feed was very close to this value (mean value for the feed with added sugar segment of the run was 0.106). This agreement between the laboratory prediction and actual feed redox potential is in contrast to HWVP-11. The predicted sugar concentration for the April 1985 High-Bay test (3.0 g/L) did not sufficiently reduce the feed. Again, the laboratory predicted a ferrous-to-ferric ratio of 0.116 while the actual feed ratio ranged from 0.004 to 0.04.

This is thought to be due to uncompleted chemical reactions of the waste components with the formic acid (a reductant). Waste used in the makeup of feed for the actual HBCM-11 melter test was heated to 90°C until the addition of formic acid was completed, about two hours. It is now thought that the forming reactions were not fully completed at this time and temperature. The changes in the redox response with time observed in both HBCM-11 feeds during the melter test support the thesis that ongoing chemical reactions were occurring in the feeds and resulted in the changing redox response.

FIGURE 25. Redox Response of PSCM-22 Feeds and Glasses

In contrast to the HBCM-11 melter test, waste used in PSCM-22 melter feed was heated to 90°C, the formic acid was added over a two-hour period, and the mixture was heated to 101°C and held at that temperature for an additional two hours. Laboratory tests indicated that 3.5 grams of sugar per liter of melter feed were required to be added to PSCM-22 feed to achieve the target ferrous-to-ferric ratio of 0.1. Figure 25 shows that PSCM-22 melter feed did have a stable redox response at approximately the 0.1 ferrous-to-ferric level during the first 200 hours of the run. When the sugar was removed from the feed, the feed ferrous-to-ferric ratio dropped to approximately 0.004. Figure 25 also indicates that the ferrous-to-ferric ratio of the melter output glass closely paralleled the glass produced from melter feed in the laboratory but was offset

to slightly higher values. Part of this offset can be attributed to the higher average bulk glass temperatures found during the PSCM-22 melter test (1180°C) compared to the laboratory crucible glass (1080°C). As the temperature increases in this range, the ferrous-to-ferric equilibrium in the glass is shifted toward the ferrous state. The offset between feed and glass could be reduced by increasing the temperature of the crucible test. As shown in Figure 25, the offset between the ferrous-to-ferric response of the feed and the melter glass is consistent and could be compensated for with a correction factor. A correction factor would need to be derived for each specific melter the feed was processed in to obtain a more accurate redox prediction. This would be completed during the cold start-up of HWVP.

As stated in the Run Plan, the second segment of the test was expected to generate a slightly oxidizing glass melt. During this segment an evaluation of the stability of the melter under oxidizing conditions was to be made. Laboratory tests prior to the segment showed that the feed without added sugar was slightly reducing (ferrous-to-ferric ratio of 0.002). The forming step used during reference feed preparation introduced sufficient TOC to maintain a reducing feed. Because of this, the envisioned oxidized glass was never obtained. The mean $\text{Fe}^{+2}/\text{Fe}^{+3}$ of the feed for this segment of the test was 0.002 and the glass generated never went below 0.01 (mean ratio was 0.016). Figure 25 shows the $\text{Fe}^{+2}/\text{Fe}^{+3}$ as a function of time for this feed. It appears, based on melter stability and the higher feed rates obtained during this segment of the test, that a minimal $\text{Fe}^{+2}/\text{Fe}^{+3}$ is desired. It may be decided that reductant additions will be made only as necessary to adjust for variations in waste stream composition.

Chloride contamination of the $\text{Fe}(\text{OH})_3$ used in the Auxiliary melter feed was sufficient to raise the ferrous-to-ferric ratio of this feed to 1.2 (no sugar was added to the Auxiliary feed). Because processing a melter feed with a low redox potential (high $\text{Fe}^{+2}/\text{Fe}^{+3}$) was not an objective of this test, adjustments to the feed were made in an effort to correct this situation. The hypothesis was made that the majority of the chloride in the feed was in the form of FeCl_3 . Based on this assumption, NaNO_3 was added to the feed in a

stoichiometric 1:1 molar ratio. The nitrate salt addition was made to oxidize the FeCl_3 to Fe_2O_3 and thus raise the redox potential to a more desirable value (see Equation 3).

Subsequent analysis of feed samples showed that the addition was successful. The ferrous-to-ferric ratio of the feed was lowered to 0.028 for the remainder of the run. The $\text{Fe}^{+2}/\text{Fe}^{+3}$ of the glass melt increased from 0.023 to 0.245 because of the reducing nature of the initial Auxiliary feed. Once the adjustment of the feed was made, the ratio in the glass started to decline. Because of the termination of the run, the melt redox did not return to an equilibrium state.

Based on the events of PSCM-22, desired $\text{Fe}^{+2}/\text{Fe}^{+3}$ in actual HWVP melter feeds may be obtainable using only minimal additions of oxidizing (NaNO_3 or more suitable oxidizers) or reducing (sugar or other reductant) agents. Formating the waste stream during feed preparation may be sufficient to obtain the desired ratio in the feed and variations in the waste stream may be compensated for with the addition of the appropriate agent. It is obvious that a better knowledge of the state of the actual waste is required. Until feed preparation and characterization tests are performed on actual waste, more precise feed preparation requirements cannot be fixed.

CRYSTALLINITY AND SETTLING POTENTIAL FOR SPINEL CRYSTALS

A significant amount of crystallinity was observed in the glass samples from the PSCM-22 melter test. This was expected due to the relatively high level of Cr_2O_3 in the HW-39 glass composition and was also observed in the previous HBCM-11 melter test. Three sampling methods were employed to evaluate the extent of crystal formation and to quantify the amount of crystal settling within the melter: 1) melter output glass evaluation, 2) melter core samples, and 3) evaluation of a dual chambered settling collection boat from the bottom of the melter.

Spinel Crystal Settling

A major objective of PSCM-22 was to evaluate the extent of spinel crystal settling associated with the use of HWVP melter feed, which has a high Cr concentration. Analytical (ICP) results from feed and glass samples taken at approximately 24-hour intervals during the run are shown in Table 4. From the table, it is clear that 1) the weight percent Cr_2O_3 in both the feed and the glass varied considerably from sample to sample and 2) the average weight percent Cr_2O_3 in the glass is less than that in the feed.

Evaluation of glass and feed samples from HWVP-11 showed the same trend as PSCM-22. In addition, SEM analysis of glass samples showed that the spinel crystals formed during the 5-day High-Bay run were larger than those seen in previous runs or laboratory melts. Because of the long duration of PSCM-22, it was decided that additional tests would be run to determine if the accumulation of spinel crystals on the melter floor could be quantified. Glass samples taken from an Inconel sample boat placed on the melter floor just prior to the start of PSCM-22, core samples taken at the beginning and end of the run, and day-to-day feed and glass sample analyses were to be used to determine if spinel settling was occurring to the extent that problems in processing HWVP melter feed would be encountered.

Figure 22 is a plot of weight percent Cr_2O_3 (unnormalized) vs-run time. Because the glass in the melt tank prior to PSCM-22 had a low Cr_2O_3 concentration compared to HWVP melter feed, the value of wt% Cr_2O_3 in the glass did not reach an equilibrium value until approximately 180 hours into the run. After this time the Cr_2O_3 level remained fairly constant. The average values of feed and glass Cr_2O_3 wt% from hour 180 to the end of the run (hour 427) are 1.30 and 1.18, respectively, or a 9.2% difference between feed and glass. This can be interpreted as 90.8% of the Cr_2O_3 entering the melter in the feed exiting in the glass overflow.

In an effort to evaluate the accuracy of analytical procedures, 4 feed samples from different segments of the run were sent to the laboratory for preparation and analysis. Two of the feed samples were split in half and treated as separate samples. Portions of each of the resulting 6 samples were then melted in laboratory crucibles. The feed and glass samples were then

randomized and sent to the analytical lab for ICP analysis. Based on these results, it was hoped that by eliminating the residence time in a melter any differences between feed and glass analyses for Cr_2O_3 would become apparent. Under ideal conditions the results of these analyses would show that Cr_2O_3 in sample #1 feed was the same as sample #1 glass.

The results of the analyses showed that this was not the case. None of the 6 analyses was successful in detecting all the Cr_2O_3 in the glass (feed wt% was greater than glass wt% in all cases). The extent of the difference between Cr_2O_3 level varied substantially. In the best case, the wt% in the glass was 98.4% that in the feed. In the worst case, the glass was 81.2% that in the feed. If the worst case is not included (the results of the worst case are suspect because it was one of the samples that was split into two "identical" samples prior to analyses; the results of the sister sample were similar to the remainder of the group), the difference between average feed and glass wt% Cr_2O_3 was 96.1% (average feed wt% was 1.29, and glass was 1.24).

An estimation of the amount of Cr settling that occurred during PSCM-22 is based on the following assumptions:

- total feed processed = 17,100 L
- total oxides/L feed = 0.390 kg/L
- weight percent Cr_2O_3 detected in the feed = 1.30
- percent Cr_2O_3 detectable in the glass = 96.1
- percent total Cr_2O_3 detected in the glass = 90.8
- percent Cr_2O_3 left in the melter = 5.3
- total Cr_2O_3 introduced to melter = 82.1 kg
- total Cr_2O_3 exiting the melter = 82.1 kg
- total Cr_2O_3 left in the melter = 4.6 kg

Assuming all the Cr_2O_3 left in the melter is in the form of chromite spinel crystals, 6.8 kg of crystals are formed. This is equivalent to a perfectly dense layer of thickness 0.18 cm being deposited on the melter floor over the entire experiment. Assuming the volume percent of crystals present in

melter sludge is 19% (based on samples taken from the Inconel sample boat), the height of sludge present at the end of the run would be 1.0 cm. Because air sparging, needed to clear the freeze valve prior to draining the melt cavity, disturbed the sludge layer on the melter floor, an accurate estimate of spinel crystal settling was not obtained following PSCM-22. Further work is needed to quantitatively determine Cr_2O_3 settling.

Output Glass Analysis

Output glass samples were obtained at regular intervals throughout the melter test. In addition to the ICP analysis reported earlier in this report, thin sections of output glass samples were made and examined optically and by scanning electron microscopy/energy-dispersive x-ray (SEM/EDX) to evaluate the crystalline phases present in the glass. The pre-run glass (0.06 wt% Cr_2O_3) was free of any detectable crystalline material. As the Cr_2O_3 content increased in the melter glass to a steady-state level, the amount of crystalline material observed in the output glass samples increased. During the first 200 hours of the run, the cubic spinel phase, $(\text{Ni,Fe})(\text{Fe,Cr})_2\text{O}_4$, observed in previous tests was the only phase observed. In the latter period of the run (200 hours to the end of the test) an increasing population of the hexagonal crystal phase was observed in the output glass and in the post-run melter core sample. Two major changes occurred in melter operating conditions at approximately the same time the increase in hexagonal phase was observed: 1) the redox level changed from a ferrous-ferric ratio of 0.15 to approximately 0.02, and 2) a number of temperature excursions about 1200°C began to occur in the melter. It is thought that the increase in hexagonal crystalline phase was due to the elevated temperatures within the melter and not the change in the redox state of the glass. Previous laboratory testing found no observable change in phase morphology over the 0.15 to 0.02 range of ferrous-ferric ratios. Laboratory tests have found that the cubic phase is the predominant phase at temperatures below 1200°C. At 1250°C and above, the amount of hexagonal was observed to increase and appeared to become the dominant phase. The exact temperature between 1200°C and 1250°C, above which the hexagonal phase starts to grow, has not been determined.

Melter Glass Core Analysis

Core samples were taken in an attempt to obtain a vertical profile of the melt from the bottom of the melter to the melt surface. The cores were obtained by inserting a stainless steel pipe with a remotely operated bottom closure into the melt. The pipe was slowly inserted into the melt, the bottom sealed, and then removed from the melter. Three cores were obtained at different times: directly prior to, immediately after, and 40 days after the conclusion of the melter run. Each core contained 20-25 cm of glass. The cores were sectioned into 5-cm lengths and core-drilled to obtain glass samples from each of these segments. Thin sections were obtained for the whole length of the cores by this method.

The pre-run core had no detectable crystalline phase as was expected due to the low Cr_2O_3 content.

The core taken immediately after the run contained a significant amount of crystalline material. In all but the bottom 1.8 cm of the core, the amount and types of crystalline material resembled the phases observed in the output glass samples in the latter half of the run. The bottom 1.8 cm of the core contained a settled layer of crystalline material of much greater density than that of the upper portion of the core. The thickness of this settled layer is comparable to the estimated thickness of a settled layer based on the mass balance calculations discussed earlier.

The core taken 40 days after the run contained no observed crystalline phases. After the run but prior to the elapsed 40 days, the melter was partially flushed with a glass frit with a low Cr_2O_3 content. Therefore, some dilution of the HWVP glass occurred. We also expect that settling of the crystalline material from the bulk glass occurred.

Settling Boat Analysis

A dual-chamber Inconel "boat" was placed on the bottom of the melter at the beginning of the run to quantify the amount of settling that occurred during the PSCM-22 melter run. The first chamber of the boat was covered on the eighth day of the run. At the end of the run, attempts to cover the second chamber of the boat failed due to the corrosion of the guide rod on the boat.

The boat was retrieved after the melter was drained. A significant amount of sludge was stirred up while the melter was being drained and was deposited in the open chamber of the boat, which invalidated any collection data from this chamber.

When uncovered, the glass in the first chamber of the boat had a clean surface, indicating that little, if any, of the sludge of the draining operation had contaminated this chamber. A core was drilled vertically from near the center of the first chamber and was examined optically, by SEM/EDX, and by electron microprobe wavelength dispersive analysis (microprobe/WDS). The core taken from the boat was 4.5 cm in length. The top 2 cm of the glass core was free of crystal material with only a very few agglomerates of crystals observed. The bottom 2.5 cm of the core consisted of a settled layer of cubic spinel. The Cr_2O_3 concentration from ICP analysis of the glass-only layer was 0.52 wt%, which is approximately the solubility limit of Cr_2O_3 in these types of nuclear waste glasses. The Cr_2O_3 concentration in the settled layer was 18.3 wt%. The crystals present in this layer were in a glass matrix of the nominal HW-39 composition.

The reason for the disparity between the thickness of the settled layer in the melter core samples and the settled layer observed in the boat is not known. The method of melter core sampling is obviously crude and prone to sampling error. The pipe bottom closure may have raised the pipe off the bottom of the melter during closure. It is also possible that the dissolution to the boat may have added to the amount of crystalline material within the boat. It was not possible to distinguish the chrome contribution from the Inconel boat through mass balance estimate. However, the boat was severely corroded.

Analysis of the amount of crystalline phase in the settled layer was conducted by using an image analyzer which totals the light (crystals) and dark (glass) areas of an optical micrograph. Assuming spherical particles, the volume percent of the crystal phase was estimated. This analysis estimated approximately 19 vol% spinel in this settled layer.

Microprobe/WDS analysis of the crystals found in the settled layer confirms that crystals are basically an iron-nickel-chrome spinel. The

composition of the crystals is 63 wt% Cr_2O_3 , 21 wt% FeO , and 8 wt% NiO as oxides with smaller amounts of MgO , MnO , CoO , and Al_2O_3 .

COLD CAP MODELING

A secondary objective of PSCM-22 was to generate additional data for use in the application of a predictive computer model developed by Routt and Porter.^(a) This theoretical model of heat transfer considerations in a slurry-fed ceramic melter was derived for a cylindrical joule-heated melter. The work was done in support of the SRL melter program but should be reasonably applicable to PNL systems.

The model derives material and energy balances over the entire melter cross section. It solves a large set of simultaneous equations to predict feed rates and cold-cap coverages for various operating conditions. For PSCM operation, this model predicts 22.1 L/h of feed at 50% cold-cap coverage (feed pile radius = 33 cm). Extrapolation to 80% coverage gives 35.3 L/h of slurry processed. At 100% coverage, the predicted feed rate is 44.2 L/h. Actual experience during PSCM-22 indicated that maximum feed rates (approaching 100% cold-cap coverage) for feed with and without added reductant were 39 L/h and 52 L/h, respectively.

Results obtained from PSCM-22 have been added to an existing data file which will be used to modify and improve the Routt model. This work is scheduled for FY 86 under the Nuclear Waste Treatment Program (NWTP).

LABORATORY PREDICTIONS OF MELTING BEHAVIOR

The melting rate for PSCM-22 feeds (with and without added sugar) could not be accurately determined using the laboratory melting rate test.^(b) The cold caps produced by these feeds were very thin and collapsed very quickly

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- (a) Routt, K. R., M. A. Porter. 1980. "Theoretical Predictions for Continuous Slurry Feeding of a Cylindrical Joule-Heated Melter." DPST-80-605, SRL.
 - (b) Bates, S. O. September 1985. HWVP Interim Milestone 020207B: The Correlation Between Laboratory-Scale Melting Tests and Pilot-Scale Melter Tests.

onto the glass both in the melter and in the crucible. Experience has shown that as the cold cap thins, changes in the cold-cap "mass" become more difficult to determine due to an indistinct cold-cap/glass interface. The thinner cold caps also appeared to be affected by other factors, such as the proximity of the wall of the crucible and feed off-gas evolution. Although the laboratory crucible melting test has provided a very good prediction of melting rates for most melter feeds, the test does not provide an accurate melting rate prediction for feeds that produce this thin type of cold cap. A new continuously fed laboratory-scale melting rate test has recently been developed and is currently being tested. This new laboratory melting rate test will provide steady-state melting rates and allow measurement of the actual amount of glass throughout as a function of time. This test should not have the cold-cap limitations of the crucible test and should be used to evaluate the melting rates of feeds with cold-cap behavior similar to that of the PSCM-22 feeds.

Melting characteristics such as cold-cap thickness, gas permeability, and density can have a marked effect on the processability of a given feed composition. While quantitative measurements of these cold-cap characteristics have not been made, qualitative comparisons have been made between cold caps from the melting rate test and the cold cap actually observed in the HBCM and PSCM. The thin, dense, flexible cold caps observed in the laboratory tests appeared to be identical to cold-cap samples observed in the PSCM-22 melter test.

MELTER POUR EVALUATION

COMPATIBILITY WITH HWVP CANISTER DESIGN

The objective of this investigation was to evaluate the operability of the PSCM overflow when filling canisters with small fill openings. Past experience has been with canisters having 20 cm (8") diameter fill openings. For HWVP the current reference canister has a 12.7 cm (5") diameter opening. Limited testing during PSCM-21 and 22 was planned to provide preliminary information on the compatibility of the HWVP melter with the reference canister, using the PSCM as a working model.

During PSCM-21, pour testing was made difficult by the deteriorated condition of the Inconel pour tip in the overflow section. Testing conducted during this run was to evaluate the techniques that were to be used during PSCM-22. Between PSCM-21 and 22 it was determined that repair was impossible and the pour tip was removed. The resulting pour tip was the V-notched end of the K-3 trough block.

A circular insert was positioned in the overflow stream during portions of PSCM-22 to evaluate the degree of deflection encountered during a glass pour. The insert was attached to a rod and inserted into the top of the 55-gal drums used to collect glass during certain segments of the run. Figure 26 shows one of the 55-gal drums including the insert arrangement. The hole in the center of the insert was 10.8 cm (4.25") in diameter. This corresponds to the inner diameter of the reference canister with a canister closure sleeve inserted (11.4 cm - 4.5" diameter) and some allowance for a throat protector, which will be used but has not been designed. Several 55-gal drums were filled using the insert. During testing of the insert, there were no instances where the glass stream deviated enough to contact the metal ring. The only time glass actually fell outside the simulated canister opening was when glass dripped from the edge of the trough block and from glass accumulation on the overflow section due to sampling.

Glass samples (ingots) are taken during each air lift using a graphite sample boat. The sample boat is held in the glass pour stream just under the trough block until the boat is full. The sampler is then removed from the

stream and set out to cool. Many times the removal of the sample boat causes the glass stream to be drawn along with the boat. The glass stream can then strike the vertical surface of the overflow throat (this section is located just above the bellows) and can adhere to the throat wall. During PSCM-22 the accumulation of glass in this section was enough to interfere with the falling

FIGURE 26. Simulated Canister Opening Insert

glass stream. This caused the glass stream to fall outside the insert opening. Once the primary glass pour stream started to intersect with glass accumulation in the throat additional glass started to agglomerate. Before the throat section was completely sealed off, the glass protrusion was chipped clear and normal operation resumed. Again, this problem was not due to incompatibility between the overflow and canister opening.

Throughout the run, when the insert was used while filling 55-gal drums, the glass stream showed no tendency to drift outside the simulated canister opening (nominal airlift rate was 120 kg/hr). This was true for periods of air lifting at high rates (~240 kg/hr) and during periods of continuous overflow which generates low flowrates (~15 kg/hr).

Preliminary results provided by this limited testing indicated that the drop distance of the glass stream from the pour tip to the top of a canister under the PSCM does not have a negative impact on the compatibility of the melter with small canister openings. The drop distance was approximately 150 cm (60"). However, it is reasonable to assume that shortening the drop distance would increase the confidence for successfully pouring into the small canister openings with a production melter. At this time various options are being evaluated for modifications to the PSCM overflow design. In addition to shortening the drop distance modifications are being considered that would permit full-scale HWVP canisters to be filled with the PSCM. This would require modifying the floor of the overflow section. If the recommended modifications are made to the PSCM overflow, additional testing would be conducted to evaluate the results of those changes.

EFFECTS OF OVERFLOW TEMPERATURES ON GLASS POUR STREAM

The tendency of glass to string from the overflow pour face into the canister is undesirable. Nesting, which can accompany stringing can lead to obstruction of the canister opening. During normal PSCM glass air lifts a solid glass stream up to 0.64 cm (0.25 in.) O.D. is maintained. The glass stream is quite steady and little chance exists for the development of obstructions during the main pouring event. However, once the air lift is turned off, the flow rate of glass still in the trough begins to slow. As a result, the

flow rate of glass from the face tip is not sufficient to maintain a full stream and the O.D. of the stream decreases. A broken stream of glass is encountered during the final stages of a pour. As long as the glass temperature is maintained above some critical temperature, T_c , the last glass will drip from the face tip. If, however, the glass temperature is below T_c the glass will string from the tip and a long glass filament results. This situation can lead to nesting in the area above the canister.

Tests were conducted during PSCM-22 to evaluate the range of temperatures, or more correctly, the range of glass viscosities, that allow for acceptable pour conditions. Overflow temperatures of 900°C to almost 1100°C were tested. During these tests the glass air lift was operated until a stable glass pour was obtained and glass and overflow temperatures were consistent. Trough and face heater power levels were adjusted to maintain comparable face and trough temperatures. Glass temperatures were measured using a 0.64 cm O.D. thermocouple placed in the trough approximately 5 cm from the face. Table 5 contains the temperature data for the ten pours conducted. In all cases the

TABLE 5. Temperatures at Glass Pouring

<u>Pour No.</u>	<u>Temperatures (°C)</u>		
	<u>Trough</u>	<u>Face</u>	<u>Glass</u>
A-1	1099	1050	1105
A-2	1098	1051	1105
A-3	1098	1051	1102
B-1	1056	1048	1092
B-2	1057	1049	1073
C-1	1017	1010	1068
C-2	1010	997	1068
D-1	945	948	NR ^(a)
D-2	960	947	1052
E-1	929	897	1025
E-2	923	898	1050

(a) Not Recorded.

air lifted glass was hotter than the overflow section area. After stable conditions were established the air lift was turned off and the remaining glass was allowed to drain from the trough. Estimates of drop characteristics such as drop rate, stream length before drop formation and drop size were made and videotaped. Table 6 contains a summary of the glass pour results. Glass

TABLE 6. Summary of Glass Pour Test Results

Pour No.	Est. Glass Temp (°C)	Est. Glass Viscosity (g/cm s)	Comments
A-1	1097	99	Drop formation was acceptable during all phases of pour. 0.3 to 1.0 cm diameter drop size
A-2	1096	99	
A-3	1095	100	
B-1	1058	125	Drop formation degraded from Case A. However, drops still acceptable. 0.3 to 0.6 cm diameter drop size
B-2	1057	125	
C-1	1002(a)	230	Glass string up to 15 cm long occurs before drop forms. Still judged acceptable. 0.6 to 1.0 cm diameter drop size
C-2	998(a)	230	
D-1	945(a)	500	Similar to Case C
D-2	944(a)	500	
E-1	895(a)	1000	Glass string up to 30 cm long occurs before drop forms. Tip of string is dark following drop
E-2	894(a)	1000	

(a) Estimate based on average of trough and face temperatures. All others based on glass thermocouple in trough.

viscosities were determined from HWVP-11 data shown in Figure 27 based on either the average of the trough and face temperatures during drop formation or the glass thermocouple reading.

Testing covered an order of magnitude change in glass viscosity. Results indicated that pouring was acceptable at glass viscosities less than or equal to 500 poise. At 1000 poise, the stringing length reached approximately 30 cm. before drop formation occurred and cooling of the glass string tip was evident by its darkened appearance. Therefore, to ensure acceptable glass pour termination, operation of the overflow section temperature in the 1050°C to 1100°C range should minimize the chances for glass nesting in the canister flange area.

FIGURE 27. Viscosities of HWVP-11 and HW-39 Glasses

HALF-SCALE CANISTER FILL FOR SANDIA NATIONAL LABORATORY

Ten canisters were filled with glass for the Transportation Technology Center (TTC) located at Sandia National Laboratory, Albuquerque, New Mexico. These canisters are half-scale versions of the DWPF canisters which will be shipped to the WIPP site. Six of the canisters were shipped to TTC for impact testing of the half-scale shipping cask, which will take place early in FY 86.

Filling these canisters provided a special challenge, as the neck is only 6.35 cm (2.5") I.D.. The glass pour stream has been observed to waver more than that distance, so special provisions were made to direct the molten glass into the canister without causing a buildup of glass on the outside of the canister. The arrangement used is shown in Figure 28. A stainless steel funnel

FIGURE 28. Funnel Arrangement Used During SNL Canister Fills

was attached below the bellows, and a graphite funnel was connected below it with a flexible sleeve. The stainless funnel was coated with a molybdenum mold release compound before it was installed. The flexible sleeve was necessary to allow centering the canister under the pour stream, and to prevent placing a load on the canister from the melter discharge adapter. The canisters were placed on a load cell to determine initial, intermediated, and final gross weights.

The final canister weight requirement was higher than could be achieved with the HWVP glass, so 60 kg of lead shot was added to each canister at specified gross weights. The position of the lead additions to the canister were chosen so that the center of gravity would not be appreciably different than for a glass-only canister. The final canister gross weight required was 312 ± 2 kg. All canisters filled met this criteria.

The canisters were filled by continuously overflowing the melter until the canister was about 230 kg gross weight and all the lead shot had been added. The balance of the 312 kg was airlifted into the canister at approximately double the rate to allow for canister change out.

The funnel arrangement worked without any noticeable glass buildup until the final canister. During the last canister pour, it is suspected that a chunk of glass broke free from a build-up in the throat section of the overflow section and formed an obstruction in the stainless steel funnel. Glass from the pour stream then adhered to the obstruction and formed a plug. The canister was removed from underneath the melter along with the connecting section. A 55-gal drum replaced the canister while the stainless funnel was cleared of glass. The canister was put back under the melter and the final glass addition was made using the airlift.

OFF-GAS CHARACTERIZATION

Off-gas and effluent characterization studies have been established as part of the melter development programs being conducted at PNL. The objective of these studies are to establish the off-gas properties of liquid-fed joule-heated melters as a function of melter operational parameters and to determine the operational performance of key off-gas processing components.

MELTER OFF-GAS SYSTEM OPERATIONAL DESCRIPTION

The off-gas system components studied during PSCM-21 testing were an ejector venturi scrubber (EVS) a hydrosonic scrubber and a high efficiency mist eliminator (HEME). Operational off-gas performance characteristics of EVS and HEME were obtained throughout all phases of PSCM-22 testing as were melter effluent emission characteristics. The performance of the hydrosonic scrubber was evaluated during the last two days of PSCM-22 testing. During operation the hydrosonic was valved into the off-gas system between the EVS and HEME otherwise it was completely valved out (see Figure 12). Although the EVS was operated invariantly throughout PSCM-22, the performance of the HEME was evaluated under two operating modes: with and without a co-current spray wash. The HEME spray wash was initiated on July 24, 1985 at 2:00 p.m. and was terminated on July 29, 1985 at 7:40 a.m. However, subsequent use of the hydrosonic scrubber with its associated water injection may have maintained wet HEME operating conditions throughout the remainder of the test.

EFFLUENT SAMPLING

One of the major tasks involved in melter off-gas support studies is that of melter emission characterization. The simulated feed used in the present melter experiment contained stable elemental substitutes for all major volatile and semivolatile isotopes, which are present in Hanford waste, and are of radiological or chemical concern. The trace elements added to the melter feed and their resultant concentrations are summarized in Table 2, which details the overall simulated Hanford slurry feed composition. The only major changes that occurred in feed composition during PSCM-22 were as follows:

July 23: Sugar eliminated from the feed

July 26: Ru added to one feed batch

August 1: New feed stock prepared and processed

The sampling network developed in support of melter emission characterization studies were designed to determine the composition of melter-generated gaseous and particulate effluents. Effluent sampling was simultaneously conducted before and after all active off-gas scrubbing devices.

Continuous real time analyses of melter generated gases was limited during the present test to O_2 , CO, CO_2 , NO and NO_2 effluent species. In addition, quasi-continuous monitoring of off-gas H_2 concentrations were also conducted using a gas chromatograph and mass spectrometer. Most of the gaseous data generated has been archived for potential future use. Only mass spectrometric will be reported in this report.

MELTER GENERATED GASES

LFCM processing of feeds containing the formate anion are capable of producing significant concentrations of H_2 and CO. Because of its low flammability limit ~4.8%, hydrogen generation can produce safety related processing problems. Previous PNL melter tests involving SRL formatted feeds^(a) have shown that the nominal relative production rates of CO_2 , H_2 and CO to be 1, 0.5 and 0.1 respectively. However, application of lid heat effectively eliminates H_2 and CO through plenum oxidation.

The relative production ratios of CO_2 , H_2 and CO observed in the present test are 1, 0.1 and <0.1. The presence of highly oxidizing NO_x gases in the melter exhaust may be responsible for reducing the net off-gas yield of hydrogen produced by HWVP reference feed. Whatever the cause, CO_2 normalized H_2 off-gas concentrations were lower during the processing of HWVP feed than they were on the average during SRL feed processing.

(a) Goles, R. W., and G. J. Seigny. 1983. Off-Gas Characteristics of Defense Waste Vittrification Using Liquid-Fed Joule Heated Ceramic Melters. PNL-4819, Pacific Northwest Laboratory, Richland, Washington.

NATURE OF MELTER FEED COMPONENT LOSSES

In order to characterize the pathways and magnitudes of melter feed component losses, a differential sampling system was employed, which was composed of an aerosol filter, a condenser, and a series arrangement of three gas-scrubbing units. The manner in which any given element is distributed across the five discrete sampling fractions of this device is indicative of the physical state or states assumed by the effluent. Data generated with this differential sampling system during the present melter test is summarized in terms of DFs. DF values are decontamination factors which are ratios of the rate in which feed components enter a device to the rate at which they are evolved from it. Particulate DFs are partial DFs relating to only a single loss mechanism: aerosol emission. The data collected by this differential sampler during PSCM-22 testing clearly showed that melter gas-phase losses to the off-gas system are only significant for B, C, N, S, and the halogens, which all readily form volatile acidic gases. Table 7 summarizes the % of the total mass of each of these off-gas effluents which was exhausted in the gaseous state from the various off-gas system components listed. This volatility data demonstrates that the presence of feed component sugar has little effect upon melter volatility losses. Indeed, the formate component of the feed alone was fully capable of maintaining the reducing conditions within the melter as is clearly illustrated by the absence of Ru volatility.

The data in Table 7 also unequivocally illustrates the propensity of the halogen feed components to be evolved from the melter in the gaseous state. It is also evident from this data, that gaseous halogens have the ability to penetrate all off-gas processing equipment studied. This fact is especially important for iodine because of the long-lived ^{129}I isotope. The degree to which these volatile isotopes penetrate the scrubbers listed will be dealt with in the next section. This DF data can be used in conjunction with Table 7 values to assess the radiological implications of LFCM radioactive gas phase losses.

The PSCM-22 melter volatility data, apart from sulfur, is quite comparable to similar data obtained during a previous High Bay melter test: HWVP-11. All of the effluent emission data obtained during this previous test is summarized in Appendix B. Like PSCM-22, the HWVP-11 volatility data was not strongly

TABLE 7. % Volatile Effluent Losses

<u>Element</u>	<u>Date/Time</u>	<u>% Volatile Losses</u>			
		<u>Melter</u>	<u>EVS</u>	<u>HEME</u>	<u>Hydrosonic</u>
C	7/19-8/1	<100	<100	<100	<100
B	7/19 1100	89	13	21	--
	7/20 1000	88	8.3	19	--
	7/20 2200	87	9.8	62	--
	7/21 1000	89	14	72	--
	7/21 2100	86	11	76	--
	7/25 1000	91	40	70	--
	7/26 1000	88	3.7	80	--
	7/27 1000	88	12	99	--
	7/31 1100	89	45	97	48
	7/31 2300	91	32	94	69
	8/1 1100	96	69	<100	99
C1	7/19 1100	60	<100	26	--
	7/20 1000	59	<100	23	--
	7/20 2200	49	<100	<100	--
	7/21 1000	58	<100	45	--
	7/21 2100	44	<100	50	--
	7/25 1000	73	<100	<100	--
	7/26 1000	89	<100	<100	--
	7/27 1000	75	<100	<100	--
	7/31 1100	62	<100	85	<100
	7/31 2300	53	<100	<100	<100
	8/1 1100	<100	<100	91	<100
F	7/19 1100	84	5.6	48	--
	7/20 1000	84	7.8	57	--
	7/20 2200	79	8.4	72	--
	7/21 1000	85	9.5	<100	--
	7/21 2100	78	9.2	50	--
	7/25 1000	91	32	77	--
	7/26 1000	89	23	82	--
	7/27 1000	88	20	99	--
	7/31 1100	87	10	23	45
	7/31 2300	91	17	87	78
	8/1 1100	82	14	97	77
I	7/19-8/1	<100	--	--	--
N	7/19-8/11	<100	<100	<100	<100

TABLE 7 (contd). % Volatile Effluent Losses

Element	Date/Time	% Volatile Losses			
		Melter	EVS	HEME	Hydrosonic
S	7/19 1100	81	79	74	--
	7/20 1000	84	80	73	--
	7/20 2200	85	81	97	--
	7/21 1000	87	84	97	--
	7/21 2100	85	84	97	--
	7/25 1000	89	<100	<100	--
	7/26 1000	83	<100	<100	--
	7/27 1000	49	<100	<100	--
	7/31 1100	58	51	<100	50
	7/31 2300	80	<100	<100	3
	8/1 1100	95	96	99	99

influenced by feed changes occurring on April 21, 1985 which resulted when chemical glass formers were substituted for 2/3 of frit used during the previous two days of processing. The lower % sulfur volatility recorded during HWVP-11 is probably due to the lower reductant loading in HWVP-11 feed which caused sulfur to accumulate within the melter as an insoluble $\text{SO}_4^{=}$ phase.

Melter Aerosol Emission Performance

Since effluent gas phase losses have been shown to be exclusively limited to B, C, N, S and Halogens, most melter off-gas system losses are, necessarily, associated with aerosol emission. Consequently, the off-gas performance (DF) of the liquid-fed melter can be conveniently measured in terms of gross off-gas aerosol loading. Melter aerosol DFs are especially useful in assessing processing stability since the extent of gross feed entrainment is directly related to non-steady-state processing conditions. Furthermore, particulate DFs establish a common basis from which performance comparisons can be made. Melter aerosol emission and off-gas performance data are summarized in Table 8. This data is also graphically illustrated in Appendix A.

The melter aerosol DFs throughout PSCM-22 were quite consistent and reproducible. If one arbitrarily excluded data obtained on July 25 and August 1, the % standard deviations associated with the nine remaining melter DFs in Table 8 is 7%, otherwise an 18% value is obtained. The average aerosol melter DF throughout PSCM-22 was 320 which is significantly, 50%, lower than values

TABLE 8. Melter Aerosol Emission and Off-Gas Performance

<u>Date-Time</u>	<u>Aerosol DF</u>					
	<u>Melter</u>	<u>EVS</u>	<u>Packed Tower</u>	<u>Hydrosonic</u>	<u>HEME</u>	<u>Total</u>
7/19 11:00	290	3.3	<1.2	---	--	1.1E3
7/20 10:30	340	3.4	<1.3	---	--	1.5E3
7/20 22:00	320	3.	---	---	15	1.8E4
7/21 10:00	370	3.5	---	---	15	1.9E4
7/21 21:00	300	4.4	---	---	16	2.1E4
7/25 09:30	450	3.3	---	---	16	2.4E4
7/26 10:00	320	3.8	---	---	22	2.7E4
7/27 09:30	310	3.9	---	---	12	1.4E4
7/31 10:30	330	4.4	---	4.1	--	>5.9E3
7/31 23:00	310	3.3	---	5.5	--	>5.6E3
8/01 11:30	210*	1.8	---	15*	---	>5.7E3
7/19 - 8/1	330+14%	3.7+11%	<1.2	4.8	16+20%	5.3E4

*Not included in average

obtained from previous PSCM tests involving SRL formatted feed streams.^(a) Although there was quite a variability in melter aerosol DFs observed during the recent HWVP-11, the average melter performance for that test (see Appendix B) was also higher than that recorded during PSCM-22. However, recent PSCM tests involving WVNS or related feed formulations have exhibited comparable melter behavior.^(b) The significance of the above comparisons are somewhat questionable in that there can be quite a variation in average melter behavior between tests even when consistent performance is achieved throughout each experiment. The only sure means by which average melter emission behavior can be determined is by measuring melter performance for a series of repetitive independent tests.

The consistency of melter DFs listed in Table 8 also indicate that the sugar feed component, which was eliminated on July 23, has no significant influence upon melter aerosol emission. Similarly, melter performance was not significantly affected by the substitution of chemical glass former for 2/3 of

- (a) Goles, R. W., and G. J. Sevigny. 1983. Off-Gas Characteristics of Defense Waste Vitrification Using Liquid-Fed Joule Heated Ceramic Melters. PNL-4819, Pacific Northwest Laboratory, Richland, Washington.
- (b) PNL LFCM Run Summaries: PSCM-19, PSCM-20 and PSCM-21.

the frit in the HWVP-11 feed stream as is shown in Table B-2 of Appendix B. The variability in HWVP-11 data is, however, large enough that small affects could conceivably be masked, but, certainly, no large impact upon melter performance was observed by this substitution.

EVS Aerosol Off-Gas Performance

The aerosol performance of the EVS was also very consistent throughout PSCM-22. This is exactly what would be expected in light of the low variability of the melter source term. The low EVS performance recorded on August 1 was not averaged in with the rest of the data in Table 8 since the melter was restarted on that day with a freshly prepared feed batch and steady state conditions probably did not exist. In fact most all of the data collected on August 1, 1985 was not typical of the previous 20 days of melter testing and so will not be used in deriving average performance parameters.

The average EVS aerosol DF of 3.7 is somewhat lower than has been achieved previously. However, unlike the previous tests, PSCM-22 did employ a film cooler. Since PSCM-22 used a noncondensable gas (air) as the film cooler sweep fluid, a large process effluent dilution resulted upstream of the EVS which may have reduced contacting and resultant scrubbing efficiency. Steam collapse of the melter effluent exhaust during EVS quenching was also significantly reduced or eliminated by film cooler operations which may have contributed to the overall loss of EVS performance.

Packed Tower Aerosol Off-Gas Performance

During PSCM-22 testing a packed tower utilized for NO_x scrubbing was interposed between the EVS and high efficiency scrubbing device; i.e., the Hydrosonic and/or HEME. Since this type of scrubbing column would not be expected to have a significant impact upon effluent aerosols after EVS processing, sampling after this device was not routinely conducted. However, aerosol samples were inadvertently collected after this device on July 19 and 20 with, unfortunately, an incorrectly positioned sampling nozzle (e.g., sampling probe pointing downstream). The results obtained as shown in Table 8 confirm the expectation of low aerosol removal efficiency for this device. In all discussions that follow, the aerosol DFs of this packed tower are assumed to be 1.

HEME Off-Gas Aerosol Performance

During PSCM-22 the off-gas performance of the HEME was evaluated with and without a co-current waterspray and with and without pretreatment by a hydrosonic scrubber. Since hydrosonic pretreatment could affect HEME performance by altering the overall size distribution of effluent aerosols, HEME DFs for the last two days of testing will be discussed separately.

HEME aerosol off-gas performance when not used in conjunction with the hydrosonic scrubber was relatively consistent and reproducible throughout PSCM-22 as is shown in Table 8. The average DF measured throughout this period of testing was 16, which is a factor of three improvement over previous PNL test results.^(a) However, this DF value is still at least a factor of three below engineering design expectations; i.e., $DF = 50$. Past operating experience suggests that solution breaching of the filter followed by subsequent retrainment of soluble effluents is responsible for HEME performance deterioration.

This filter penetration mechanism is supported by the chemical composition of the matter penetrating the HEME element as will be discussed later. The fact that the low flow rate HEME water spray, which was initiated on July 24, had no observable impact upon HEME DF, is not inconsistent with this penetration mechanism if it is recognized that water mist carryover from the pack tower was probably much greater than that introduced by the HEME spray nozzle. On the other hand, only dry operating conditions have been successful in producing acceptable HEME performance. All of the above suggests that wetting a HEME causes performance deterioration. The rate of mist carryover or the degree of HEME wetting may also determine the degree to which performance is reduced.

The use of a hydrosonic scrubber upstream of the HEME during the last two days of testing reduced the size of the off-gas samples down stream of the HEME to the point that gravimetric or gross overall DFs could not be reliably determined. However, individual elemental HEME DFs to be discussed in the next section were not found to be significantly affected by hydrosonic pretreatment of

(a) PNL LFCM Run Summaries: PSCM-19, PSCM-20 and PSCM-21.

the off-gas stream. Consequently, overall HEME DFs could not have been influenced to any great extent by hydrosonic scrubber operation.

Hydrosonic Aerosol Off-Gas Performance

The DF performance of the hydrosonic scrubber used during the last two days of PSCM-22 testing is also detailed in Table 5. The DF data obtained from samples collected on July 31 are consistent with past PNL evaluations^(a) and with limited SRL experience.^(b) The increase in hydrosonic performance on August 1 illustrates the ability of this device to respond to higher aerosol loadings resulting from decreased performance of the melter and EVS. The combined overall DF across the melter EVS and hydrosonic was essentially constant (5.9E3, 5.6E3 and 5.6E3) during the period of hydrosonic testing even though significant melter and EVS performance changes did occur.

DETAILED FEED COMPONENT DFs

Individual feed component DFs established for the melter, the EVS, HEME and the hydrosonic scrubber during PSCM-22 are summarized in Tables 9 through 12, respectively. These data are also graphically displayed in Appendix A.

Melter Performance

On the average, melter feed component DFs are quite reasonable and comparable to values obtained from the previous HWVP-11 melter test which also used an HWVP feed formulation (see Appendix B). Cs is the only real exception to this statement. The Cs data associated with the previous HWVP-11 test exhibited an average which was a factor of ~10 greater than the corresponding result obtained from PSCM-22. The reason for this difference is the increased quantity of formate anion used in the preparation of PSCM-22 feed. The very low but consistent Cs DFs recorded during PSCM-22 are strikingly similar to previous PSCM data obtained during the processing of formated SRL feeds.^(c)

(a) PNL LFCM Run Summaries: PSCM-19, PSCM-20 and PSCM-21.

(b) Savannah River Laboratory. 1983. Waste Management Technical Progress Report. DP-82-125-4, Aiken, South Carolina.

(c) Gotes, R. W., and G. J. Sevigny. 1983. Off-Gas Characteristics of Defense Waste Vitrification Using Liquid-Fed Joule Heated Ceramic Melters. PNL-4819, Pacific Northwest Laboratory, Richland, Washington.

TABLE 9. PSCM-22 MELTER DF'S

ID	AVG	7/19/85 1102 HR	7/20/85 1025 HR	7/20/85 2210 HR	7/21/85 0955 HR	7/21/85 2115 HR	7/25/85 0936 HR	7/26/85 1007 HR	7/27/85 0937 HR	7/31/85 1038 HR	7/31/85 2305 HR	8/1/85 1124 HR
AL	750	520	670	600	670	760	1200	740	770	740	790	1500
B	63	54	67	69	69	74	72	60	59	50	58	89
BA	190	130	140	140	150	140	370	220	230	180	220	520
CA	730	760	900	860	920	820	180	720	690	670	800	1900
CE	450	370	420	470	380	430	700	410	410	380	470	—
CL	1.3	1.1	1.2	1.5	1.4	1.8	1.2	1.2	1.1	1.3	1	1
CR	180	180	220	210	230	220	210	140	140	140	150	580
CS	14	12	13	13	16	15	14	13	13	15	11	4.6
CU	250	170	220	200	220	220	390	270	280	290	210	43
F	3.2	2.7	3.1	3.3	3.3	3.5	3.1	2.8	3	3.3	3.6	3.4
FE	260	210	250	240	260	240	390	240	250	240	280	650
I	1.3	1.8	1	1.2	1.2	1.4	< 8.2	< 11	< 14	1.6	1.1	1
LA	610	570	690	650	730	630	790	490	520	530	530	1000
LI	500	430	500	510	560	540	680	470	460	460	430	280
MS	630	580	750	700	780	690	420	540	510	590	700	2000
MN	300	250	310	78	340	300	460	300	320	310	350	640
MO	57	45	54	58	58	64	63	52	54	56	63	73
N	< 94	< 5	< 7.7	< 5.7	< 7.3	< 6.3	< 210	< 350	< 350	< 2.6	< 3.6	< 8.6
NA	260	210	250	260	280	280	360	260	260	270	210	81
NB	290	240	290	260	280	280	430	270	280	270	320	1100
NI	2100	1900	1800	1400	2200	1200	2300	2600	2300	2500	2300	2100
RU	42	—	—	—	—	—	—	42	—	—	—	—
S	1.8	1.8	1.9	1.8	1.7	2.2	1	1	2.7	2.6	1.3	1
SI	780	690	850	790	900	760	1100	650	650	650	750	1900
SR	230	190	220	230	240	220	330	230	220	220	250	540
TI	250	120	160	140	150	140	490	290	290	300	410	2300
ZM	160	110	140	240	170	99	190	180	200	81	160	76
ZR	420	330	480	420	490	390	600	370	370	370	420	2100
TTL	180	150	180	180	190	180	230	200	210	160	160	90

TABLE 10. PSCM-22 VENTURI DF'S

ID	AVG	7/19/85 1104 HR	7/20/85 1027 HR	7/20/85 2208 HR	7/21/85 0954 HR	7/21/85 2116 HR	7/25/85 0938 HR	7/26/85 1008 HR	7/27/85 0935 HR	7/31/85 1040 HR	7/31/85 2307 HR	8/1/85 1125 HR
AL	32	6	5.4	6.1	5.9	4.4	63	96	36	56	40	37
B	11	11	11	13	12	12	9.7	13	11	9.3	11	32
BA	290	240	180	1200	230	140	120	620	27	68	57	48
CA	160	380	90	100	750	110	1.5	47	47	69	32	50
CL	6.2	4.6	5.3	4.2	5	3.5	7.8	8.1	11	8.8	4	4.4
CR	14	22	22	23	27	29	3.8	3.4	3.2	3.5	2.8	15
CS	1.4	1.3	1.5	1.5	1.3	1.4	1.3	1.3	1.4	1.6	1.6	1.5
CU	7.2	7.5	5.9	6.6	6.6	5.5	7.2	9.3	7.7	13	2.4	1.4
F	14	9.3	11	13	13	12	16	14	15	17	14	6.4
FE	92	170	140	110	110	58	29	120	64	70	57	16
LA	440	470	460	500	490	570	250	330	---	---	---	72
LI	2.6	2.3	2.4	2.2	2.4	2.4	2.5	3.1	3.1	3.4	2.5	1.5
MG	94	73	63	68	58	1	3	17	27	400	230	37
MN	220	230	320	730	330	22	19	180	120	120	160	12
MO	1.6	1.8	1.6	1.5	1.6	1.4	1.5	1.6	1.4	1.7	1.5	1.8
NA	2.1	2	2.1	2	2.1	2	2	2.4	2.3	2.6	1.9	1.3
ND	190	240	210	210	240	210	110	140	---	---	---	52
NI	3.7	4.2	4.9	5.6	2.4	2.1	1.4	6.1	3.6	5.4	1	1
RU	1.4	---	---	---	---	---	---	1.2	1.4	1.4	1.4	---
S	4.3	1.3	1.5	1.5	1.6	1.5	13	8.7	3.1	2	9	1.6
SI	59	6.4	26	29	26	20	100	170	140	46	32	9.3
SR	260	340	380	310	390	150	9.5	94	110	490	290	56
TI	970	1100	---	1400	1200	1900	1000	180	---	84	---	72
ZH	19	5.3	50	15	47	1.1	2.6	4.1	---	---	27	3.9
ZR	670	410	540	780	1500	930	98	140	1400	570	340	20
TTL	5	4	4.3	4.5	4.3	4.8	5.5	6.1	5.7	5.1	5.4	2.9

TABLE 11. PSCM-22 HEME DF'S

ID	AVG	7/20/85 2204 HR	7/21/85 0953 HR	7/21/85 2117 HR	7/25/85 0932 HR	7/26/85 1010 HR	7/27/85 0934 HR	7/31/85 1044 HR	7/31/85 2307 HR	8/1/85 1127 HR
AL	10	—	—	—	—	—	10	—	—	—
B	7.5	6	7.5	7.3	19	13	1	1.5	5.4	—
BA	8.7	1.9	15	—	1.4	7.3	16	4.5	15	—
CA	3	—	—	—	—	—	3	—	—	—
CL	14	11	10	11	15	20	24	1.6	21	3.4
CR	16	5.5	18	18	19	20	3.2	18	22	1
CS	19	9.5	19	18	17	23	20	18	25	53
CU	16	16	16	16	17	25	5.7	13	19	34
F	6.1	8.6	9	7.1	6.6	7.6	1	6.7	3.2	1.5
FE	44	12	8.6	18	280	1.9	1	35	1	46
LI	19	19	19	19	16	22	18	16	21	42
MG	1	—	—	—	—	—	1	—	—	—
MN	36	1	5.6	260	13	1	1	3.4	6.4	6.1
MO	19	16	18	18	16	22	25	17	21	46
N	3.5	—	—	—	—	—	—	3.1	3.9	1.9
NA	17	14	14	14	16	21	15	20	20	27
NI	64	180	—	—	16	—	1	55	—	—
RU	32	—	—	—	—	31	56	16	25	—
S	18	2.1	1.9	1.8	16	20	9.5	23	72	2.4
SR	6.2	14	—	—	1.2	—	1	8.9	—	—
TI	41	8.3	52	—	12	150	—	15	6.5	—
ZM	3.9	7.9	5.8	3.1	1.1	1.4	1	5.1	5.8	18
ZR	25	1	1	37	57	95	1	4	2.8	1
TTL	5.6	2.8	2.6	2.5	7.1	18	2.4	3.5	5.9	2

TABLE 12. • PSCM-22 HYDRO-SONIC DF'S

<u>ID</u>	<u>AVG</u>	<u>7/31/85</u> <u>1042 HR</u>	<u>7/31/85</u> <u>2308 HR</u>	<u>8/1/85</u> <u>1126 HR</u>
AL	4.5	4.5	---	---
B	6.2	8.2	4.1	1.8
BA	7.3	9.7	4.9	1.3
CA	5.9	5.9	---	---
CL	4.9	3.3	6.6	14
CR	5.3	4.3	6.4	20
CS	4.3	3.8	4.8	15
CU	7.3	4.7	9.9	13
F	3.5	4.5	2.6	5.8
FE	6.1	6.5	5.7	5.4
LI	5	4.2	5.9	17
MG	1.2	1.2	---	---
MN	5.5	5.6	5.3	30
MO	4.8	4.5	5.1	16
NA	5	4	5.9	17
NI	6.1	8.5	3.6	82
RU	4.8	4.5	5.1	---
S	2.3	4	1	1.4
SI	1.1	1.1	---	---
SR	11	8.1	13	42
TI	18	18	---	38
ZN	1.4	---	1.4	10
ZR	8.8	5.4	12	87
TTL	1.7	1.5	1.9	3

Formated feeds are exceptionally effective in promoting the Cs volatilization/condensation process in the melter plenum and as a result have been responsible for the lowest recorded melter Cs DFs measured at PNL. PSCM-22 feed, clearly is no exception to this rule. It should be noted that the sugar component of the feed which was eliminated on July 23 had no apparent influence upon Cs or, for that matter, overall melter losses. The enhanced PSCM-22 Cs loss rate must only be related to the presence of the formate in the melter feed.

The melter emission rates for the halogens followed during PSCM-22 (Cl, F, and I) were without exception quite high. Moreover, the loss mechanism for these group VII elements were predominantly associated with gas phase emissions as has been discussed in the previous section. Fluorine exhibited the highest average DF of 3.2 followed by chlorine and iodine, which both exhibited average DFs of 1.3. The less than values listed for iodine DFs on July 25 through July 27 resulted from analytical difficulties encountered in collecting and detecting iodine in nitric acid scrubbing solutions which were utilized for only those three days. It should also be noted that unlike fluorine and chlorine, chemically combined iodine in aerosol matter was never detected in off-gas samples; consequently, the total iodine DFs located in Table 6 may be lower than indicated if undetected aerosol iodine was present.

The average PSCM-22 melter DF for fluorine, 3.2, was lower than the corresponding value, 5.0, obtained during HB85-1 (see Appendix B). However, PSCM-22 data is much more consistent and the analytical feed data is much more reliable. Consequently, if all things were equal, the PSCM-22 fluorine data should be weighed much more strongly than the results obtained from the HWVP-11 test. But since the feed streams were not equivalent, it is only prudent to accept the current data as being most representative of fluorine melter emission behavior.

Like the halogens, sulfur exhibited a very low average melter DF-1.8. The higher formic acid feed loading must have been responsible for promoted higher volatile loss rates of sulfur than occurred in HWVP-11 (see Table B-2, Appendix B). With such a low DF, very little, if any, sulfur was accumulating within the melter as a separate insoluble sulfate phase.

The total melter DF associated with Ru was determined only on July 26, 1985 since only a single batch of feed was spiked with the element. Its relatively low DF, 42, is indicative of an initial volatilization mechanism; however, its subsequent conversion to an aerosol is quite prompt since all detected Ru melter off-gas system emissions were confined to the aerosol sample fraction.

Sr melter emission behavior was quite similar during both PSCM-22 and HWVP-11 tests. In each test, Sr emission characteristics tracked the behavior of other nonvolatile feed components such as Fe and Nd. Although the PSCM-22 DFs for these elements were slightly lower than the corresponding data measured during HWVP-11 testing, the results are without exception more reproducible. Moreover, the differences in the average Sr behavior between the two tests may be totally ascribable to the differences in feed stream composition.

Finally, the less than values listed for nitrogen in Table 9 are due to the inability of the off-gas sampling system to scrub out all nitrogen oxide gases. The higher than normal values listed on July 25 - July 27 are due to the use of nitric acid scrubbing solutions which masked nitrogen scrub solution information for those days.

EVS Performance

The performance of the EVS during PSCM-22 as illustrated in Table 10 and Appendix A, was quite predictable. This type of scrubber is a low efficiency device that is only designed to deal with large diameter aerosols. As a result, EVS elemental DFs should be direct measures of the mass mean diameters of the aerosols responsible for individual feed component losses. For instance, feed components which are volatilized and quickly converted by reaction and/or condensation to predominantly sub-micron aerosols would be expected to exhibit low EVS DFs. And this is exactly what is observed for the classic semivolatiles: Cs, Li, Na, and Ru. On the other hand, nonvolatiles whose escape mechanism is based solely on gross feed entrainment would be expected to exhibit much higher DFs. Such is indeed the case as is clearly shown by the DF results obtained for Ba, Ca, Fe, La, Nd, Sr, etc. The low overall aerosol DF of the EVS, 3.7, discussed earlier, is a reflection of the overall mass median

diameter of the aerosols evolved during PSCM-22. It is clear that the gross feed entrainment process was not dominating aerosol generating mechanisms during the present test.

Since the EVS is not designed to be an effluent gas scrubber, gaseous effluent DFs would also be expected to be low. This is also found to be true as the EVS data for B, Cl, F and S clearly show. The absence of EVS iodine DF data in Table 10 is a reflection of special analytical difficulties associated with the detection of this element. Although iodine performance data beyond the melter is lacking, there is no reason to believe that its off-gas behavior is any less penetrating than the other halogens which are more easily detected and quantified.

HEME Performance

The performance of the HEME during PSCM-22 is numerically summarized in Table 11 and is graphically displayed in Appendix A. Since most aerosols penetrating the EVS are sub-micron, all aerosolized effluents should exhibit essentially the same HEME DFs. This is indeed what is found in Table 11 data. Effluents with volatile components on the other hand would be expected to exhibit lower than average DFs. This expectation is also realized as is illustrated by the B, Cl, F, and S DF data. It should be noted that the variability of DF data for certain elements is a reflection of analytical uncertainties associated with results at or near detector limits. In addition, blank corrections at low concentrations are also major contributors to the data uncertainties and spread. Inconsistencies in elemental DFs data should be used as a basis for questioning HEME results. Examples of questionable data are illustrated by the results obtained for Fe, Ni, Sr, Ti and Zr.

The Cs HEME DFs recorded during PSCM-22 were a factor of ~3 higher than has been observed during previous PSCM testing. These values are fairly consistent and are apparently unaffected by the use of the upstream hydrosonic scrubber during the last two days of PSCM-22 operation. These DFs however, remain a factor of 3 below HEME design expectations. The reason for deficient performance may involve solution breaching of the filter element as discussed in the previous section. The absence or the large scatter associated with insoluble HEME effluent DF data in Table 11 makes testing this hypothesis

difficult at best. However, all previously obtained HEME operational data suggest dry operation is required in order to achieve expected design performance; i.e., DF = 50.

HYDROSONIC PERFORMANCE

The hydrosonic elemental DFs observed during PSCM-22 (see Table 12) are, with few exceptions, lower on the average than values recorded with the HEME. Moreover, the DFs observed are not strongly element dependent which suggest most aerosols penetrating the EVS are sub-micron. This has indeed been shown to be true during previous LFCM tests.^(a)

Overall, the hydrosonic DFs recorded during the present test are in good agreement with limited past experience both at PNL^(a) and SRL.^(b) However, in truth, very little experimental data characterizing single-stage hydrosonic performance in LFCM applications exist. Most characterization measurements have been performed utilizing dual cascaded hydrosonic units. In this configuration, Cs DFs of 50 have been routinely achieved.^(b) Single stage Cs DFs observed during the present test are an order of magnitude lower than this value as are most other average elemental DFs. If one optimistically assumed that the combined DF associated with cascaded hydrosonics is the square of single stage performance, PSCM-22 would have achieved a Cs DF of ~20 from this dual configuration which is significantly less than has been recorded at SRL.^(b)

As has been previously stated, the restart of the melter on August 1, 1985 significantly affected melter emission characteristics. Because both melter and EVS DFs were uncharacteristically low on August 1 the hydrosonic DFs measured on that day are not really comparable with previously obtained data and so were not included in the average included in Table 12. However, the data in Table 12 clearly demonstrates the ability of the hydrosonic scrubber to respond to process upsets which act to reduce EVS scrubbing efficiency.

(a) PNL LFCM Run Summaries: PSCM-19, PSCM-20 and PSCM-21.

(b) Savannah River Laboratory. 1983. Waste Management Technical Progress Report. DP-82-125-4, Aiken, South Carolina.

APPENDIX A
PSCM- 22 GRAPHICAL DF DATA

Melter and off-gas system performance characteristics are presented graphically in this appendix. Axis identifications and scale definitions are given in the title of each graph. T_0 is the origin of the time axis whereas dT represents the time interval between axis markers. All DF values plotted are normalized to the maximum DF value in the set. As a result, the ordinate (DF) scale represents 0-100% of this maximum value which is listed in the title of each graph.

MLTR NORMALIZED TOT DF TIME DISTRIBUTION
T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=232.36)

EVS NORMALIZED TOT DF TIME DISTRIBUTION
T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=6.08)

HEME NORMALIZED TOT DF TIME DISTRIBUTION
T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=17.94)

MLTR NORMALIZED AL DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1473.13)

EVS NORMALIZED AL DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=95.60)

HEME NORMALIZED AL DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=10.13)

MLTR NORMALIZED B DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=89.45)

EVS NORMALIZED B DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=32.02)

HEME NORMALIZED B DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=19.19)

MLTR NORMALIZED BA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=520.63)

EVS NORMALIZED BA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1198.07)

HEME NORMALIZED BA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=15.80)

MLTR NORMALIZED CA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1863.02)

EVS NORMALIZED CA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=751.22)

HEME NORMALIZED CA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=2.98)

MLTR NORMALIZED CE DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=699.46)

EVS NORMALIZED CE DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1)

HEME NORMALIZED CE DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1)

MLTR NORMALIZED CL DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1.81)

EVS NORMALIZED CL DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=10.79)

HEME NORMALIZED CL DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=23.53)

MLTR NORMALIZED CR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=581.48)

EVS NORMALIZED CR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=29.03)

HEME NORMALIZED CR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=22.17)

MLTR NORMALIZED CS DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=16.09)

EVS NORMALIZED CS DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1.59)

HEME NORMALIZED CS DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=53.07)

MLTR NORMALIZED CU DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=388.64)

EVS NORMALIZED CU DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=12.96)

HEME NORMALIZED CU DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=34.02)

MLTR NORMALIZED F DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=3.56)

EVS NORMALIZED F DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=17.25)

HEME NORMALIZED F DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=9.03)

MLTR NORMALIZED FE DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=652.78)

EVS NORMALIZED FE DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=172.96)

HEME NORMALIZED FE DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=276.91)

MLTR NORMALIZED I DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=13.99)

EVS NORMALIZED I DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1)

HEME NORMALIZED I DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1)

MLTR NORMALIZED LA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1045.52)

EVS NORMALIZED LA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=567.76)

HEME NORMALIZED LA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1)

MLTR NORMALIZED LI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=678.95)

EVS NORMALIZED LI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=3.44)

HEME NORMALIZED LI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=42.18)

MLTR NORMALIZED MG DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=2001.82)

EVS NORMALIZED MG DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=398.11)

HEME NORMALIZED MG DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=.97)

MLTR NORMALIZED MN DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=637.74)

EVS NORMALIZED MN DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=731.19)

HEME NORMALIZED MN DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=261.50)

MLTR NORMALIZED MO DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=73.19)

EVS NORMALIZED MO DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1.81)

HEME NORMALIZED MO DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=46.13)

MLTR NORMALIZED N DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=345.69)

EVS NORMALIZED N DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1)

HEME NORMALIZED N DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=3.93)

MLTR NORMALIZED NA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=355.59)

EVS NORMALIZED NA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=2.64)

HEME NORMALIZED NA DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=26.97)

MLTR NORMALIZED ND DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1135.01)

EVS NORMALIZED ND DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=239.93)

HEME NORMALIZED ND DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1)

MLTR NORMALIZED NI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=2578.61)

EVS NORMALIZED NI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=6.06)

HEME NORMALIZED NI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=183.49)

MLTR NORMALIZED RU DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=42.46)

EVS NORMALIZED RU DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1.43)

HEME NORMALIZED RU DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=55.61)

MLTR NORMALIZED S DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=2.68)

EVS NORMALIZED S DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=12.56)

HEME NORMALIZED S DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=72.13)

MLTR NORMALIZED SI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1893.82)

EVS NORMALIZED SI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=166.22)

HEME NORMALIZED SI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1)

MLTR NORMALIZED SR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=535.06)

EVS NORMALIZED SR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=486.58)

HEME NORMALIZED SR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=13.77)

MLTR NORMALIZED TI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=2271.73)

EVS NORMALIZED TI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1856.16)

HEME NORMALIZED TI DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=152.76)

MLTR NORMALIZED ZN DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=242.69)

EVS NORMALIZED ZN DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=49.93)

HEME NORMALIZED ZN DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=17.56)

MLTR NORMALIZED ZR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=2051.01)

EVS NORMALIZED ZR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=1469.83)

HEME NORMALIZED ZR DF TIME DISTRIBUTION

T->(T₀=7/19/85 0000; dT=8H): DF↑(FS=95.47)

APPENDIX B

TABLE B-1. HB85-1 % Gas Phase Effluent Losses

<u>Element</u>	<u>% Gas Phase Melter Loss</u>						
	<u>4/18</u> <u>0900</u>	<u>4/18</u> <u>1600</u>	<u>4/19</u> <u>0900</u>	<u>4/19</u> <u>1600</u>	<u>4/21</u> <u>0900</u>	<u>4/21</u> <u>2200</u>	<u>4/22</u> <u>1000</u>
B	62	69	78	81	79	71	71
F	79	72	92	90	94	84	<100
S	<100	<100	19	22	47	41	34

TABLE B-2. HB85-1 MELTER DF'S

ID	AVG	4/18/85 0906 HR	4/18/85 1624 HR	4/19/85 0914 HR	4/19/85 1534 HR	4/21/85 0837 HR	4/21/85 2154 HR	4/22/85 0954 HR
AL	2200	590	1400	1800	3000	4600	2300	2000
B	130	120	140	160	170	140	110	110
BA	290	81	120	230	280	640	320	330
CA	650	290	530	860	1100	890	490	420
CE	530	140	260	770	690	1000	490	320
CR	260	61	100	200	250	600	330	310
CS	100	78	210	50	66	110	92	120
CU	290	77	120	220	320	650	320	330
F	5	6.9	7.4	3.9	5.3	3.6	4.2	3.5
FE	310	77	130	260	310	700	330	350
LA	730	190	320	630	800	1600	820	760
LI	690	320	510	760	1700	720	380	390
MG	700	270	490	930	1200	1000	510	500
MN	340	110	170	220	430	820	390	260
MO	100	41	61	83	110	170	130	130
NA	350	160	240	370	470	570	320	350
ND	300	77	130	280	330	630	330	290
NI	8100	5200	7400	3800	18000	---	6600	---
S	67	54	65	86	99	56	53	55
SI	850	360	620	970	1300	1300	690	680
SR	330	87	130	270	310	750	370	390
TI	740	270	430	1100	680	1000	1100	570
ZR	620	210	320	500	760	1200	720	640
TTL	430	220	340	470	590	580	400	390