**FY-97 Operations of the Pilot Scale Glass Melter
to Vitrify Simulated ICPP High Activity
Sodium Bearing Waste**

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ABSTRACT

A 3.5 liter refractory-lined joule-heated glass melter was built to test the applicability of electric melting to vitrify simulated high activity waste (HAW). The HAW streams result from dissolution and separation of Idaho Chemical Processing Plant (ICPP) calcines and/or radioactive liquid waste. Pilot scale melter operations will establish selection criteria needed to evaluate the application of joule heating to immobilize ICPP high activity waste streams. The melter was fabricated with K-3 refractory walls and Inconel 690 electrodes. It is designed to be continuously operated at 1150°C with a maximum glass output rate of 10 lbs/hr. The first set of tests were completed using surrogate HAW-sodium bearing waste (SBW). The melter operated for 57 hours and was shut down due to excessive melt temperatures resulting in low glass viscosity (<30 Poise). Due to the high melt temperature and low viscosity the molten glass breached the melt chamber. The melter has been dismantled and examined to identify required process improvement areas and successes of the first melter run. The melter has been redesigned and is currently being fabricated for the second run, which is scheduled to begin in December 1997.

SUMMARY

A 3.5 liter joule-heated refractory-lined melter was built to test the applicability of electric melting ICPP HAW. The pilot scale melter was constructed from K-3 fused cast refractory, Inconel 690 electrodes, and designed to operate at 1150°C.¹ Materials of construction and operating parameters were based on ICPP crucible study test results,² crucible scale melter corrosion tests,³ and United States Department of Energy (US DOE) contractor collaborations.⁴ The melter tests were designed to identify potential glass formulation HAW-SBW mixtures that vitrify with low corrosion rates, acceptable viscosity vs. temperature profiles, and perform well when subjected to product consistency (PC) leach test. The glass melter operated for 57 hours before requiring shut down due to excessive melt temperatures (>1375°C), thus causing the glass viscosity to become less than 30 Poise, allowing the glass to breach the melt chamber.

The melter was initially started using external resistance heaters until the glass became electrically conductive,⁵ approximately 800°C, to begin joule heating. An Accurate screw feeder delivered surrogate feed at a specified rate to the melt chamber (0 - 20 lbs/hr). Once the chamber was filled with molten glass, the glass overflowed through a "tea-pot" spout to exit the melter into an annealing furnace. Observations concluded that during joule heating the glass entered the pour spout, but as the glass began to gravity flow through the pour spout, glass temperature decreased and viscosity increased to cause the glass to solidify in the pour spout. This phenomenon caused the molten glass that remained at the target temperature (1150°C) and viscosity (30 - 100 Poise) to flow toward, contact, and dissolve the pour spout heater, which was operating at 1200°C. The molten glass that was still in the pour spout then solidified and eliminated the opportunity for the glass to exit the melter.

Visual inspection of the melt chamber indicated that the mortar between the refractory cracked and corroded during melter operations. High melt temperatures, approximately 1375°C resulting from loss of temperature control, and low glass viscosity allowed the molten glass to drain from the melter chamber through these open seams in the refractory. The glass was captured between the melter stainless steel shell and refractory located directly underneath the melt chamber. Visual and microscopic inspections determined that the K-3 refractory and Inconel 690 had a very low corrosion rate, below the expected corrosion rates compared to previous test results.^{3,6,7} Design changes include increasing the wall thickness to 8 inches, two layers of 4-in. refractory, as compared to one layer of 4-in. refractory. The two refractory layers are now staggered to avoid direct paths to the outside walls. These changes allow the glass to decrease in temperature as it travels away from the melter chamber and solidify/seal the cracks in the refractory. The external start-up heaters were also replaced by silicon carbide heaters installed in the melter lid. The pour spout heater configuration has also been redesigned to ensure that the molten glass maintains temperature and exits the pour spout into the annealing furnace.

Excessive melt temperatures during this run were due to thermocouple failures. Infrared thermocouples were installed on the outside of the melter shell and positioned to measure refractory surface temperature, 4 in. from the melt chamber. The temperature readings were adjusted using heat transfer calculations from the melt chamber to the monitored surface. Three problems with the infrared thermocouples were identified; (1) infrared thermocouples are very difficult to calibrate because the blackbody coefficient of K-3 refractory significantly changes between 700 - 1150°C. (2) Theoretical heat transfer calculations are difficult to establish, particularly in identifying all representative heat transfer losses of the entire melter assembly. (3) Infrared thermocouples require precision fabrication to ensure the monitors measure the target surface temperature. Monitoring temperature of the glass melter has been discussed in detail with Westinghouse Savannah River Corporation (WSRC) and Pacific Northwest National Laboratory (PNNL) vitrification development personnel, thus resulting in a consensus to

incorporate in-melt Type R thermocouples protected by Inconel 690 thermowells. These thermocouples and thermowells are located in the melter lid and designed for replacement, if necessary, during melter operations.

Melter test results also identified many components that performed successfully. These include the Bailey Distributed Control System, Accurate Feeder, and melter materials of construction. Inconel 690 and K-3 refractory performed with little or no corrosion, but the Inconel 690 electrodes did melt due to the high melt temperatures. The "tea-pot" spout design initially performed as designed, the glass entered the pour spout but did not exit because the heater was dissolved by the molten glass. Control parameters such as monitoring the current density across the Inconel 690 electrodes to a maximum of 4 amps/in.² has been verified by corrosion results of the electrodes.⁸ Current density will continue to be a primary control feature for all future melter tests. Other improvements include, (1) controlling heat-up and cool down rates to 10 degrees Celsius per hour to reduce refractory cracking from thermal cycling; (2) incorporating a sealed off-gas system, larger off-gas lines, and a variable speed controller for the blower; (3) installing the start-up and pour heaters inside the melt chamber to reduce outside wall temperature; and (4) adding a layer of refractory so there are now no direct seams from the chamber to the outside walls. One improvement that was not incorporated into melter design, due to significant cost, is precision cutting the K-3 refractory. Precision cutting would reduce and/or eliminate the need for mortar between the refractory bricks.

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ACRONYMS

BDAT	Best Demonstrated Available Technology
DCS	Distributed Control System
EDS	Energy Dispersive Spectroscopy
EPA	Environmental Protection Agency
HAW	high activity waste
HEPA	high-efficiency particulate air
HLLW	High Level Liquid Waste
HLW	High Level Waste
ICP	inductively coupled plasma
ICPP	Idaho Chemical Processing Plant
INEEL	Idaho National Engineering and Environmental Laboratory
LMITCO	Lockheed Martin Idaho Technologies Company
MOG	melter off-gas
PC	product consistency
PNNL	Pacific Northwest National Laboratory
RCRA	Resource and Conservation Recovery Act
SBW	sodium bearing waste
SEM	scanning electron microscopy
SNF	spent nuclear fuel
US DOE	United States Department of Energy
WMA	Waste Management Authority
WSRC	Westinghouse Savannah River Corporation

FY-97 Operations of the Pilot Scale Glass Melter to Vitrify Simulated ICPP High Activity Sodium Bearing Waste

INTRODUCTION

Between 1954 and 1997 the Idaho Chemical Processing Plant (ICPP) has converted High Level Liquid Waste (HLLW), generated from spent nuclear fuel (SNF) dissolution and reprocessing, to a granular solid (calcine). This calcine is considered a mixed hazardous waste under current Resource and Conservation Recovery Act (RCRA) regulations. The ICPP is currently storing 1.8 million gallons of radioactive liquid wastes and over 3,800 cubic meters of calcine in stainless steel bins. Additional calcine is being generated as the tank farm liquid waste is calcined and additional waste is generated from continued plant operations. For final disposal, the calcine and radioactive liquid waste must be processed into an acceptable immobilized form prior to placement in a federal repository. The primary objectives of the High Level Waste (HLW) Immobilization Technology Development program are to: (1) develop qualified leach resistant waste forms; and (2) establish immobilization processes for the high activity waste (HAW) streams.⁹ The HAW streams result from the dissolution and separation of ICPP calcines and radioactive liquid waste. The Environmental Protection Agency (EPA) has identified vitrification, the process of converting wastes into a glassy substance, as the Best Demonstrated Available Technology (BDAT) for immobilization of wastes generated during the reprocessing of nuclear fuel.¹⁰

Operation of a pilot scale joule-heated glass melter with simulated high activity waste is being performed to evaluate immobilization processes. The glass melter was designed to operate at 1150°C to produce durable waste glass. Based on laboratory test results and collaborations with United States Department of Energy (US DOE) contractors, the melter system has been designed and installed as part of a integrated pilot plant. This report describes the melter system, test results, and gives conclusions and recommendations for future testing. The appendix includes the Bailey distributed control system operating screens that the operator uses to control and monitor melter operations.

The pilot scale glass melter was designed and built to test and evaluate integrated technologies for each of the following processes: (1) feeding simulated high activity waste (dry feed) and glass formers, (2) glass melting, (3) melter off-gas treatment, (4) pouring glass from the melter, and (5) materials of construction evaluation. Operating the integrated pilot plant provides Lockheed Martin Idaho Technologies Company (LMITCO) personnel with direct operating experience and process knowledge necessary to evaluate and select technologies to support the Batt Settlement Agreement milestone of submitting a RCRA Part B permit application in 2012, and the Site Treatment Plan operational date of September 30, 2019.

PILOT PLANT LAYOUT

The pilot scale glass melter is located in the ICPP-637 Low Bay laboratory on the Idaho National Engineering and Environmental Laboratory (INEEL) site. Figure 1 illustrates the overall layout and configuration of the glass melter system. The system includes a refractory-lined joule-heated melter, melter off-gas system, screw feeder, glass annealing furnace, and Bailey control system. The melter includes a 6"W x 6"L x 8"H melt chamber with an overall external dimension of 20"W x 24"L x 20"H. The melter is then surrounded by stainless steel to protect personnel from thermal and electrical hazards measuring 36"W x 36"L x 24"H. The melter system is located inside a stainless steel module that includes a single high-efficiency particulate air (HEPA) filter, external to the melter off-gas system, and two large doors for easy access. The glass melter is located 49 in. from the floor on 1-in. stainless steel plate which is supported by 4" x 4" x 0.375" square tubing. Below the melter is a modified Blue M 1200°C furnace that is raised and lowered with a scissors lift for capturing the glass that exits the melter. The screw feeder is located 28 in. above the top of the melter shell, which is accessed using a three-step platform. The off-gas system includes a sintered metal filter and packed bed scrubber, two HEPA filters, blower, and stack. The melter control station, located outside the stainless steel module, includes a personal computer with Bailey Control software, layout is given in Figure 1.

Figure 1. Pilot plant layout.

PROCESS AND EQUIPMENT DESCRIPTION

Simulated HAW oxides and glass formers are mixed from reagents and manually loaded into the Accurate screw feeder. The feeder augers the material to be vitrified to the melter at a selected feed rate between 0 - 20 lbs/hr. The feed enters the melter chamber and is heated to molten glass. Once the melter chamber is filled, the glass exits through the pour spout ("tea-pot" spout design) into a catch pot in the annealing furnace. For start-up, the external heaters heat the melter chamber until the feed material becomes electrically conductive, at which time joule heating is initiated to increase and/or maintain molten glass temperature. The pour spout and drain line are also lined with resistance clam shell heaters for additional heat. All resistance heaters are rated for 1200°C operation. Crucible melter test results indicate that the simulated material, shown in Table 1, becomes electrically conductive at 800°C. The same feed composition in Table 1 was also used in the first pilot scale melter test.

Joule heating is provided by a Robicon transformer that allows a range of 0 - 208 V and 0 - 80 amps of power than can be applied to the Inconel 690 electrodes. A maximum of 4 amps/in.² is the design limit of the electrodes based on laboratory crucible scale melter operations and information from design manuals.¹¹ The molten glass can exit the glass melter either through the "tea-pot" spout or pour spout and enter a stainless steel pan which is located in the Blue M annealing furnace. The annealing furnace is programmable and operated between 800 - 1200°C.

The off-gas emissions are monitored and controlled by the Bailey Distributed Control System (DCS) during all melter operations. A flowrate range of 6 - 12 liters per minute (lpm) is maintained to ensure the emissions are removed from the melter chamber. The off-gas exits the melter and enters a sintered metal filter, packed bed scrubber, and two HEPA filters prior to being released from the stack. Figure 2 illustrates the melter system during continuous operations.

Solid Feed System

The solid feed system consists of a Accurate Loss-in-Weight screw feeder. Simulated high activity sodium bearing waste and glass formers are prepared from reagents in the laboratory and mixed using a Turbula mixer. The mixed feed is then manually loaded into the screw feeder hopper. The Accurate Loss-in-Weight screw feeder is operated by inputting a desired feed rate in lbs/hr. The controller sends a signal to the screw feeder motor to increase and/or decrease the feeder screw speed (rpm), thus varying the feed rate. The Accurate Controller is controlled by the Bailey DCS. The DCS allows melter operators to turn ON/OFF the feeder and input desired feed rates. This data is collected and printed by the DCS for operating records.

The feed material exits the screw feeder and enters into a sealed mass flow hopper. The mass flow hopper is designed to ensure no bridging of the feed material occurs during melter operations. A lid was placed on the hopper to prevent particulate emissions from entering the pilot plant module. Melter operators can visually inspect the molten glass or cold cap through the discharge port of the hopper.

The Accurate Controller, which is located on the outside of the module near the DCS computer, displayed messages and alarms during melter operations, such as "Current Feed Rate is 5 lbs/hr." These displays include: feeder refill alarm, current feed rate, average feed rate, amount of feed in hopper, and mode of operation. This feed system will also be utilized for slurry feeding the melter,¹² when such options are investigated in the future.

Table 1. Simulated HAW-SBW and glass compositions.

Glass Components	100 Gram Batch	Waste Components	Waste Oxide wt%	Glass Formulation wt%	Glass Formulation mole %
SiO ₂	42.96	SiO ₂	0.40	46.32	61.26
Al ₂ O ₃	0.05	Al ₂ O ₃	0.17	0.05	0.04
B ₂ O ₃	13.01	B ₂ O ₃	0.10	14.03	16.01
		BaO	0.00	0.00	0.00
CaCO ₃	0.87	CaO	1.74	0.52	0.74
CdO	0.00	CdO	0.00	0.00	0.00
		Cr ₂ O ₃	0.02	0.01	0.00
CaF ₂	0.00	CaF ₂	0.00	0.00	0.00
Fe ₂ O ₃	1.76	Fe ₂ O ₃	1.64	1.89	0.94
		FeO			
K ₂ CO ₃	8.11	K ₂ O	19.87	5.96	5.03
MnO ₂	0.00	MnO ₂	0.01	0.00	0.00
Na ₂ CO ₃	10.35	Na ₂ O	6.25	6.53	8.36
Na ₄ P ₂ O ₇	18.12	P ₂ O ₅	65.14	19.54	5.84
ZrO ₂	0.23	ZrO ₂	0.83	0.25	0.16
AmO ₂	0.00	CmO ₂	0.00	0.00	0.00
NpO ₂	0.00	NpO ₂	0.01	0.00	0.00
PuO ₂	0.05	PuO ₂	0.19	0.06	0.02
CeO ₂ for UO ₂	2.15	CeO ₂ for UO ₂	7.52	2.26	0.66
Cs ₂ CO ₃	0.00	Cs ₂ O	0.00	0.00	0.00
SrO	0.00	SrO	0.01	0.00	0.00
TcO	0.00	TcO	0.00	0.00	0.00
Li ₂ CO ₃	0.00	Li ₂ O	0.00	0.00	0.00
PbO	2.37	PbO	8.53	2.56	0.91
AMP	0.00	AMP	0.00	0.00	0.00
MoO ₃	0.00	MoO ₃	0.00	0.00	0.00
MgO	0.00	MgO	0.00	0.00	0.00
TiO ₂	0.00	TiO ₂	0.00	0.00	0.00
CeO ₂	0.00	CeO ₂	0.00	0.00	0.00
Nd ₂ O ₃	0.00	Nd ₂ O ₃	0.00	0.00	0.00
ZnO	0.00	ZnO	0.00	0.00	0.00
NiO	0.01	NiO	0.05	0.02	0.02
Sm ₂ O ₃	0.00	Sm ₂ O ₃	0.00	0.00	0.00
Total	99.99	Total	100.0	100.0	100.00

Figure 2. Pilot scale melter system—continuous operation.

Insulation

The glass melter was constructed from K-3 refractory and Kaowool 12C insulation board. The glass melter insulation was installed to reduce heat losses and to protect exterior surfaces from high temperatures. The K-3 refractory is surrounded by at least 3 in. of insulation, see Figure 3. The insulation and refractory is enclosed by a stainless steel shell. The stainless steel shell provides a shield that protects melter operators from contacting electrical and temperature hazards. The melter was designed to be used for numerous tests planned in fiscal years FY-97 through FY-00.

Electrodes

The joule electrodes (see Figure 4) are constructed from Inconel 690.¹³ Each electrode measures 6" wide x 4 1/2" high x 3/8" thick. Other alloys tested in the laboratory for suitability as melter materials with INEEL wastes include Inconel 601, 617, and 625, Nitronic 50, and Hastalloy C-4.¹ Based on these test results and collaboration with other US DOE contractors Inconel 690 was selected as the best suited material for melter electrodes.⁴ Crucible scale melter test results determined that the maximum current density across the electrodes is 4 amps/in.². Crucible dip test results with Inconel 690 and surrogate SBW-HAW determined that as the viscosity of the glass decreased, corrosion rates of the electrodes increase. An operating pouring viscosity envelope of 30 - 100 Poise at 1150°C was established for glass melter operations.

Figure 3. Pilot scale melter insulation.

Figure 4. Pilot scale melter Inconel 690 electrodes.

Melter Pour Spout

The melter pour spout was fabricated from fused cast K-3 refractory as shown in Figure 5. The pour spout was designed to direct the molten glass from the melter chamber through a 4 ½ in. riser and into the pour spout. The molten glass exits the pour spout through Hexalloy tubing. The Hexalloy tubing guides the molten glass to the annealing furnace and glass catch pot. The Hexalloy tubing is externally heated by two 1200°C clam shell resistance heaters.

Melter Lid

The melter lid was constructed from K-3 refractory, Kaowool 12C insulation board, and stainless steel. The lid assembly includes feed and off-gas ports and two infrared thermocouples. The melter lid was assembled using ½-in. all-thread bolts that were countersunk in the plenum side of the lid and protected by K-3 refractory inserts to eliminate corrosion. The melter lid assembly inserts into melter chamber and rests on the stainless steel protective shielding that surrounds the melter assembly.

Start-up and Pour Heaters

The start-up and pour heaters were installed to heat the melter feed until conductive to initiate joule heating and maintain temperature in the pour/drain line areas. The heaters ensure that the molten

Figure 5. Pilot scale melter pour spout.

glass temperature is 1150°C, thus allowing the viscosity to remain between 50 - 100 Poise. The Thermcraft heaters were operated either ON/OFF and are rated for 1200°C operation. No method of determining whether or not the heaters are working, such as monitoring current, was installed.

Bottom Drain

The bottom drain in the laboratory scale glass melter was installed to drain the molten glass from the melter chamber. The drain was fabricated from Inconel 690, Hexalloy, and heated by Thermcraft clam-shell resistance heaters. The molten glass enters the drain by gravity feed and thickens as the temperature decreases (viscosity greater than 100 Poise) during continuous operations. When the operator drains the melter, the clam-shell heater on the pour spout is turned off, and the annealing furnace is repositioned from the pour spout to the drain line. The drain spout heaters are turned ON to increase temperature to 1200°C. Once the glass is heated to 1150°C, the viscosity significantly decreases allowing the glass to pour from drain spout.

Bailey, Distributed Control System

The Bailey DCS was selected to monitor and control the pilot scale melter system for two reasons; (1) Availability; the DCS system is currently installed in ICPP-637 to operate calcination pilot plants and available at the glass melter pilot plant module. (2) Cost; the cost of purchasing a control system capable of providing on-line monitoring and control would be much more than the allocated funds to perform melter fabrication, assembly, and testing. The DCS features a large library of over 200 versatile function codes resident in active modules. These algorithms can be used in sequences or nested to the on-board function blocks to deliver control strategies for given processes. The DCS was configured to monitor

and control nearly all melter operations. In addition to controlling the pilot scale melter, the DCS also notifies operators of process upsets, unsafe operations, and emergencies by alarming and identifying the "out-of-tolerance" instrument(s).

Melter operating control screens, shown in Appendix A, were installed on the DCS. These screens are utilized by the melter operators to monitor and control melter operations. Upper and lower control envelopes were established for each control module. Several control modules were integrated so the DCS could automatically adjust melter system parameters to ensure the control envelopes were not exceeded. Print-outs at designated time intervals during the melter test were also configured to obtain complete and accurate records of melter operations.

Off-Gas Equipment

The melter off-gas system includes a blower, sintered metal filter, packed-bed scrubber, and two HEPA filters, shown in Figure 6. The off-gas blower is a DuroFlow positive displacement blower capable of operating from 150 to 1000 CFM. The DuroFlow blower is operated using a 15 HP electric motor capable of being controlled with a variable frequency drive. The variable frequency drive was not purchased for this first test, therefore off-gas flow rate was controlled by three pneumatic by-pass valves. The off-gas flowrate is monitored by a Sierra Flowmeter. The flowmeter and pneumatic by-pass valves were integrated and controlled by the DCS to operate the off-gas system at a specified flowrate and pressure differential. The target off-gas flowrate and pressure differential are determined for each feed type by utilizing thermal equilibrium calculations (using HSC for Windows) to determine emissions as a function of feed rates. The sintered metal filter was installed to collect particulate from the off-gas stream prior to entering the packed bed scrubber and HEPA filters. The particulate collected in the sintered metal filter could easily be sampled during melter operations.

The packed bed scrubber consists of a 4.5-in. diameter by 54-in. long stainless steel vessel divided into three sections. The top section consists of a demister packed with stainless steel wool that is supported by the distributor plate assembly. The middle section (25 in. in length) is filled with 1/4-in. stainless steel raschig rings which are supported by a perforated support plate. The bottom section of the scrubber includes 3/8-in. polyethylene raschig rings supported by a second distributor plate. Caustic scrub solution exits the bottom of the scrubber and is collected in a carboy. The scrub solution is recycled from the carboy back to the top section of the scrubber. The caustic scrub solution's pH and color is monitored to determine when clean solution is required. The used scrub solution is sent to analytical for analysis and disposed per the registered Waste Management Authority (WMA).

The off-gas stream HEPA filters were installed on the roof of ICPP-637 near the Duroflow blower and Dioctyl Phthalate (DOP) tested prior to melter operations. The filters were installed to remove any particulate that may have passed the sintered metal filter and packed bed scrubber.

PILOT PLANT TEST RESULTS

Melter Start-up

For start-up, the melter chamber was charged with 15.12 lbs of surrogate feed and heated with the resistance start-up heaters until the glass became conductive. Calculations determined that once the 15 lbs of start-up material became a molten glass, the chamber would be approximately half full. Melter test data shown on Table 2 indicates that it took 34 hours to heat the melter from 23 to 800°C, thus averaging 22.85°C/hr. During start-up it was noted that a cold cap was formed at 500°C and again at 800°C. The cold cap was manually broken to increase the volume of molten glass, therefore when joule heating was initiated, more power could be applied without exceeding the 4 amps/in.² current density limit. The cold cap was formed due to the 6 lpm off-gas flowrate, which cooled the top portion of the start-up material. Additional insulation was also added to the melter during initial heat-up, to minimize heat losses from the melter assembly. Joule heating initiated when the melter reached 800°C. The start-up heaters remained ON to provide supplement heat until the electrodes were completely submerged in molten glass. Table 3 shows a snapshot of melter operations, just after joule heating was initiated, on 4-8-97 at 11:50 - 13:30.

Melter Feed System

Once the start-up surrogate feed reached 1050°C, dry surrogate feed (the composition is shown in Table 1), was fed to the melter. The feed was augured from a loss-in-weight feeder to the melter chamber per a operator specified feed rate. The target temperature for continuous operations was 1150°C, but once again increasing the volume of conductive glass in the chamber allowed the operators to increase power across the joule electrodes. During feed operations the power to the joule electrodes and feeder was temporarily shut off to measure the glass level and clear suspended feed from the feed line. At 1:00 A.M. on 4-7-97, the glass chamber was full of molten glass, therefore additional feeding would result in the glass to begin exiting the riser into the pour spout. Per visual inspection of the molten glass, the glass viscosity looked to be less than 100 Poise, and at this time the infrared thermocouple readings were considered erroneous. Therefore, feeding the glass melter continued until an additional 7.93 lbs of dry surrogate feed was fed to the chamber. At 4:39 A.M., 4-9-97, power significantly dropped (see Table 2), alarming the operators to shut off power to the electrodes and feeder. The glass level in the melt chamber was determined to be less than 1 in., thus melter power was turned off.

Melter Off-Gas

During standard operating testing of the off-gas system, it was determined that when the off-gas flowrate was greater than 30 lpm, gas lifting of the scrub solution would occur. This problem was temporarily solved by restricting the flow of solution and the off-gas vacuum to less than 20 lpm. A catch trap was also installed downstream of the scrubber to protect the flow meter from water droplets.

During the melter start-up, it was determined that the off-gas scrub solution would not be turned on and the melter off-gas (MOG) flow would be set at 5 lpm. These settings would be changed when the glass melted and prior to adding dry feed. The gas flow volume was increased to 12 lpm in preparation for the dry feed. At sometime during the next 30 minutes, the off-gas line plugged either because of the feed addition or the shifting of the material already deposited in the line entrance because of the

Table 2. Pilot plant test results.

[illegible]

increased flow. The flow control valve opened all the way allowing approximately 2.5 psi to pull the scrub solution (water) from the carboy up into the scrubber. The pump also supplied some of the liquid. Since the off-gas line plugged near the melter, the entire line was under the 2.5 psi vacuum, thus causing the scrub solution to pull toward the melter, filling the sintered metal filter vessel and off-gas line approximately twelve inches above the filter vessel. At this point on the melter had no off-gas suction. This event occurred less than 30 minutes into feed addition. The flow indication was a result of the scrub solution return line becoming the supply vent for the scrubber and the system downstream. The result was the return line in the carboy was no longer submerged in the scrub solution because the scrub solution was sucked into the off-gas line.'

Off-gas samples were analyzed from several points in the system: (1) filter solution, (2) off-gas line solution, (3) scrubber solution, (4) filter solids, and (5) off-gas line unwetted dry solids. The summary of these results are shown in Table 4. The solutions were analyzed by inductively coupled plasma (ICP), emission spectroscopy, and atomic absorption spectroscopy. Specific particles were selected and analyzed scanning electron microscopy (SEM). Two of these samples are shown in Tables 4 and 5 for comparison to the dry feed that was added to the melter and solids deposited on the off-gas line as the major background matrix in the SEM micrographs. Boron is not detectable by the SEM but can be assumed to be there from comparison with the solution samples. Scanning all the recovered solids from the solution and estimating the amount in the unwetted line, no more than 10 grams of condensable material was transferred to the off-gas.

Table 4. Element analysis summary of MOG solid and solution samples.

Element	Filter Solution (micro-g/mL)	Line Solution (micro-g/mL)	Scrubber Solution (micro-g/mL)	Line Solids wt%	Filter Solids wt%	Glass as Added wt%	SEM P-2 wt%
Na	271	57.5	8.68	14.06	5.757	12.42	5.41
K	487	110	14.2	26.45	6.800	5.28	0.843
B	272	58.49	7	*	*	4.66	*
Ca	7.56	28.78	32.32		4.910	0.40	
Pb	10.02	0	0	tr	tr	2.55	
Fe	1.12	0.122	0	tr	2.680	1.41	0.0817
P	16.52	3.58	0	0.93	3.837	2.44	0.411
Cr	23.36	4.41	0.0414	1.9	6.470	0.00	
Mo	0.0102	0.046	0.00748	tr	0.270	0.00	0.026
Al				0.35	2.643	0.03	0.204
Cl				0.47			
Si				0.44	15.017	23.18	3.56
Ce				tr	tr	1.24	0.092
Zr						0.19	
F					tr		
O+						46.39	
* Boron was not detectable by the SEM.							
tr Trace of element detected.							
Blank Not detected in solid or not analyzed for in solutions.							
+ Oxygen is by difference.							
SEM P- Are specific particles collected from the unwetted MOG line.							

Table 5. Mole ratios of elements to sodium in the MOG solid and solution samples.

Element	Filter Solution	Line Solution	Scrubber Solution	Line Solids	Filter Solids	Glass as Added	SEM P-2	SEM P-4
Na	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
K	1.05980	1.12821	0.96479	1.10944	1.18124	0.25070	0.843	0.612
B	2.13551	2.16429	1.71585	*	*	0.79889	*	*
Ca	0.01601	0.28723	2.13674		0.85292	0.01838		
Pb	0.00410			tr	tr	0.02284		
Fe	0.00170	0.00087		tr	0.46555	0.04680	0.0817	0.095
P	0.04523	0.04619		0.04908	0.66647	0.14568	0.411	0.692
Cr	0.00038	0.00034	0.00002	0.05977	1.12391			tr
Mo	0.00001	0.00019	0.00021	tr	0.04690		0.026	
Al				0.02121	0.45918	0.00111	0.204	0.079
Cl				0.02169				tr
Si				0.02571	2.60857	1.52786	3.56	0.539
Ce				tr	tr	0.01643	0.092	
Zr						0.00390		
F					tr			
O+						5.3710		

* Boron was not detectable by the SEM.

tr Trace of element detected.

Blank Not detected in solid or not analyzed for in solutions.

+ Oxygen is by difference.

SEM P- Are specific particles collected from the unwetted MOG line.

Chemical analyses of the solution revealed the presence of nearly all of the glass components. Most of the dissolved and dry solids can be attributed to sodium and potassium meta-borates. Enrichment of the boron over the glass composition was expected because of its greater volatility. Aluminum and silica particles were found in both the off-gas samples and in the solids recovered from the sintered metal filter. These elements could have escaped the glass matrix as aerosols. The silicate components should not be attributed to entrained feed because the off-gas line was plugged early in feed addition. These non-borate components of the starting glass appear to be from aerosols formed by foam bubbles breaking the surface during post melt heating. SEM pictures show the presence of small spherical particles that represent condensing aerosols. These particles contain non-borate components of the glass while the glass matrix feed material is primarily Na, Si, and K.

Table 5 compares the mole ratios of the detected elements to the detected sodium for each of the six samples and the added feed material. It can be noted that the potassium to sodium ratio in most of the samples is near 1.0 while the ratio in the feed was 0.251. The enrichment in potassium is not explainable and unexpected, but may be due to greater volatility in the melter since operating temperatures were greater than 1375°C. The ratio K/Na in the analyzed particles is lower than 1.0 but still greater than the added feed. This indicates that volatile compounds were able to leave the glass matrix at the elevated temperatures. It can also be noted that the solution samples that contain compounds were boron rich. Again, the volatile condensables may be meta-borates of sodium and potassium. This was partly confirmed by the data collected from thermal equilibrium calculations (using HSC for Windows) with the glass components.

Chromium was detected in most of the samples. Chromium is a major constituent of stainless steel, K-3 refractory, and the Inconel 690 electrodes. Nickel was only found in trace amounts in one of the off-gas particles analyzed by the SEM, nickel is a constituent of stainless steel and Inconel 690.

The presence of Mo was also detected, the source has not been identified. This element, Mo, does form a very volatile oxide and was not a major component of the surrogate feed.

Melter Temperature Measurement

Melter temperature was monitored using (10) infrared thermocouples that measured refractory surface temperature at various locations near the melter chamber (see Figure 2 for locations). Power to the joule electrodes was monitored by the infrared thermocouples. Test data (see Table 2) indicates that the highest temperature, per the infrared thermocouples, during the melter test was 1052°C. Per off-gas analysis and electrode evaluations it has been determined that the melt temperature was greater than 1375°C. The target temperature of 1150°C and viscosity target of 30 - 100 Poise were substantially exceeded. At this temperature the viscosity could have been less than 10 Poise, resulting in high mobility of the glass and significant corrosion to the electrodes and refractory. Test data was compared to crucible scale melter operations and determined that the infrared thermocouples registered accurate temperature readings up to 900°C. Many factors contributed to the infrared thermocouples registering lower temperatures, but the blackbody coefficient of the K-3 was visually recognized and determined as significantly different than what was used for thermocouple calibration. During calibration of the infrared thermocouples, the emissivity of K-3 refractory was assumed as being constant during melter operations, but the emissivity of the refractory changed significantly. Per observations during melter operations a color change of the refractory was noted when the temperature of the melter reached 850°C. Also, the lens on one of the infrared thermocouples was damaged due to radiation heat. This particular thermocouple registered an invalid temperature reading. Temperature control of melter operations in the future will be conducted utilizing in-melt Type R thermocouples contained in Inconel 690 sheaths.

Melter Lid

Once the melter cooled to 100°C, the melter lid was removed from the melter chamber. As shown in Figures 7 and 8 the melter lid assembly was observed to be in excellent condition with no corrosion. Some glass deposits adhered to the K-3 refractory, but no evidence of deposition in the off-gas or feed lines was detected. Some evidence of oxidation of the feed line and off-gas line can be seen in both figures, but this was expected because both lines were fabricated with 304L stainless steel. As stated earlier, the feed lines and off-gas lines did plug during melter operations. The feed line was manually cleared during melter operations, but the off-gas line was not cleared during the test. Disassembly of the off-gas line indicated that the line plugged at its first bend. Therefore, the off-gas line was not plugged in the lid or melter interface.

Melter Electrodes

The melter electrodes were removed from the melter chamber, visually inspected, and analytically tested. The front and back electrodes, shown in Figures 9 and 10, were cross sectioned and metallurgically examined. Per visual inspection, both electrodes appeared as if the metal melted and began to drip from their surfaces. There is also some evidence of small beads of Inconel resting on the bottom of the chamber. In order to determine the cause for the electrodes to melt, the electrodes were prepared for metallurgic examination.

Figure 7. Pilot scale melter—melter lid.

Figure 8. Pilot scale melter—bottom view of melter lid.

Figure 9. Pilot scale melter—front electrode.

Figure 10. Pilot scale melter—back electrode.

The electrodes were mounted, polished, and examined using a microscope. The photomicrographs are shown in Figures 11, 12, and 13. The small bubble formations indicate that the electrodes clearly melted at the top, middle, and bottom sections. The photomicrographs also show that the bottom of the electrodes have undergone significantly more melting than the top of the electrodes. In order to determine if the melting was caused from temperature or high current density, melter operation test results were reviewed. A maximum current density of 4 amps/in.² was maintained during melter operations. The maximum current density was calculated based on the assumption that the entire electrode was completely submerged in conductive glass. If the electrodes are only 50% submerged the maximum current density is 2 amps/in.². Per review of the test results, the maximum current density was exceeded when the glass level in the chamber decreased. Figures 11, 12, and 13 confirm that the electrodes did melt more at the bottom than the top. This difference is a combination of residence time the electrodes were subjected to the high temperature molten glass and high current densities when the glass level decreased. Figure 11, top of the electrode, clearly shows that the electrode melted, at which time the maximum current density was not exceeded. Per review of the photomicrographs and melter test results, the molten glass attained a temperature greater than 1375°C, the melting point of Inconel 690. Figure 14 shows the front Inconel 690 electrode when removed from the melter chamber during disassembly.

The cross sectioned electrodes were then chemically analyzed using Energy Dispersive Spectroscopy (EDS) on the Scanning Electron Microscope (SEM). Tables 6, 7, and 8 show the elemental concentrations for the glass, Inconel 690 plate, and the melted Inconel 690. This was performed so the SEM test results can be evaluated to determine if there is evidence of chemical reactions with the glass

Figure 11. Photomicrograph—top of Inconel 690 electrode showing cast and wrought microstructure at 200x.

Figure 12. Photomicrograph—middle of Inconel 690 electrode showing cast and wrought microstructure at 37.5x.

Figure 13. Photomicrograph—bottom of Inconel 690 electrode showing cast and wrought microstructure at 37.5x.

Figure 14. Pilot scale melter—Inconel 690 front electrode when removed from melter chamber during melter disassembly.

Table 6. Glass compositions determined using scanning electron microscope.^a

Cation	K-Ratio	Weight Fraction	Atomic Fraction	Compound	wt%	wt% Norm
Naka	0.0006	0.0028	0.0232	Na	0.28	1.29
Alka	0.0057	0.0124	0.0879	Al	1.24	5.75
Sika	0.0246	0.0466	0.3177	Si	4.66	21.65
Clka	0.0004	0.0006	0.0032	Cl	0.06	0.27
Kka	0.0052	0.0058	0.0284	K	0.58	2.69
Crka	0.1187	0.1401	0.5164	Cr	14.01	65.14
Mnka	0.0008	0.0009	0.0032	Mn	0.09	0.43
Feka	0.0029	0.0035	0.0121	Fe	0.35	1.63
Nika	0.0022	0.0024	0.0080	Ni	0.24	1.14
Total		0.2151			21.51	

a. SEM EDS cannot quantify oxygen, concentrations are approximations.

Table 7. Inconel 690 plate determined using scanning electron microscope.^a

Cation	K-Ratio	Weight Fraction	Atomic Fraction	Compound	wt%	wt% Norm
Sika	0.0007	0.0017	0.0060	Si	0.17	0.30
Crka	0.1695	0.1622	0.3066	Cr	16.22	28.39
Mnka	0.0045	0.0043	0.0077	Mn	0.43	0.75
Feka	0.0581	0.0568	0.0999	Fe	5.68	9.93
Nika	0.3221	0.3464	0.5799	Ni	34.64	60.63
Total		0.5713			57.13	

a. SEM EDS cannot quantify oxygen, concentrations are approximations.

Table 8. Melted Inconel 690 determined using scanning electron microscope.^a

Cation	K-Ratio	Weight Fraction	Atomic Fraction	Compound	wt%	wt% Norm
Sika	0.0005	0.0013	0.0033	Si	0.13	0.16
Crka	0.0439	0.0409	0.0572	Cr	4.09	5.13
Mnka	0.0001	0.0001	0.0002	Mn	0.01	0.02
Feka	0.0782	0.0664	0.0865	Fe	6.64	8.83
Nika	0.6607	0.6887	0.8528	Ni	68.87	86.36
Total		0.7975			79.75	

a. SEM EDS cannot quantify oxygen, concentrations are approximations.

and melted and/or Inconel 690 plate. If a chemical reaction is observed the compositions for these reactions can be compared.

In Figures 15, 16, 17, 18, and 19 the left photomicrograph (17x) shows the electrode and a white box to indicate the location of magnification. The right photomicrograph (680x) includes an "X" where the SEM performed chemical analysis. Light phase, bright phase, and dark phase are referred to in the text and figures, these phases only represent coloration of scanned area of the right photomicrograph.

Figure 15 shows the dark phase at the glass/metal interface. Over 86% of the detected components was determined as silicon and potassium with a small amount of nickel, iron, and chrome (see Table 9). This indicates that the dark phases on the electrode mainly consist of the components from the glass and trace amounts of the electrode.

Figure 15. Glass/metal interface—dark phase.

Figure 16. Glass/metal interface—bright phase.

Figure 17. Glass/metal interface—light phase.

Figure 18. Melted metal—light phase.

Figure 19. Melted metal—dark phase.

Table 9. Glass/metal interface—dark phase determined using scanning electron microscope.^a

Cation	K-Ratio	Weight Fraction	Atomic Fraction	Compound	wt%	wt% Norm
Alka	0.0014	0.0018	0.0404	Al	0.18	3.57
Sika	0.0205	0.0384	0.8126	Si	3.84	74.68
Pka	0.0000	0.0000	0.0000	P	0.00	0.00
Kka	0.0042	0.0060	0.0911	K	0.60	11.65
Crka	0.0014	0.0017	0.0189	Cr	0.17	3.21
Feka	0.0018	0.0021	0.0227	Fe	0.21	4.15
Nika	0.0012	0.0014	0.0142	Ni	0.14	2.73
Total		0.0515			5.15	

a. SEM EDS cannot quantify oxygen, concentrations are approximations.

Figure 16 shows the bright phase at the glass/metal interface. Over 96% of the composition detected is nickel and phosphorus (see Table 10). This indicates that the phosphorus, which is from the glass composition, reacts with the melted nickel. A concern regarding phosphorous induced corrosion to the electrodes was identified prior to the pilot plant melter test, but on-going testing is needed to quantify and verify this condition.

Figure 17 shows the light phase at the glass/metal interface. Approximately 93% of the detected elements was chromium and silicon (see Table 11). The chromium and silicon were found in the glass (see Table 6), but no chromium is in the surrogate feed composition (see Table 1). The high chromium

Table 10. Glass/metal interface—bright phase determined using scanning electron microscope. ^a

Cation	K-Ratio	Weight Fraction	Atomic Fraction	Compound	wt%	wt% Norm
Alka	0.0003	0.0011	0.0043	Al	0.11	0.23
Sika	0.0007	0.0016	0.0057	Si	0.16	0.31
Pka	0.0371	0.0727	0.2413	P	7.27	14.51
Kka	0.0000	0.0000	0.0000	K	0.00	0.00
Crka	0.0158	0.0153	0.0302	Cr	1.53	3.05
Feka	0.0000	0.0000	0.0000	Fe	0.00	0.00
Nika	0.3778	0.4101	0.7185	Ni	41.01	81.91
Total		0.5007			50.07	

a. SEM EDS cannot quantify oxygen, concentrations are approximations.

Table 11. Glass/metal interface—light phase determined using scanning electron microscope. ^a

Cation	K-Ratio	Weight Fraction	Atomic Fraction	Compound	wt%	wt% Norm
Alka	0.0009	0.0021	0.0412	Al	0.21	2.41
Sika	0.0055	0.0101	0.1924	Si	1.01	11.72
Pka	0.0000	0.0000	0.0000	P	0.00	00.00
Kka	0.0018	0.0018	0.0252	K	0.18	2.13
Crka	0.0585	0.0704	0.7242	Cr	7.04	81.64
Feka	0.0008	0.0010	0.0091	Fe	0.10	1.11
Nika	0.0008	0.0009	0.0078	Ni	0.09	0.99
Total		0.0863			8.63	

a. SEM EDS cannot quantify oxygen, concentrations are approximations.

content in the glass has been determined by laboratory melter tests to come from the K-3 refractory. Test results also show that over time the amount of chromium going from the refractory to the glass decreases. However, most of the chromium shown in these photomicrographs is the chromium from the Inconel 690. The chrome has chemically reacted with the glass, much like the chrome from the K-3 refractory. The addition of chromium to the glass matrix causes the glass viscosity to increase. Again, more studies are needed to be conducted before the extent of the chromium content in the glass can be quantified.

Figure 18 shows the light phase of the melted metal. The components that were detected on this photomicrograph (see Table 12) are nearly the same as the analysis completed on the melted metal. The amount of silicon in Table 12 is insignificantly greater than the melted metal.

Figure 19 shows the dark phase of the melted metal. The main component detected is nickel, but nearly 10% phosphorus was also found (see Table 13). Again, a reaction between the phosphorus, glass component, and nickel, electrode component, was detected.

The SEM analysis on the Inconel 690 electrodes does provide a good basis that future electrode test coupons can be compared. However, due to the "out-of-range" operating temperature of the molten glass for this pilot run, no conclusions can be made regarding corrosion of the electrodes for this feed

Table 12. Melted metal—light phase determined using scanning electron microscope. ^a

Cation	K-Ratio	Weight Fraction	Atomic Fraction	Compound	wt%	wt% Norm
Crka	0.0284	0.0263	0.0574	Cr	2.63	5.14
Mnka	0.0000	0.0000	0.0000	Mn	0.00	0.00
Feka	0.0494	0.0415	0.0843	Fe	4.15	8.10
Nika	0.4157	0.4447	0.8584	Ni	44.47	86.76
Total		0.5125			51.25	

a. SEM EDS cannot quantify oxygen, concentrations are approximations.

Table 13. Melted metal—dark phase determined using scanning electron microscope. ^a

Cation	K-Ratio	Weight Fraction	Atomic Fraction	Compound	wt%	wt% Norm
Pka	0.0330	0.0630	0.1609	P	6.30	9.24
Crka	0.0216	0.0205	0.0312	Cr	2.05	3.01
Mnka	0.0007	0.0006	0.0009	Mn	0.06	0.09
Feka	0.0270	0.0227	0.0322	Fe	2.27	3.33
Nika	0.5438	0.5732	0.7748	Ni	57.52	84.33
Total		0.6821			68.21	

a. SEM EDS cannot quantify oxygen, concentrations are approximations.

composition. Conclusions can be made that chemical reactions between the glass and electrodes will occur, if the electrode melting temperature is exceeded.

Melter Chamber

Inspection of the melter chamber indicated that the K-3 refractory did not undergo significant corrosion during melter testing. However, the refractory and Monofrax Type K cement (mortar) cracked at very critical locations, such as around the melt chamber. As shown in Figure 20 the Type K cement and refractory cracked near the melter chamber, thus allowing the molten glass to drain from the chamber. For the most part, the molten glass drained between the seams of the K-3 refractory blocks, which was sealed by the Type K cement. The glass viscosity was low, thus allowing the glass to flow through small cracks or seams. This is another indication that the temperature of the molten glass was much greater than 1150°C. Figure 21 shows the molten glass drain path and where the glass collected upon exiting the melt chamber. The white colored material in the glass is Kaowool insulation that dissolved when contacted by the molten glass.

Figure 22 shows a side view of the melter assembly prior to complete disassembly. Many cracks in the refractory are apparent on all parts of the melter. Per visual inspection the glass only breached the melter on the left side. Because of the number of cracks in the refractory during cool-down, it was determined that complete disassembly of the melter before reuse was required.

Figure 20. Glass chamber and side walls during disassembly.

Figure 21. Glass melter showing where the molten glass breached the melt chamber.

Figure 22. Glass melter prior to complete disassembly.

Melter Pour Spout

Once it was determined that the melter would require rebuilding due to the cracked refractory adjacent to the melter chamber, complete disassembly of the melter was initiated. As shown in Figure 23, the glass entered the pour spout, but due to its low viscosity and high temperature the glass leaked through very small seams at the pour spout/refractory interface and contacted the pour spout resistance heater. The resistance heater materials of construction are designed to withstand temperature, but not corrosive glass, therefore the heater was dissolved and failed to operate during pour operations.

The riser, shown in Figure 24, was in the same condition as the pour spout. Molten glass leaked between the seams of the refractory, thus destroying the resistance heater. Figure 24 shows the riser refractory block, which is laying down on the melter chamber base, split from the vertical pour spout block. The amount of molten glass in the riser area is much less than that found in the pour spout. The green colored material on the base block is refractory mortar, the light greenish yellow material is glass that has cooled in the riser block. The pour spout and riser areas, like the melter chamber, was breached by molten glass entering through small cracks and seams, thus another indicator that the glass was high in temperature and low in viscosity.

Figure 23. Melter pour spout.

Figure 24. Riser block assembly.

Melter Control

The DCS performed as designed. The melter operators relied on the DCS to control, monitor, and provide online output of critical pilot plant operating parameters. Since this was the first test of the pilot scale melter, many features such as maximum power and alarm setpoints were initially setup very conservatively. Many of these setpoints were modified during melter operations that allow the operator more time and flexibility to correct the alarms. The control system was programmed to alarm and shut down melter operations once a parameter exceeded its preset upper and lower control limits.¹⁴ The upper and lower control envelopes were changed during pilot plant operations. These changes increased the

control envelopes so the DCS would not alarm and shut down pilot plant operations. For all future tests the upper and lower control limits in the DCS will be programmed similar to the start of the first test, except the alarms will not shut down the melter. Melter operators concurred that during pilot plant operations, the operator should be stationed at the controls, therefore if an alarm occurs and justifies shut down, the operator can engage the emergency stop switch. The melter control system performed very well and will be utilized in future melter pilot plant testing.

Conclusions and Recommendations

Operation of the pilot plant melter system indicates that HAW-SBW can be vitrified to a molten glass. Operation of this melter test has illustrated the importance of selecting proper instrumentation, materials of construction, and processes to successfully immobilize INEEL high level wastes. Per review of the melter test results and operations, many recommendations and conclusions have been identified and incorporated into design and operation of the upcoming pilot plant melter test.

The pilot plant melter operated for 57 hours and was shut down because molten glass breached the melter chamber. Temperature control for the glass melter was inadequate, thus causing the melt temperature to be greater than 1375°C. Per review of melter test results, it has been determined that if the target temperature of 1150°C was maintained, melter operations would have been successful. Monitoring melter temperature has undergone a thorough review by LMITCO, PNNL, and WSRC personnel. These reviews and collaborations have concluded that in-melt type R thermocouples protected by Inconel 690 sheaths should replace the infrared thermocouples. In addition to in-melt thermocouples, melt temperature will be periodically monitored by manually submerging a thermocouple in the melt to ensure the thermocouples are adequately monitoring melt temperature.

The start-up and pour heaters operated successfully during this melter test, but per reviews a design change has been recommended. These changes include moving the start-up heaters to the melter chamber head space, very similar to the location of the DWPF melter start-up heaters. These heaters are silicon carbide resistance heaters that are protected by alumina sheaths. This same configuration is being utilized in the pour spout. This configuration provides direct heating of the chamber and pour spout areas, whereas the initial melter design required the resistance heaters to heat the refractory outside walls, thus conductively heating the chamber and pour spout areas. These design changes will allow the outside bricks to cool, therefore if the glass flows through cracks and seams away from the melt chamber, temperature will decrease, thus cooling the glass to solidify and seal the glass melter. This is the same design strategy is used by PNNL and WSRC engineers.

Another concern that has been addressed is the formation of heat stress fractures of the refractory during the first melter run. Per discussions with Monofrax K-3 representatives, the heat-up and cool-down rates need to be controlled to 10°C/hr. The Bailey DCS logic has been designed to monitor and control the temperature heat-up and cool-down rates. Controlling temperature will reduce the amount of stress fractures of the refractory, thus allowing cycling of the melter. This is important considering the amount of time required for rebuilding. However, some time will be spent to operate the melter during heat-up and cool-down, thus lengthen the total operating time of the melter pilot plant.

The deposits in the off-gas stream were analyzed and determined to be enriched in potassium and boron. The metaborates were expected from the calculated thermal equilibrium calculations (applying HSC for Windows) and work of other investigators. The potassium enrichment over that of the sodium was not expected and may be a result of the higher volatility of sodium at high temperatures. Plugging of the off-gas line was caused by a higher accumulation of condensables than expected. These

condensables collected just downstream of the off-gas line/melter interface, thus causing the off-gas system to become inoperable during feed operations. This phenomenon can be mostly attributed to the high temperatures.

Design changes to the off-gas system will allow it to retain more of the condensables in a position such that they will fall back into the melt or at least find it more difficult to enter the off-gas line. These changes include lowering the flow velocity by installing a larger diameter off-gas line at the melter entrance. A removable orifice has been added to the off-gas line at the melter/off-gas interface. This will aid in carrying the condensables rather than allowing surrogate feed to enter of the off-gas system. This design change is recommended because of operating experience at the DWPF.¹⁵ The amount of material available to enter the off-gas will be less than that during the first run because of the lower operating temperature and other design changes that increase the effectiveness of the feed port seal.

To avoid gas lifting of the scrub solution, another vessel has been constructed that will contain the contents of the carboy to protect the off-gas system. The bottom of the scrubber could have been redesigned such that the scrubber would contain the scrub solution volume, but it was desired that a transparent vessel be used so that the solution color and volume could be easily monitored during a melter testing. This change would require other modifications such as level instrumentation and view ports.

Many lessons have been learned and documented from the first melter test. All these provide operating experience to support the high level waste technology development mission. This run, although not entirely successful, provides vital information, knowledge, and experience necessary to evaluate and select suitable immobilization processes. This experience has resulted in the incorporation of specific modifications into the design of the pilot plant melter and operating procedures. The redesigned pilot plant melter is scheduled for operation in October 1997.

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Appendix A

Bailey, Distributed Control System, Operating Screens

Melter Control Display

- A 794 DEG C
MELTER HIGHEST
HI-TEMP
- B 0.00 WATTS
JOULE HEATER
J-ML-1
- C 0.00 AMPS
JOULE HEATER
I-ML-1

- D OFF
MELTER STARTUP
HE-ML-1
- E OFF
MELTER DRAIN
HE-ML-2
- F OFF
MELTER POUR
HE-ML-3
- G OFF
MELTER THROAT
HE-ML-4

MELTER OFFGAS SYSTEM

F BLO-ML-1
OFFGAS BLOWER
HANDSWITCH

ON ☒
OFF

P-ML-1
MELTER OFFGAS
PRESSURE

0.00 IN H2O

P-ML-3
OFFGAS SCRUBBER
OUTLET PRESSURE

0.04 PSIG

G P-ML-2
OFFGAS MANIFOLD
VACUUM

PSIG 5.00
4.50
2.27 ☒ 2.20

0.00
50.2% AUTO LOCAL
TRGT SET: 2.20

T-ML-11
MELTER OFFGAS
TEMPERATURE

19.12 DEG C

H F-ML-1
MELTER OFFGAS
FLOW RATE

LPM 30.00
15.00
12.20 ☒ 12.00 5.00
0.00
42.8% AUTO LOCAL

[illegible]

A-3

A-4

11 12:03			11 12:23			11 12:43			11 13:03			11 13:23			11 13:43			11-Feb-1997 13:43:00		
																	F T-ML-6	PV		
																	789.52 DEG C	1300.0		
																	789.5	800.0		
																	G T-ML-7	PV		
																	788.22 DEG C	1300.0		
																	788.2	800.0		
																	H T-ML-8	PV		
																	191.80 DEG C	1300.0		
																	192.1	800.0		
																	I T-ML-9	PV		
																	0.00 DEG C	1300.0		
																	* 1	BAD	800.0	
																	J T-ML-10	PV		
																	790.53 DEG C	1300.0		
																	790.5	800.0		
E	G	H	J	F	G	H	J	F	G	H	J	F	G	H						
11 12:03			11 12:23			11 12:43			11 13:03			11 13:23			11 13:43			11-Feb-1997 13:43:00		

A-5

11 12:04 11 12:24 11 12:44 11 13:04 11 13:24 11 13:44 11-Feb-1997 13:45:00

G HE-ML-4
MELTER THROAT
HEATER

ON NP

OFF ☐

J HS-ML-5
MATERIAL FEEDER
TOTALIZER RESET

RESET P

OFF ☐

A-6

11 12:03 11 12:23 11 12:43 11 13:03 11 13:23 11 13:43 11-Feb-1997 13:43:00

```

I      F-ML-2-0
        MATERIAL FEED
        RATE SET POINT

                        10.00
        6.00 LB/HR
                        0.00

```

F-ML-2-I
MATERIAL FEED
RATE

0.00 LB/HR

* 1