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Revision 0

DM100 and DM1200 Melter Testing with High Waste Loading glass Formulations for Hanford High-Aluminum HLW Streams

Test Plan 09T1690-1

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Office of River Protection

P.O. Box 450
Richland, Washington 99352

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Test Plan

DM100 and DM1200 Melter Testing with High Waste Loading Glass Formulations for Hanford High-Aluminum HLW Streams

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SECTION 1.0
INTRODUCTION

This Test Plan describes work to support the development and testing of high waste loading glass formulations that achieve high glass melting rates for Hanford high aluminum high level waste (HLW). In particular, the present testing is designed to evaluate the effect of using low activity waste (LAW) waste streams as a source of sodium in place of chemical additives, sugar or cellulose as a reductant, boehmite as an aluminum source, and further enhancements to waste processing rate while meeting all processing and product quality requirements. The work will include preparation and characterization of crucible melts in support of subsequent DuraMelter 100 (DM100) tests designed to examine the effects of enhanced glass formulations, glass processing temperature, incorporation of the LAW waste stream as a sodium source, type of organic reductant, and feed solids content on waste processing rate and product quality. Also included is a confirmatory test on the HLW Pilot Melter (DM1200) with a composition selected from those tested on the DM100. This work builds on previous work performed at the Vitreous State Laboratory (VSL) for Department of Energy's (DOE's) Office of River Protection (ORP) to increase waste loading and processing rates for high-iron HLW waste streams [1] as well as previous tests conducted for ORP on the same waste composition [2, 3]. This Test Plan is prepared in response to an ORP-supplied statement of work [4].

It is currently estimated that the number of HLW canisters to be produced in the Hanford Tank Waste Treatment and Immobilization Plant (WTP) is about 12,500 [5]. This estimate is based upon the inventory of the tank wastes, the anticipated performance of the sludge treatment processes, and current understanding of the capability of the borosilicate glass waste form. The WTP HLW melter design, unlike earlier DOE melter designs, incorporates an active glass bubbler system. The bubblers create active glass pool convection and thereby improve heat transfer and glass melting rate. The WTP HLW melter has a glass surface area of 3.75 m^2 and depth of ~1.1 m. The two melters in the HLW facility together are designed to produce up to 7.5 MT of glass per day at 100% availability. Further increases in HLW waste processing rates can potentially be achieved by increasing the melter operating temperature above 1150°C and by increasing the waste loading in the glass product. Increasing the waste loading also has the added benefit of decreasing the number of canisters for storage.

The current estimates and glass formulation efforts have been conservative in terms of achievable waste loadings. These formulations have been specified to ensure that the glasses are homogenous, contain essentially no crystalline phases, are processable in joule-heated, ceramic-lined melters and meet WTP contract requirements. The WTP's overall mission will require the immobilization of tank waste compositions that are dominated by mixtures of aluminum (Al), chromium (Cr), bismuth (Bi), iron (Fe), phosphorous (P), zirconium (Zr), and sulfur (S) compounds as waste-limiting components. Glass compositions for these waste mixtures have been developed based upon previous experience and current glass property models. Recently, DOE has initiated a testing program to develop and characterize HLW glasses with higher waste loadings [6]. Results of this work have demonstrated the feasibility of increases in waste-loading from about 25 wt% to 33-50 wt% (based on oxide loading) in the glass depending on the waste stream [2, 3]. It is expected that these higher waste loading glasses will reduce the HLW canister production requirement by about 25% or more.

DOE HLW treatment programs have featured joule heated ceramic melter technology for the vitrification of high level tank waste. The melter technology used at the West Valley Demonstration Project (WVDP) in New York and at the Defense Waste Processing Facility (DWPF) at the Savannah River Site (SRS) process(ed) HLW in ceramic melters at an operating temperature of 1150°C. Historically, HLW melters are operated at temperatures of 1150°C to allow for sufficient temperature control for normal as well as upset conditions in an operating melter, while still protecting the electrodes from potential damage due to unanticipated high temperature swings. Since the HLW melters deployed in the United States at West Valley and DWPF do not actively mix the glass pool, temperature variations within the glass pool can be relatively large (~ ±75°C) with respect to the nominal operating temperature since natural convection within the glass pool is limited in the viscous molten glass. In advancing the technology, Energy*Solutions*/VSL have demonstrated on very large scale melters (Energy*Solutions* M-Area facility, RPP-WTP HLW pilot melter, and the RPP-WTP LAW pilot melter) that active mixing of the glass pool using Energy*Solution*'s patented bubbler technology significantly reduces the temperature gradient within the glass pool and allows the melter to be controlled in a tighter operating band. As a result, the operating temperature of the melter can be modestly increased to about 1175°C with the current materials of construction, (and up to 1225°C with changes of electrode and bubbler materials) while maintaining the operating integrity of the melter at the higher temperature. Tests conducted with various HLW waste streams on the DM100 and DM1200 melters have demonstrated increases in glass production rates from 0 to 225 percent while increasing the processing temperature from 1150°C to 1175°C [2, 7]. Further increases in operating temperature to higher temperatures (1200°C) have the potential to further increase processing rate as well as increased waste loading, both of which translate into significant cost savings.

Under a separate contract to support the WTP, the VSL is developing and testing glass formulations for WTP HLW waste compositions to provide data to meet the WTP contract requirements and to support system design activities [8-10]. That work is based upon small-scale batch melts ("crucible melts") using waste simulants. Selected formulations have also been tested in small-scale, continuously fed, joule-heated melters (DM100) [11-14] and, ultimately, in the HLW DM1200 Pilot Melter [7, 13-20]. Such melter tests provide information on key process factors such as feed processing behavior, dynamic effects during processing, secondary phase formation, processing rates, off-gas amounts and compositions, foaming control, etc., that cannot be reliably obtained from crucible melts. This sequential scale-up approach in the vitrification testing program ensures that maximum benefit is obtained from the more costly melter tests and that the most effective use is made of those resources.

The glass formulation and melter testing work proposed in this Test Plan is aimed at identifying glass compositions that maximize waste processing rates for one of the four waste streams previously specified by ORP [6]. The previous tests with these waste streams demonstrated substantial increases in waste loading; however, production rates with aluminum and aluminum in combination with sodium limited wastes were only a third to a half of the rates obtained for bismuth, chromium, and iron limited wastes [2]. Subsequently, tests were conducted demonstrating increased glass production rate while retaining high waste loading and acceptable glass properties for the aluminum limited waste through the manipulation of the glass formulations and glass forming additives [3]. The current work will build upon these glass formulations by determining the feasibility of incorporating an LAW waste stream as an additive source. This information will provide ORP with a basis for projection of the amount of Immobilized High Level Waste (IHLW) to be produced at Hanford, and evaluation of the likely potential for future enhancements of the WTP over and above the present well-developed baseline. It should be noted that the compositions of the four specified waste streams differ significantly from those of the feed tanks (AZ-101, AZ-102, C-106/AY-102, and C-104/AY-101) that have been the focus of the extensive technology development and design work performed for the WTP baseline. In this regard, the work proposed in this Test Plan is complementary to, and necessarily of a more exploratory nature than the work in support of the current WTP baseline. It should be noted, therefore, that to the extent that the present effort is successful, considerable further work would be required to bring the level of confidence in the new glass composition regions to a similar level of maturity to that of the current WTP baseline. Additional testing at the crucible and melter scales will be needed to determine the robustness of the new compositions with respect to variations in the feed compositions that may result from process variations as well as for the collection of data to support engineering and permitting requirements using a WTP prototypic off-gas system. In addition, since the high waste loading glass compositions are likely to be in a new composition space as compared to the current WTP compositions, additional effort will be required to develop and extend the current qualified glass composition region and supporting models to include these new compositions.

1.1 Test Objectives and Success Criteria

The principal objective of the work described in this Test Plan is to evaluate the effects of incorporating a LAW waste stream as a sodium source with an aluminum limited HLW waste composition specified by ORP, while maintaining high waste loadings and acceptable glass properties. Also evaluated will be the effects of the form of aluminum in the waste, the type of reductant used, glass temperature, waste solids content, as well as further enhancements of the glass formulation on waste and glass processing rates. This will be accomplished through a combination of crucible-scale tests, confirmation tests on the DM100 melter system, and demonstration at pilot scale (DM1200). The DM100-BL unit was selected for these tests since it was used previously with the HLW waste streams evaluated in this study [2, 3], tests on HLW glass compositions [11-20] that were used to support subsequent tests on the HLW Pilot Melter [3, 7, 13-20], to assess the volatility of cesium and technetium during the vitrification of an HLW AZ-102 composition [21], and to evaluate glass composition and properties on the production rate of a high iron HLW waste composition [22, 23]. The same melter was selected for the present tests in order to maintain comparisons between the previously collected data. The proposed tests will provide information on melter processing characteristics and off-gas data, including formation of secondary phases and partitioning. Once the glass formulation development work is complete, the data will be reviewed with ORP prior to making a decision on which formulation should be advanced to DM100-BL testing. A similar review process with ORP will occur after DM100 tests to select the composition for testing on the DM1200.

The principal objectives of this work are to:

- Assess the effect of using the LAW waste as an additive source with an aluminum-limited HLW waste stream.
- Determine the effect on glass production rates of decreased feed solids content as a result of the incorporation of LAW waste as an additive source with an aluminum-limited HLW waste stream.
- Determine the effect on glass production rates of reductant type used as a result of the incorporation of LAW waste as an additive source with an aluminum-limited HLW waste stream.
- Determine the effect on glass production rates of boehmite as an aluminum source while processing an aluminum-limited HLW waste stream.
- Develop and test an improved aluminum-limited glass with Al_2O_3 concentration of about 24 wt% and waste loading of ~ 45%, and high processing rate.
- Test product glasses for properties including viscosity, electrical conductivity, crystallinity, gross glass phase separation, and chemical durability (TCLP and PCT).
- Determine the effect of modest increase in melter operating temperature on production rate and melter emissions for six different feed compositions.

This Test Plan provides an outline of the test objectives for the melter tests; a description of the melter systems and experimental methods; the proposed melter test matrix; test monitoring, sampling and analysis methods, and the planned schedule.

1.2 Test Overview

This section provides a brief overview of the components of the testing that will be performed. Further details are provided in the remainder of the document.

1.2.1 General Approach

The general approach for the development and identification of each HLW glass composition involves the following sequence of progressively more realistic tests at larger scales:

(1) The form of aluminum in the simulated waste will be changed from hydroxide to boehmite in the feed formulation previously used on the DM100 [3]. This composition will be processed on the DM100 in two 50-hour segments; one will be at the nominal operating temperature of 1150°C and the other will be at an elevated temperature of 1200°C to determine the effect of glass temperature on production rate and melter emissions. Data from this test will be compared to that collected in previous tests conducted under the same conditions to determine the effect of the aluminum form in the waste on feed properties, production rate, and emission rate.

(2) The source of sodium for a previously processed feed formulation [3] will be changed from the additive sodium carbonate to a simulated LAW waste stream. In addition to sodium, the LAW waste stream contains significant quantities of nitrate as well as much lesser amounts of aluminum, nitrite, sulfate, and phosphate. Sugar as reductant is added in proportion to nitrates during LAW tests to mitigate foaming. Tests will be performed on the DM100 with four different feeds: two feed solids contents (500 g glass per liter to compare to previous tests and about 400 g glass per liter as a consequence of adding the aqueous LAW waste stream) as well as two reductant types (sugar and cellulose). For tests using the LAW waste stream as the sodium source, eight DM100 tests (totaling 400 hrs) will be performed. One 50-hour test segment will be at the nominal operating temperature of 1150°C and the other will be at an elevated temperature of 1200°C to determine the effect of glass temperature on production rate and melter emissions. Collectively, this data set will be used to show the effects of using the LAW waste stream as a sodium source, reductant type, waste solids content, and glass temperature on production rate at constant melter operating conditions. A crucible melt of the new glass composition will be prepared prior to melter testing to ensure that the changes in the glass composition from the incorporation of the LAW waste stream do not have a deleterious effect on important processing properties or chemical durability.

(3) One of the reductants will be selected for processing on the DM1200 based on results from the DM100 melter tests using the simulated LAW waste stream as a sodium source. Four two-day melter tests will be performed: the first two at the feed solids content of 500 g glass per liter to compare to previous tests conducted with an aluminum-limited glass formulation [3], and the second at the feed solids content of about 400 g glass per liter resulting from the addition of the LAW waste stream to the feed. For each feed solids content, a two-day test will be conducted at both the bubbling rate adjusted to achieve a target production rate of 1050 $kg/m^2/day$ and the optimized bubbling rate to achieve the maximum glass production rate. All tests will be conducted at the nominal glass pool temperature of 1150°C. Data from these tests will demonstrate the ability to achieve the target production rate with aluminum-limited HLW stream with a LAW stream as a sodium source, as well as the effect of reductants, nitrates, and feed solids content on production rate and melter emissions.

(4) Glass formulations and modified feed formulations will be prepared at crucible-scale and characterized to select a composition that maximizes the processing rate for the high

aluminum waste composition specified by ORP. The form of the aluminum will be aluminum hydroxide to compare with previously conducted tests [3]. The glass formulations will be actively designed in that characterization data from the preceding set of crucible melts will be used to design the next set of formulations. Characterization of the melts will include composition, PCT, TCLP, viscosity, electrical conductivity, and liquidus temperature (crystal content). In addition, the formulations will be subjected to a small-scale melt rate screening test to provide a basis for down-selection of the preferred compositions for subsequent melter testing. Not all crucible melts will be analyzed for all properties. A new glass and modified feed formulation will be selected for melter testing and be fully characterized. Approximately seven crucible melts and feed formulations will be prepared and characterized to support the above.

(5) The optimized glass formulation selected from crucible studies to be performed as a part of this work will be processed on the DM100 in two 50-hour segments: one will be at the nominal operating temperature of 1150°C and the other will be at an elevated temperature of 1200°C to determine the effect of glass temperature on production rate and melter emissions. Data from this test will be compared to that collected in previous tests conducted under the same conditions to determine the effect of the optimized glass formulation on production rate, and emission rate.

1.2.2 Parameter Variations to be Tested

In summary, the approach involves small-scale testing that economically addresses glass composition and feed additive modifications that are likely to provide improvements in aluminum incorporation and melting rate, followed by confirmatory melter testing on the most promising candidates. As noted above, the intent of this effort is to complement the present WTP baseline glass formulation work; accordingly, only glass formulations that are compositionally distinct from the present WTP baseline compositions will be tested in this work to ensure that the maximum benefit to ORP is gained.

1.3 ORP Review of Test Program

In order to keep ORP apprised of the progress of the test program small-scale and crucible melt test results will be reviewed via telecom prior to each DM100 test. The review will focus on the testing results from the completed test phase and the rationale for the compositions recommended for use in the subsequent tests. In addition, results from DM100 tests will be provided to ORP and the data will be reviewed via telecom to select a glass and feed composition for the DM1200 test.

Other reporting requirements are given in Section 6.0.

SECTION 2.0
MELTER SYSTEMS DESCRIPTION

2.1 DM100

2.1.1 DM100 Feed System

A schematic diagram of the DM100 vitrification system is shown in Figure 2.1. The melter feed is introduced in batches into a feed container that is mounted on a load cell for weight monitoring. The feed is stirred with a variable speed mixer and constantly recirculated except for periodic, momentary interruptions during which the weight is recorded. In typical operation, the feed is introduced into the melter using a series of valves operated in a timed sequence to mimic the operation of an air displacement slurry (ADS) pump. However, in these tests, a peristaltic pump will be used in order to provide a more uniform delivery of feed to the melt surface. Feed will be directed from the recirculation loop that extends to the top of the melter then diverted to the peristaltic pump, which regulates the flow of feed through a Teflon-lined feed line and water-cooled feed tube into the melter.

2.1.2 DM100 Melter System

Cross-sectional diagrams of the DM100-BL melter are shown in Figures 2.2.a-c. The DM100-BL unit is a ceramic refractory-lined melter fitted with a total of five electrodes: two pairs of opposing Inconel 690 plate electrodes and a bottom electrode. Power can be supplied in either three-phase or single-phase configurations. All of the tests in the present work will be performed with the upper and lower electrodes on each side connected together and powered by a single-phase supply; the bottom electrode will not be powered. Melt pool agitation is achieved by either a removable lance entering from the top of the melter or a permanent bubbler installed through the bottom electrode. In these tests, the lance bubbler will be used. The glass product is removed from the melter by means of an airlift discharge system. The melter has a melt surface area of 0.108 m^2 and a variable glass inventory between 110 kg, when only the bottom pair of electrodes is used and about 170 kg when both pairs of electrodes are used.

2.1.3 DM100 Off-Gas System

For operational simplicity, the DM100-BL melter is equipped with a dry off-gas treatment system involving gas filtration operations only, as shown in Figure 2.1. Exhaust gases leave the melter plenum through a film cooler device that minimizes the formation of solid deposits. The film-cooler air has constant flow rate and its temperature is thermostatically controlled. Consequently, the exhaust gases passing through the transition line (between the melter and the first filtration device) can be sampled at constant temperature and airflow rate. The geometry of the transition line conforms to the requirements of the 40-CFR-60 air sampling techniques. Immediately downstream of the transition line are cyclonic filters equipped with internal coarse filter elements followed by conventional pre-filters and High-Efficiency Particulate Air (HEPA) filters. The temperatures of the cyclonic and the HEPA filters are held above 100°C to prevent moisture condensation. The entire train of gas filtration operations is duplicated and each train is used alternately. An induced draft fan completes the system.

2.1.4 DM100 Sampling Points

A variety of sampling points is available on the DM100 system. The sample types and frequencies are shown in Table 2.1 and are described further in Section 5. The sampling points that will be used in this work are as follows:

- *Melter Feed*: Samples of the melter feed will be taken either from the parent feed batch or the melter feed line to provide confirmation of the feed composition.
- *Glass Product*: Samples of the glass product will be taken from glass that is air-lift discharged into steel cans.
- *Glass Pool*: Glass samples will also be taken directly from the glass pool ("dip" samples).
- *Off-gas 1:* Isokinetic sampling of melter exhaust will be conducted at a point located immediately downstream of the film cooler.
- *Off-gas 2:* A sampling point located down stream of the HEPA filter will be used for continuous emissions monitoring (CEM) by Fourier transform infra-red spectroscopy (FTIR) of a wide variety of gaseous species, including NO, NO_2, N_2O, CO, and SO_2.

Glass product from the melter tests will be characterized for composition as well as processing and product quality requirements. Analyses of glass and off-gas samples will provide data for mass balances around the melter for key constituents. In addition to samples collected for analysis at VSL, glass samples will be collected for shipment to ORP, as indicated in Tables 2.1 and 2.2. Samples collected for shipment to ORP will be treated in a manner consistent with quality control guidance in Chapter 1 of SW-846. Detailed procedures which will be followed for chain of custody are given in SW-846 Sections 9.2.2.6 and 9.2.2.7. The latter provides instruction for notebook documentation, sample labeling, custody seals, and chain of custody records. The glass to be shipped will be non-radioactive and non-hazardous; however, all applicable DOT regulations stipulated in 10 CFR 49 will be followed.

2.2 DM1200

2.2.1 Feed System

The feed material for these tests will be prepared and controlled according to VSL specifications by a chemical supplier, as detailed in Section 3. Each batch of feed slurry will be shipped to VSL in lined 55-gallon drums (approximately 16 per shipment), which will be staged for unloading into the mix tank. Both the mix tank and the feed tank are 750-gallon polyethylene tanks with conical bottoms that are fitted with mechanical agitators; the feed tank is also fitted with baffles to improve mixing. Any required feed additive can be added to the mix tank. Five calibrated load cells directly mounted on the legs of the feed tank are used to measure additions to, and removal from, the feed tank and are electronically monitored to determine the feed rate to the melter. The requisite amount of feed is pumped to the feed tank from the mix tank; measured amounts of water will be combined by weight with the feed at this point to adjust the concentration of the melter feed. The material in the feed tank is constantly recirculated from the feed tank discharge outlet, at the tank bottom, to the tank inlet at the top, which provides additional mixing.

The feed is introduced into the melter using an ADS pump, which is the present WTP baseline. The feed transfer line extends from the outlet of the ADS pump in the feed tank to the top of the melter. Feed is introduced into the melter through an un-cooled feed nozzle that is located above the center of the glass pool. Only one feed tube is used to represent the planned number of feed tubes per unit melt surface area in the full-scale WTP HLW melter. The operation of the ADS pump is controlled from the melter computer control system. The ADS pump works by opening the pump reservoir to the feed tank using a double-acting air cylinder and mechanical link to actuate the poppet. The reservoir is filled with slurry by gravity. After sufficient time is allowed to fill the reservoir (a few seconds), the poppet is toggled to close the reservoir to the tank and open the transfer line. After a desired delay time (dependent on the desired feed rate) the reservoir is pressurized with air to transfer the slurry (about 1.6 liter/shot) to the melter. This cycle is repeated at the rate required to provide the desired feed rate.

When necessary, a backup system is used to introduce feed into the melter with an air operated diaphragm (AOD) pump system that simulates the pulsed feeding action of an ADS pump. The recirculation loop extends to the top of the melter where feed is diverted from the recirculation loop into the melter through a Teflon-lined feed line and water-cooled feed tube. Two computer-operated pinch valves, one on the feed line and one on the recirculation loop, are activated in a timed sequence to introduce feed into the melter at the desired rate. The feed rate is regulated by adjusting the length of each pulse, the time between each pulse, and the pressure applied to the recirculation loop.

2.2.2 Melter System

The DuraMelter 1200 (DM1200), which is the HLW Pilot Melter, will be used for these tests. Cross-sectional diagrams of the melter illustrating the discharge chamber and electrode configuration are provided in Figures 2.3 and 2.4. The DM1200 is a Joule-heated melter with Inconel 690 electrodes and thus has an upper operating temperature of about 1200°C. The melter shell is water-cooled and incorporates a jack-bolt thermal expansion system. The footprint of the melter is approximately 8 ft. by 6.5 ft. with a 4 ft. by 2.3 ft. air-lift discharge chamber appended to one end; the melter shell is almost 8 ft. tall. The melt surface area and the melt pool height are approximately 32 percent and 57 percent, respectively, of the corresponding values for the full-scale HLW melter. The discharge riser and trough are full-scale to verify pouring performance. Other aspects of the discharge system are also prototypical such as the chamber ventilation scheme. The glass contact refractory is Monofrax® K-3 while the plenum area walls are constructed of Monofrax® H refractory. The surface of the glass pool is 34" by 54" with a nominal glass depth of 25". The resultant melt volume is approximately 45,000 cubic inches (735 liters), which represents a glass tank capacity of more than 1.7 metric tons of glass. However, since the typical operating glass level is closer to 29 inches, the effective glass volume during testing is actually about 849 liters, giving an inventory of about 2.0 metric tons. The DuraMelter™ 1200 is fitted with one pair of electrodes placed high on opposite walls of the melter as well as one bottom electrode. The side electrodes are 11" by 34" giving an electrode area for the pair of about 750 sq. in. Depending on the glass level, the plenum space extends about 33" to 36" above the melt surface resulting in a plenum volume ranging from about 43 to 46 ft^3.

The single-phase power supply to the melter electrodes (250 kW design power) is derived from the DuraMelter 1000 transformers by wiring them in parallel and using a single large silicon controlled rectifier. Current can be passed either from the side electrodes to the bottom electrode or between the two side electrodes only, by rearranging jumpers; only side-to-side operation will be used for the present tests. Programmable process controllers are installed and can be used to control temperature or power. The melt temperature is controlled by configuring the process controller to maintain constant power and adjusting the power set-point as needed to maintain the desired operating temperature. Alarms can be set to detect out-of-range temperatures or power in the melter. Backup process controllers are installed to be used in case of failure of the main controllers. The entire system is supported by a back-up generator that is tripped on in the event of a power outage.

The DuraMelter 1200 has several other features. The lid refractory is prototypic and also includes a two-piece construction, which simulates the seam needed for the LAW lid that was planned to be fabricated in three pieces. Nozzles are provided for the off-gas film cooler, a standby off-gas port, discharge airlift, along with 11 ports available for top-entering bubblers, start-up heaters and other components as needed. In addition, a bubbler arrangement is installed in the bottom electrode with the objective of developing permanent bubblers for possible use on future melters. For the present tests, the optimum bubbler configuration established during previous tests with HLW simulants [7] consisting of two double-outlet, top-entering bubblers will be used, located in positions to mimic conditions in the WTP HLW melter. Figure 2.5 shows a schematic of the prototypical double-outlet bubbler design that was based on the combination of the results from these DM1200 tests and room-temperature tests that were performed in a transparent fluid simulating the properties of the glass melt [24]. These bubblers have outlets 8 inches apart and are placed on the melter floor. The orientation of the bubblers in the melter, as shown in Figure 2.6, results in one of the bubbling outlets being 11.3 inches from the feed tube.

2.2.3 Off-Gas System

The melter and entire off-gas treatment system are maintained under negative pressure by two Paxton external induced draft blowers. This negative pressure is necessary to direct the gases from the melter to the prototypical off-gas system. The off-gas treatment system, shown schematically in Figure 2.7, consists of a submerged bed scrubber (SBS); a wet electrostatic precipitator (WESP); a high-efficiency mist eliminator (HEME), a high-efficiency particulate air (HEPA) filter; a thermal catalytic oxidation unit (TCO); a NO_x removal system (SCR); a packed-bed caustic scrubber (PBS); and a second HEME. Note that the PBS and the second HEME are not part of the WTP off-gas train, which effectively ends at the SCR. The HEME is used to limit entrained particle carryover into the balance of the VSL ventilation system. The system can be functionally divided into four subsystems:

Particulate Removal: Components from the SBS to the HEPA serve to remove essentially all of the particulate from the gas stream with an estimated removal efficiency of greater than 99.9999% for particles greater than 0.3 μm in size. In the WTP facility, this provision serves to segregate the radioactive from the non-radioactive components in the system for maintenance and handling purposes.

VOC Control/Acid Gas: The TCO unit is designed to oxidize any hazardous organics that are present in the off-gas stream. This is followed by a SCR to remove NO_x gases and a PBS to remove remaining acid gases.

Stack System: The emergency/bypass exhaust system, which includes a second HEPA, and the primary off-gas system both feed into the building stack system for exhausting to the atmosphere.

Liquid Processing: Components including the water spray lines, liquid sampling and water storage tanks, as well as the effluent evaporator, function to sample and process the system liquids for recycle or discharge.

With minor exceptions, the DM1200 off-gas system processing sequence follows the proposed design for the full-scale WTP HLW melter system, except for cooling of the off-gas stream discharged from the SCR unit (which is present in the WTP off-gas train, but absent in the DM1200 system). Per WTP direction, the SBS unit that was used for previous DM1200 testing was modified in early 2004. Installation of the new system was completed in March 2004 and that unit will be used for the present tests. The changes were implemented to reflect modifications to the WTP SBS design that have taken place since the original DM1200 unit was installed. These modifications included changes to the diffuser plate design, down-comer jacket and connection to the diffuser plate, bed diameter, bed packing materials, cooling coils, and liquid overflow level.

Initial quenching of the melter exhaust gas stream is effected by the film cooler. Immediately upstream of the film cooler is the injection point for control air, which is used to regulate melter pressure. The gas entering the balance of the off-gas system is at a temperature of about 250 to 350°C and a flow rate of about 100-250 scfm, of which about 10-80 scfm is water vapor. The off-gas is then rapidly quenched by direct liquid water contact in the SBS, which also effects removal of most of the larger particulates. The piping between the film cooler and SBS has a high superficial gas velocity to minimize particulate deposition. The gas stream leaving the SBS is at a low temperature (typically between 40-50°C). Further mist and particulate removal is effected in the WESP, HEME and HEPA. The TCO and SCR follow the particle removal components and serve to destroy organic compounds and nitrogen oxides. Finally, the PBS provides acid gas removal. Water sprays are located in the WESP, PBS, and facility HEME to wash down deposits and dissolved species into their respective collection sumps from which they can be sampled. The system components are fabricated from corrosion resistant materials, including AL6XN and 316L stainless steel, and various plastics in less demanding locations. There are extensive provisions for sampling both the gas and liquid streams throughout the system in order to collect mass balance information and removal efficiency data for each treatment stage.

The off-gas system maintains the melter plenum under slight negative pressure, typically about -5 in. W.C. The plenum pressure is controlled by means of an air injection system that introduces a controlled air flow into the off-gas jumper just after the film cooler. The air is supplied by a blower through a diverter valve. The setting of the diverter valve, and therefore the air flow rate, is controlled by a process controller that responds to the signal from a melter pressure transducer. When the plenum pressure becomes more positive, the air injection flow rate is decreased, which tends to restore the pressure to the set-point. Conversely, the flow rate is increased when the plenum pressure becomes more negative.

2.2.4 DM1200 Sampling Points

Sampling points in the vitrification system are shown in Figure 2.7 and listed in Table 2.2. Table 2.2 also lists the sampling frequency at each point and the type of analysis to be performed for the present series of tests. Feed and glass samples are essential for verifying composition over the tests and, therefore, are taken relatively frequently. Melter exhaust samples will be taken to quantify the demands placed on the off-gas system. Select off-gas system sump materials will be analyzed as an indicator of potential secondary waste streams.

SECTION 3.0
WASTE SIMULANT AND GLASS FORMULATIONS

3.1 Waste Simulants

The waste stream compositions previously provided by DOE [4] are given in Table 3.1 on an oxide basis. The work described in this Test Plan will focus exclusively on the aluminum limited waste stream. Actual HLW Hanford tank wastes are aqueous solutions with suspended solids and dissolved salts including hydroxides, nitrates, nitrites, halides, and carbonates. For the purpose of the previous [3] and present work, the concentrations of the volatile components (i.e., carbonate, nitrite, nitrate, and organic carbon) are assumed to be similar to those found for the AZ-102 HLW waste [18]. With the waste compositions defined, formulation of the HLW waste simulant proceeds in a straightforward fashion. In general, oxides and hydroxides are used as the starting materials, with a slurry of iron (III) hydroxide (13% by weight) as one of the major constituents. Volatile inorganic components are added as the sodium salts, whereas organic carbon is added as oxalic acid. Although crucible melts will be prepared using the appropriate radioactive components (i.e., thorium and uranium), substitution of non-radioactive starting materials will be required in preparing the simulated waste for melter testing. The exact substitution to be made depends on the measured properties of the radioactive glass prepared in a crucible melt. Finally, the water content will be adjusted to target a glass yield of 500 g of glass per liter of feed. Two waste simulants will be employed, with the only difference being the replacement of aluminum hydroxide by boehmite in order to investigate the effects of variations in the form of aluminum in the Hanford HLW streams on feed properties and processing rates. The compositions of the waste simulants with boehmite and aluminum hydroxide formulated to produce 100 kg of waste oxides are given in Tables 3.2 and 3.3, respectively.

3.2 HWI-Al-19 Glass and Melter Feed Formulation

The HWI-Al-19 glass formulation for the ORP-directed high aluminum waste composition [6] was developed and tested on both the DM100 and DM1200 to determine processing rates [3]. These tests demonstrated that the formulation exceeded WTP requirements with respect to glass production rate and processed at a faster rate than the previous formulation (HLW-E-Al-27 [2]) with the same waste while maintaining the 45 wt% waste loading.

The composition and properties of the HWI-Al-19 formulation are listed in Table 3.4 and the melter feed composition with $Al(OH)_3$ as the aluminum source is shown in Table 3.5. Based on the results from small-scale melt rate testing, the formulation emphasized increased boron concentrations to improve melt rates and compensating changes to maintain other glass properties in acceptable ranges. The additional constituents required to form the target test glass from the HLW high aluminum waste simulant are boron, calcium, lithium, sodium, and silicon. The corresponding chemical additives that are the sources for these elements were selected based on previous testing and the current baseline chemicals for the WTP Project. The measured viscosity and conductivity of HWI-Al-19 at 1150°C are 33 P and 0.27 S/cm, respectively. No crystalline phases were observed in the as-melted sample, and heat treatment for 72 hours at 950°C resulted in 1.3 vol% crystals. Chemical durability was verified on crucible and product melter glasses with leachate concentrations well below regulatory limits [3].

Melter feeds will be produced by NOAH Technologies Corporation, the supplier of simulant and feed samples used in previous testing on the DM100 and DM1200 melter systems. An alkali iodide salt will be added to the feed corresponding to 0.1 wt% in the glass product in order to monitor iodine retention in the glass throughout these tests. Reductants and additional water to achieve the target glass yield will be added to the feed at VSL.

3.3 LAW Waste Stream as a Sodium Additive

3.3.1 Composition of LAW Waste Stream

The additive sodium carbonate in the HWI-AL-19 feed composition will be replaced by a simulated LAW waste in several of the DM100 and all of the DM1200 melter tests. The composition of the LAW waste stream provided by DOE for use in this work is listed in Table 3.6. Also given is the contribution to the feed and product glass from the LAW waste stream as a consequence of substituting the 6 wt% Na_2O in 100 kg product glass with the equivalent amount of sodium in the LAW waste. Changes to the glass composition total less than one absolute weight percent; however, the LAW stream contributes 0.61 wt% Al_2O_3, 0.16 P_2O_5, and 0.13 wt% SO_3 to the final glass composition if the 45 wt% HLW waste loading is retained. Crucible melts of glass compositions with the added LAW constituents at the 45% HLW waste loadings will be evaluated to ensure the glass still has suitable properties and does not form excessive amounts of secondary phases. If the glass properties are not found to be acceptable, the glass will be reformulated accordingly using lower HLW waste loadings if necessary. The resulting feed will also be evaluated for rheological properties to ensure that the feed containing the added reductants and LAW stream can be processed.

3.3.2 Reductant Additions

With high nitrate feeds, the addition of reductants is necessary in order to control melt foaming. Sugar, which was used for this purpose at West Valley, has also been selected as the baseline reductant for the WTP. The amount of sugar required increases with the amount of nitrates present in the feed and decreases with the amount of waste organics present in the feed, which themselves act as reductants. Excessive additions of reductants can be deleterious, leading to over-reduction of the melt and formation of sulfides and molten metals. Melter test in which the the iron redox state was monitored have shown that carbon additions greater than the targeted amount created overly reducing conditions in the glass [25-28]. Consequently, the oxidants and reductants in the feed must be suitably balanced. The basis for achieving this balance was developed by VSL and Energy*Solutions* for the vitrification of high-sodium-nitrate feeds at Savannah River's M-Area and has been successfully applied to the processing of a wide variety of simulated WTP feeds over the past six years [25-52]. In developing this approach, we elected to conservatively adopt the most reducing potential reaction as the basis for the *definition* of a "sugar" or stoichiometric ratio of 1.0 as a result of concerns for over-reducing the melt. Such a reaction, using sodium salts as an example, is:

$$C_{12}H_{22}O_{11} + 8NaNO_3 = 8CO_2 + 4CO + 4N_2 + 11H_2O + 4Na_2O$$

Fundamentally, the basis that is selected is simply a convention, since the precise stoichiometry of the reactions involved is neither known nor constant under the conditions prevailing in the melter. However, with this convention, a sugar ratio of 1.0 corresponds to one mole of sucrose per eight moles of nitrate or, more generally, 1.5 moles of organic carbon per mole of nitrate. It is then expected that significantly less sugar than this will be required in practice. The empirically determined amount required to successfully control melt foaming without significantly reducing the glass melt was found to correspond to a ratio of 0.5 when any nitrites present were counted as nitrates (i.e., 0.75 moles of organic carbon per mole of nitrate + nitrite). This approach has been employed for all WTP melter testing. It is, however, expected that slight variations around the nominal value of 0.5 may be necessary to account for differences in the reducing power of waste organics in comparison to sugar, particularly for LAW streams that are high in organics [27]. Testing with starch in place of sugar has shown that, like waste organics, starch has less reducing power than sugar [28].

The calculation of the amount of sugar needed for the melter feed with the LAW waste stream added to achieve a sugar ratio of 0.5 proceeds as follows:

- The amount of LAW waste stream required to produce 100 kg of product glass contains 9.1 moles of nitrite and 34.7 moles of nitrate, giving a total of 43.8 moles of NOx (see Table 3.6)
- The amount of HLW waste required to produce 100 kg of product glass contains 5.0 moles of nitrite and 14.6 moles of nitrate, giving a total of 19.6 moles of NOx (see Table 3.5)
- The total amount of NOx in the HLW feed associated with the production of 100 kg glass is 43.8 + 19.6 = 63.4 moles NOx.
- The required total amount of organic carbon for a sugar ratio of 0.5 is $63.4 \times 0.75 = 47.5$ moles
- The amount of HLW waste required to produce 100 kg of product glass contains only 0.03 moles of organic carbon and therefore can be neglected (see Table 3.5)

Since the molecular weight of sucrose is 342 g, $47.5 \times 342/12 = 1354$ g sugar must be added to the feed for the production of 100 kg glass. The amount of cellulose required can be calculated in the same manner in proportion to the amount of carbon in the particular form of cellulose that is used; however, the amount used may be increased, if necessary, based on redox state measurements on the glass product if they indicate a lesser reducing power for cellulose.

3.4 Development of New Glass and Melter Feed Formulation

An objective of this work is to develop and evaluate a new HLW glass composition with a waste loading of about 45% or higher, and faster melter processing rate than previously achieved [3] for the ORP specified high aluminum waste stream [6]. The new glass must also have acceptable durability and processing characteristics. The approach will employ small-scale tests and crucible melts to identify improved glass and feed formulations, beginning with and building upon the high waste loading formulation previously developed and given in Table 3.4 [3].

The glass formulation will be actively designed in that characterization data from the preceding set of crucible melts will be used to design the next set of formulations. This is an iterative approach in which the glass scientist is actively involved in the design of each glass formulation; i.e., one glass is made and characterized, and the results are used to refine the next formulation and so on. Each new formulation is obtained by varying the waste and glass former additive concentrations. This approach allows the glass scientists to use their knowledge, experience, and skills to the fullest extent in the development of new compositions with desired properties. A different approach is to use a "statistically-designed" matrix of glasses wherein a statistician (with input from the glass scientist) designs a matrix of compositions to be tested, followed by preparation and characterization of the glasses in the test matrix. This approach although used extensively, is inefficient if the objective is to develop a glass formulation with specific properties. The statistically-designed matrix approach is useful if the objective is to fill a composition space and is, therefore, better suited for property-composition model development.

Characterization of the melts will include composition, PCT, TCLP, viscosity, electrical conductivity, and liquidus temperature (crystal content). Not all crucible melts will be analyzed for all properties. The glass and feed formulations will also be subjected to small-scale screening tests to evaluate relative melt rates at the crucible scale to evaluate melt/feed interface conditions and project processing rate enhancements. The crucible tests employ heating of dried feed samples at various temperatures for fixed times followed by examination of the extent of feed conversion as a function of temperature; a variant of this test employs dried feed on top of premelted glass that is heated in a vertical gradient furnace. Once a glass and feed composition with sufficient waste loading and comparatively high melt rate (based on the screening tests) is identified, it will be characterized for all relevant properties including composition, PCT, TCLP, viscosity, electrical conductivity, and crystal content. Based on these results, glass and feed formulations for the high aluminum waste composition specified by ORP will be selected for DM100 melter testing. All of the properties listed in Table 3.7 will be measured for the glass composition selected for melter testing to ensure that they meet all processing and product quality requirements. Approximately eight crucible melts will be prepared and characterized to support the above.

After the glass composition for further testing is selected, melter feed will be prepared and characterized with respect to physical properties to ensure that they are suitable for processing in the DM100 melter.

The main variables from a glass formulation perspective are additives (glass formers and others such as reductants). The glass former additives used in WTP glass formulations include Al_2O_3, B_2O_3, CaO, Li_2O, MgO, Na_2O, SiO_2, ZnO, and ZrO_2. The WTP HLW glass formulations use only Al_2O_3, B_2O_3, Li_2O, Na_2O, SiO_2, and ZnO. One of the ORP high-Al glass compositions from the Phase 1 work contains CaO as an additive while the others do not. Al_2O_3 will not be used as an additive in this study because the waste stream to be used in these studies has a high concentration of Al_2O_3.

Since lower viscosity glass compositions tend to process faster [22, 23], the glass formulations will be formulated to have relatively low viscosities within the acceptable range. This will require formulations with lower concentrations of components such as SiO_2 and ZrO_2 and higher concentrations of alkali oxides and B_2O_3. The viscosities of the Phase 1 high-Al ORP glasses are 46 and 60 poise at 1150°C [2] and there is, therefore, some room for decreasing it to about 25 – 30 poise. However, whether a reduction in the viscosity will lead to unacceptable K-3 corrosion or crystallization can be determined only through testing. The viscosity of the HWI-Al-19 glass previously tested was 33 poise and it still had acceptable K-3 corrosion rates and a limited tendency toward crystallization [3]. As always, property composition models will be used to guide glass formulation development. However, since the high-Al glasses are outside the valid composition region for the existing models, the viscosity predictions are not likely to be very reliable. Based on initial crucible test results, the concentrations of glass former oxides will be adjusted to obtain glasses with higher alkali and B_2O_3 concentrations while maintaining acceptable processing and product properties. The properties of greatest concern when moving in this compositional direction are nepheline formation during heat treatment and K-3 refractory corrosion. Excessive crystallization of alkali aluminosilicates on heat treatment can lead to unacceptable product performance on PCT. Other factors that will be considered include the particle size of the glass forming additives, understanding that changes here also impact feed rheology; gas generation on decomposition; and adequate fluxing to enhance feed conversion rates. In addition, we believe that melt/feed interface conditions (such as the formation of refractory intermediate phases, gas retention/release in the cold-cap, etc.) will also be important factors in controlling melt rate. Consequently, study of the interface conditions will be considered as part of glass formulation development. At present, it is not anticipated that any additives beyond those listed above warrant testing; however, any recommendation to use other additives for melter testing will be supported by crucible scale test results.

SECTION 4.0
MELTER TEST MATRIX

4.1 DM100 Tests

4.1.1 General Approach

The DM100 test matrix is shown in Table 4.1. Sufficient blended feed (glass formers plus waste simulant) will be prepared by NOAH Technologies Corporation according to VSL specifications to make approximately three and a half metric tons of glass. The DM100-BL melter will be used in order to provide a direct comparison with the HLW tests previously conducted on the same melter [2, 3, 10-14, 21-23]. Two 50-hour melter tests will be performed for each of the six HLW feed compositions (five with the previously processed glass composition, HWI-Al-19, using previously untested waste aluminum and additive sodium sources and one with new glass composition) to be tested; the first 50-hr segment will be performed at the elevated glass pool operating temperature of 1200°C and the second will be performed at the nominal glass pool temperature of 1150°C. Most tests will be conducted at a nominal glass yield of 500 g per liter and a bubbling rate of 9 lpm to facilitate comparison with other tests conducted with other compositions. Feed using the LAW waste stream will also be tested at a glass yield of 400 g glass per liter to evaluate the effect on processing rates of feed dilution as a result of using the LAW waste stream as a sodium source.

The new glass formulations for the aluminum rich waste composition will be selected based on the results from small-scale testing. The melter tests with the new glass composition are of sufficient duration to ensure that the glass pool is turned over to the desired glass composition and to obtain steady-state conditions with respect to both glass production rates and partitioning between the glass product and the melter exhaust. Since the composition of melt pool prior to testing is at the target composition and there are little or no differences in the glass compositions for the other tests, the glass level will not be lowered to minimum levels prior to the other tests. Key operating parameters will be held constant to investigate the effect of the new feed compositions on the processing characteristics, including formation of secondary phases, processing rate, and relative volatility of feed constituents. The feed rate will be adjusted to achieve a near-complete cold-cap (90-100% of melt surface covered with feed). The glass production and emissions rates for each test will be compared to those achieved in previous DM100 tests at the same bubbling rate. Quantitative measurements of glass production rates, melter operating conditions (temperatures, pressures, power, flows, etc.), and off-gas characteristics (NO_X, SO_2, CO, particulate load and composition, and acid gases) will be made for each test. Discharged glass samples will be inspected for secondary phases and analyzed for properties listed in Table 3.7 including chemical composition and durability.

4.1.2 DM100 Test Parameters

Glass Temperature: The first and second 50-hour melter tests for each formulation will be at nominal glass pool temperatures of 1200°C and 1150°C, respectively.

Bubbling Rate: The glass pool is bubbled with air to facilitate mixing and increase melting rates. The bubbling rate will be held constant at 9 lpm throughout testing to facilitate comparison between tests with different glass compositions.

Feed Solids Content: For most of the tests, the glass yield will be held constant at approximately 500 g per liter to facilitate comparison with previous tests. Additional tests will be conducted at 400 g glass per liter for feed using the LAW waste stream as an additive source.

Waste Composition: Tests will be conducted with the Al-limited HLW waste composition specified by ORP.

Aluminum Source: One test will be conducted with boehmite in place of aluminum hydroxide for comparison to previously conducted tests with the same glass composition (HWI-Al-19). Subsequent tests with the new formulation will be conducted with aluminum hydroxide.

Sodium Source: Four of the tests will be conducted with a LAW waste stream in place of sodium carbonate as a source for the sodium additive. For comparison to previously conducted tests, the same glass composition (HWI-Al-19) will be used.

Glass Composition: Tests will be conducted with two base glass compositions: one selected based on the results from crucible testing and one using a formulation (HWI-Al-19) previously tested substituting either boehmite for aluminum hydroxide as the aluminum source or the LAW waste stream for sodium carbonate as an additive for sodium.

Cold Cap Coverage: The extent of the melt surface that is covered with reacting feed (the cold-cap) is a measure of the extent of processing capacity utilization. Thus, 100% cold-cap corresponds to the maximum sustainable feed rate under the given set of operating conditions. Essentially complete cold-caps will be visually maintained in all the tests.

Feed Rate: The feed rate, and therefore glass production rate, will be dependent on the feed composition and melter operating conditions. The feed rate will be varied to obtain a near-complete cold cap for each test segment.

Plenum Gas Temperature: Plenum heaters will not be used for these tests. The plenum gas temperature will be targeted in the range of 450-650°C, subject to the other constraints of these tests. The plenum gas temperature is largely determined by the cold-cap coverage, which, in turn, is controlled by feed rate and bubbling rate.

4.2 DM1200 Tests

4.2.1 General Approach

One of the reductants tested on the DM100 with feed using the LAW waste stream as a sodium additive will be selected in consultation with ORP for an eight-day test on the DM1200. Sufficient blended feed (glass formers plus waste simulant) will be prepared by NOAH Technologies Corporation according to VSL specifications to make approximately twelve metric tons of glass. The DM1200 melter at VSL will be used in order to provide a direct comparison with the HLW tests previously conducted on the same melter [3, 7, 13-20]. Four two-day melter tests will be performed: the first two at the feed solids content of 500 g glass per liter and the second two at 400 g glass per liter. For feed with each of the two solids contents, the highest achievable production rate will be targeted over the first two days by optimizing the bubbling rate, followed by another two-day period during which the bubbling rate will be adjusted to target a production rate of 1050 kg/m^2/day. All tests will be conducted at the nominal glass pool temperature of 1150°C. This approach was used in previous tests with HLW AZ-102, C-106/AY-102, and AZ-101 simulants [7, 20] as well as with the same glass composition using sodium carbonate as the sodium additive source [3]. Comparison of bubbling rates required to achieve this rate are an indicator of the ease of melting each HLW feed composition. The production rate of 1050 kg/m^2/day was selected based on the previous requirement of 3 MT/day for the WTP HLW melter and a scaling factor to account for differences in the number of bubbling outlets per unit area in the DM1200 and the WTP HLW melter. The current glass production requirement for the WTP HLW melter has increased to 3.75 MT/day along with the inclusion of more bubblers in the melter (seven vs. five). However, since the bubbler density in the new WTP HLW melter is now higher than that in the DM1200, the normalized glass production rate target *decreases* to 900 kg/m^2/day, even though the requirement for the WTP HLW melter increased from 3.00 to 3.75 kg/m^2/day. In the present work, we retain the higher glass production rate target of 1050 kg/m^2/d since it provides comparison to previous tests and adds a measure of conservatism.

The bubbling configuration that will be employed in these tests (see Section 2.2.2 and Figure 2.6) was optimized in the latter part of the WTP DM1200 testing activities and, therefore, only a limited amount of data with this optimized bubbling configuration are available. A listing of these tests together with the achieved production rates and amount of bubbling used are provided in Table 4.2 [3, 7, 20, 53, 54]. The data with production rates close to 1050 $kg/m^2/day$ show the trend for ease of melting, starting with fastest melting iron-limited HLW compositions as follows: spike C-106/AY-102 [54], AZ-101 [7], high waste loading C-106/AY-102 [20], and AZ-102 [20]. The highest production rate was obtained with the high aluminum waste that will be used in these tests; however, the higher production rate is partially attributable to the higher feed solids content. Melter tests will be of sufficient duration to obtain steady-state conditions with respect to both glass production rates and partitioning between the glass product and the melter exhaust. The target glass composition (HWI-Al-19) remains in the melt pool from the previous tests [3] therefore no melt pool turnover will be required prior to collecting relevant data. The feed rate and bubbling rate will be adjusted to achieve a near-complete cold-cap (90-100% of melt surface covered with feed). Quantitative measurements of glass production rates, melter operating conditions (temperatures, pressures, power, flows, etc.), and off-gas characteristics (NO_X, SO_2, CO, particulate load and composition, and acid gases) will be made for each test. Discharged glass samples will be inspected for secondary phases and analyzed for chemical composition and durability.

SECTION 5.0
SAMPLE AND DATA COLLECTION

5.1 Melter System Operations Data

The conditions prevailing in the vitrification system during these tests will be characterized by a variety of temperatures, pressures, flow rates, voltages, currents, and other data as well as visual observations that will be recorded throughout the tests. The majority of these values will be both electronically monitored and stored at 2-minute intervals and written into the melter laboratory notebook on an hourly basis. A listing of these parameters for DM1200 tests is provided in Table 5.1. Visual observations will be manually recorded in the laboratory notebook. The presence of secondary phases will be determined by visual observations and microscopic analysis of dip and discharge glass samples.

The glass production rate during melter tests will be calculated using the weight of feed used along with the corresponding glass yield, and separately based on the weight of glass discharged from the melter over the course of the melter test. The data used for this calculation will correspond to the steady-state cold-cap limited portion of each test segment.

5.2 Feed Characterization

5.2.1 Vertical Gradient Furnace Testing

Feed samples that have been dried at 150°C are placed on the surface of molten glass contained in a clay crucible preheated to 1150°C. The loaded crucible is then placed in a two zone furnace with the molten glass temperature maintained at 1150°C and the ambient air temperature maintained at 600°C. The conversion of feed to glass is monitored in real time by a video camera interfaced to a PC. The cross sections of the feed pile undergoing reaction at various stages (different test durations) are analyzed to estimate a batch free time equivalent. Results from new feed formulations are compared to results obtained from feed samples with known DM100 processing rates.

5.2.2 Feed Samples from Melter Tests

During DM100 and DM1200 melter tests, feed samples will be taken directly from the feed recirculation line during each test. Feed samples will be poured into a platinum/platinum alloy crucible that will be placed into a programmed furnace for drying and fusion to form a glass. The glass produced from this fusion will be ground to less than 200 mesh and sealed in 20-ml vials for subsequent analysis by x-ray fluorescence spectroscopy, or by acid digestion followed by direct-current plasma emission spectroscopy on the resulting solution. The feed samples will also be characterized for their rheological properties, density, pH, water content, and glass yield.

5.3 Glass Product

The glass product is discharged from the melter into 5-gallon steel pails and 55 gallon drums periodically using the DM100 and DM1200 air-lift systems. The discharged product glass will be sampled at the end of each test by removing sufficient glass from the top of the cans for compositional analysis, iron redox state, and secondary phase determinations. In addition, the Product Consistency Test (PCT; 7 days at 90°C) and Toxicity Characteristic Leaching Procedure (TCLP) will be performed on samples of the glass product from the DM100 melter tests. Prior to those tests, the PCT and TCLP will also be performed on the crucible melt compositions that are selected for the melter tests to ensure their compliance with the present WTP contract requirements. All of these procedures are routinely conducted at VSL and, therefore, standard operating procedures (SOPs) are in place.

Sample preparation for chemical analysis typically involves size reduction and sieving. All samples will be subjected to x-ray fluorescence spectroscopy (XRF) to determine the concentration of all elements except boron and lithium. A series of NIST reference materials will be used for confirmation of the XRF data. Boron and lithium will be determined by total acid dissolution of ground glass samples in HF/HNO_3 and subjecting the resulting solutions to direct current plasma atomic emission spectroscopy (DCP-AES) analysis.

5.3.1 Viscosity

The melt viscosity, η, is measured using a Brookfield viscometer. Measurements are performed in the temperature range of 950-1250°C and the data are interpolated to standard temperatures using the Vogel-Fulcher equation: $\ln \eta = [A/(T\text{-}T_0)]+B$, where A, B, and T_0 are fitting parameters. The equipment is calibrated at room temperature using standard oils of known viscosity and then checked at 950-1250°C using a National Institute of Standards and Technology (NIST) standard reference glass (SRM 711). Both precision and accuracy of the viscosity measurements are estimated to be within ±15 relative%.

5.3.2 Electrical Conductivity

The electrical conductivity, σ, of each glass is determined by measuring the resistance of the glass melts as a function of frequency using a calibrated platinum/rhodium electrode probe attached to a Hewlett-Packard model 4194A impedance analyzer. Measurements are performed over similar temperature ranges to those employed for the melt viscosity measurements. The results are analyzed to obtain the DC electrical conductivity. The electrical conductivity data are then interpolated to standard temperatures using the Vogel-Fulcher equation: $\ln \sigma = [A/(T-T_0)] + B$, where A, B and T_0 are fitting parameters. Estimated uncertainties in the electrical conductivity measurements are ± 20 relative%.

5.3.3 Product Consistency Test (PCT)

The product consistency test (PCT; ASTM C 1285) is used to evaluate the relative chemical durability of glasses by measuring the concentrations of the chemical species released from 100-200 mesh crushed glass (75-149 μm) to the test solution (de-ionized water in this case). PCT tests on the HLW glasses are performed at 90°C, in accordance with the current WTP contract requirement. The ratio of the glass surface area to the solution volume for this test is about 2000 m^{-1} (typically, 10 g of 100-200 mesh glass is immersed in 100 ml of deionized water). All tests are conducted in triplicate, in 304L stainless steel vessels, and in parallel with a standard glass included in each test set. The internal standard is the ANL-LRM reference glass [55] and/or the DWPF-EA glass, both of which have undergone round-robin testing. The leachates are sampled at predetermined times, the first of which is seven days. One milliliter of sampled leachate is mixed with 20 ml of 1M HNO_3 and the resulting solution is analyzed by DCP-AES; another 3 ml of sampled leachate is used for pH measurement.

5.3.4 Toxicity Characteristic Leaching Procedure (TCLP)

The TCLP will be performed at VSL using SW-846 Method 1311 that employs leaching of crushed glass (< 3/8") in a sodium acetate buffer solution for 18 hours at 22°C with constant end-over-end agitation. A mass of about 100 grams of glass is leached in 2 liters of TCLP extract, according to the extraction method for non-volatiles. The surface area to volume ratio for this test is about 20 m^{-1}, which is about two orders of magnitude lower than that in the PCT. The leachates are analyzed by DCP-AES according to VSL standard operating procedures.

5.3.5 Secondary Phases

Secondary phases in the glass samples will be determined by optical microscopy and scanning electron microscopy coupled with energy dispersive x-ray spectroscopy (SEM-EDS). Secondary phases due to crystallization and phase separation can be identified using these methods. Quantitative determination of the amount of crystals in glass samples will be made by SEM in conjunction with image analysis. Images of the secondary phases will be included in the reports, as appropriate.

5.4 Emission Samples

Melter emission fluxes will be measured to complete the mass balance for each melter test. Isokinetic melter exhaust samples (exhaust gas flow velocity equal to velocity through the gas sample probe tip) will be combined with the FTIR continuous monitoring data for gaseous species to characterize fluxes from the melter. In the DM100 system, the off-gas sampling port used for isokinetic sampling is located prior to filtration in the transition line between the two temperature monitors, whereas the sample for FTIR analysis is pulled after HEPA filtration prior to any dilution. In the DM1200 system, independent sampling ports for particulate and FTIR sampling are available throughout the off–gas treatment train (see Figure 2.7). Standard EPA isokinetic off-gas sampling trains and methods (EPA Methods 1A, 2, 4, 5, 26, 29), composed of particulate filters and liquid impingers, will be used to collect materials that will be subjected to chemical and physical analyses using the techniques described in Sections 5.2 and 5.3.

5.5 DM1200 Off-Gas System Sump Solutions

Sump solutions from the SBS, WESP, HEMEs, and PBS will be taken throughout testing to verify material balances and unit efficiency. Total suspended solids (TSS) and total dissolved solids (TDS) will be determined on select samples using standard ASTM methods. The filtered solids and liquids derived from this procedure will be analyzed to determine the total inorganic composition using methods similar to those used in glass and feed analysis. Anion concentrations will be determined by ion chromatography and ion selective electrode analyses on filtered solutions. Select sump samples may also be characterized for pH and density.

SECTION 6.0
SCHEDULE AND REPORTING

The schedule is given below.

Issue Test Plan for ORP review	3/30/09
Resolve ORP comments and reissue Test Plan	5/05/09
ORP approval of Test Plan	5/07/09
Glass formulation development	5/08/09-7/10/09
DM100 Melter Tests 1 and 2	6/01/09-6/05/09
DM100 Melter Tests 3 and 4	6/08/09-6/12/09
DM100 Melter Tests 5 and 6	6/15/09-6/19/09
DM100 Melter Tests 7 and 8	6/22/09-6/26/09
DM100 Melter Tests 9 and 10	7/06/09-7/10/09
Submit recommendation for DM1200 testing to ORP	7/15/09
ORP review of recommendation for DM1200 testing	7/16/09-7/17/09
DM1200 Melter Test	7/20/09-7/31/09
Submit crucible data to ORP for DM100 Tests 11 and 12	7/08/09
ORP review of crucible data for DM100 Tests 11 and 12	7/09/09-7/10/09
DM100 Melter Tests 11 and 12	8/03/09-8/07/09
Issue final report (draft) for ORP review	10/22/09
ORP review of final report (draft)	10/23/09-11/12/09
Resolve ORP comments and revise report	11/13/09-12/10/09
Submit final report to ORP	12/11/09

The final report will include:

- Discussion of results, conclusions, and recommendations for future tests.
- Results of glass formulation development and testing.
- Discussion of any difficulties encountered during the melter tests.
- All feed and glass formulations used in the tests.
- List of samples, providing the date/time of sampling, sample type, and analysis performed on the sample.
- Sample analysis results and glass production rates.
- Data tables of monitored parameters.
- Comparison of crucible and melter glass properties.
- Photographic records of any unique glass properties
- DM1200 and DM100 melter operating performance.

SECTION 7.0
QUALITY ASSURANCE

This work will be conducted under a quality assurance program that is based on NQA-1 (2000) and NQA-2a (1990) Part 2.7 and which is compliant with applicable criteria of 10 CFR 830.120; Office of Civilian Waste Management DOE/RW-0333P, Quality Assurance Requirements and Description (QARD) Revision 20; and DOE Order 414.1 C, Quality Assurance. This program is supplemented by a Quality Assurance Project Plan for RPP-WTP work [56] that is conducted at VSL. Test and procedure requirements by which the testing activities are planned and controlled are also defined in this plan. The program is supported by VSL standard operating procedures that will be used for this work [57]. The requirements of DOE/RW-0333P are applicable to the following specific aspects of this work:

- Crucible melt preparation
- Analysis of crucible melt glasses
- PCT
- Glass transition temperature measurement.

SECTION 8.0
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Table 2.1. DM100 Sampling Points, Types, and Frequencies.

Source	Nature of Samples	Potential Analysis	Number of Samples and Justification
Feed sample from feed tank recirculation line	Slurry converted into glass via crucible melt	Total inorganic composition, water content, density and pH	6: 1 per 100-hr hour test to verify composition
Glass Pool	Glass sample obtained by dipping a rod into the melt pool	Composition and crystal content at glass melt temperatures	12: 1 at the beginning and end of each 100-hour test.
Discharged glass	Glass	Total inorganic composition, secondary phases, and chemical durability.	140: 1 per 25 kg of glass production 6: 1 from the end of each feed composition product quality (PCT, TCLP)
		Samples for ORP use	6: 3 "baseball" size samples of glass collected from the end of each Tests 2 and 12.
Melter emissions	Filters, impinger and rinse solutions	Total inorganic composition, and particle loading	12: 1 per 50-hour test segment once steady state has been attained

Table 2.2. DM1200 Sampling Points (see Figure 2.7), Types, and Frequencies.

ID	Source	Nature of Samples	Analysis	Number of Samples and Justification
S1	Feed line sample	Slurry converted into glass via crucible melt	Total inorganic composition, water content, pH and density	Sample: 1 per day and one per feed batch received to verify composition. Analyze: 1 per test and 1 per feed batch.
S2	Discharged glass	Glass	Total inorganic composition, secondary phases	Sample: 1 per drum. Analyze: Every sample with XRF, others as needed. Samples for ORP use: 3 "baseball" size samples of glass collected from test end
S3	Melter emissions	Filters, impinger, and rinse solutions	Total inorganic composition, particle loading , and gaseous species (CEM)	Four: 1 per test segment.
S4	Submerged bed scrubber sump	Solutions and suspended solids	Total inorganic composition and pH	1 sample taken near end of testing for complete analysis.
S5	Submerged bed scrubber emissions	CEM	Gaseous species (CEM)	One measurement set during each test segment.
S6	Wet electrostatic precipitator sump	Solutions and suspended solids	Total inorganic composition and pH	1 pair of samples (pre and post deluge) taken near end of testing for complete analysis.
S7	Wet electrostatic precipitator emissions	CEM	Gaseous species (CEM)	One measurement set during each test segment.
S8	HEME #1 sump	Solutions and suspended solids	Total inorganic composition and pH	1 sample taken near end of testing for complete analysis.
S9	TCO/SCR emissions	CEM	Gaseous species (CEM)	One measurement set during each test segment.
S10	Packed bed scrubber sump	Solutions and suspended solids	pH	As needed for operations.
S11	HEME #2 sump	Solutions and suspended solids	pH	As needed for operations.
S12	HEME #2 emissions	Not part of HLW treatment train	Not part of HLW treatment train	No sampling performed
S13	Final stack emissions	Filters, impinger, and rinse solutions	Total inorganic composition, particle loading, and gaseous species (CEM)	As specified by local regulatory authority

Table 3.1. Oxide Composition of Limiting Waste Streams. (Wt%)

Waste Component	Bi Limited	Cr Limited	Al Limited	Al and Na Limited
Al_2O_3	22.45	25.53	49.21	43.30
B_2O_3	0.58	0.53	0.39	0.74
CaO	1.61	2.47	2.21	1.47
Fe_2O_3	13.40	13.13	12.11	5.71
Li_2O	0.31	0.36	0.35	0.15
MgO	0.82	0.16	0.24	0.44
Na_2O	12.97	20.09	7.35	25.79
SiO_2	12.04	10.56	10.05	6.22
TiO_2	0.30	0.01	0.02	0.35
ZnO	0.31	0.25	0.17	0.36
ZrO_2	0.40	0.11	0.81	0.25
SO_3	0.91	1.52	0.41	0.44
Bi_2O_3	12.91	7.29	2.35	2.35
ThO_2	0.25	0.04	0.37	0.04
Cr_2O_3	1.00	3.07	1.07	1.44
K_2O	0.89	0.37	0.29	1.34
U_3O_8	3.48	7.59	7.25	4.58
BaO	0.02	0.03	0.11	0.06
CdO	0.00	0.01	0.05	0.02
NiO	3.71	1.06	0.82	0.20
PbO	0.48	0.48	0.84	0.18
P_2O_5	9.60	3.34	2.16	4.10
F-	1.58	2.00	1.37	0.46
Total	100.0	100.0	100.0	100.0

Table 3.2. Compositions of the Al-Limited Waste (Oxide Basis) and the HLW Waste Simulant to Produce 100 kg of Waste Oxides (20 wt% suspended solids) Using Boehmite as the Aluminum Source.

Al-Limited Waste Composition		Al-Limited HLW Waste Simulant	
Waste Oxide	**Wt%**	**Starting Materials**	**Target Weight (kg)** *
Al_2O_3	49.21	Boehmite, AlO(OH)	64.179
B_2O_3	0.39	H_3BO_3	0.757
CaO	2.21	CaO	2.441
Fe_2O_3	12.11	$Fe(OH)_3$ (13% Slurry)	107.864
Li_2O	0.35	Li_2CO_3	0.961
MgO	0.24	MgO	0.273
Na_2O	7.35	NaOH	4.867
SiO_2	10.05	SiO_2	10.989
TiO_2	0.02	TiO_2	0.022
ZnO	0.17	ZnO	0.186
ZrO_2	0.81	$Zr(OH)_4{\cdot}xH_2O$	2.266
SO_3	0.41	Na_2SO_4	0.796
Bi_2O_3	2.35	Bi_2O_3	2.570
ThO_2	0.37	Th Surrogate	Not Used
Cr_2O_3	1.07	Cr_2O_3	1.182
K_2O	0.29	KNO_3	0.684
U_3O_8	7.25	U Surrogate	Not Used
BaO	0.11	$BaCO_3$	0.155
CdO	0.05	CdO	0.055
NiO	0.82	$Ni(OH)_2$	1.142
PbO	0.84	PbO	0.918
P_2O_5	2.16	$FePO_4{\cdot}xH_2O$	6.211
F	1.37	NaF	3.295
Carbonate	1.20#	Na_2CO_3	0.697
Nitrite	0.50#	$NaNO_2$	0.769
Nitrate	2.00#	$NaNO_3$	2.186
Organic Carbon	0.05#	$H_2C_2O_4{\cdot}2H_2O$	0.276
—	—	Water	339.820
—	—	—	—
TOTAL	100.0	TOTAL	555.561

* Target weights adjusted for assay information of starting materials
# Unit for volatile components is g/100 g of waste oxide
— Empty data field

Table 3.3. Compositions of the Al-Limited Waste (Oxide Basis) and the HLW Waste Simulant to Produce 100 kg of Waste Oxides (20 wt% suspended solids) Using $Al(OH)_3$ as the Aluminum Source.

Al-Limited Waste Composition		Al-Limited HLW Waste Simulant	
Waste Oxide	**Wt%**	**Starting Materials**	**Target Weight (kg)** *
Al_2O_3	49.21	$Al(OH)_3$	76.052
B_2O_3	0.39	H_3BO_3	0.700
CaO	2.21	CaO	2.255
Fe_2O_3	12.11	$Fe(OH)_3$ (13% Slurry)	99.643
Li_2O	0.35	Li_2CO_3	0.888
MgO	0.24	MgO	0.253
Na_2O	7.35	NaOH	4.235
SiO_2	10.05	SiO_2	10.152
TiO_2	0.02	TiO_2	0.020
ZnO	0.17	ZnO	0.172
ZrO_2	0.81	$Zr(OH)_4{\cdot}xH_2O$	2.093
SO_3	0.41	Na_2SO_4	0.735
Bi_2O_3	2.35	Bi_2O_3	2.374
ThO_2	0.37	Th Surrogate	0
Cr_2O_3	1.07	$Cr_2O_3{\cdot}1.5H_2O$	1.273
K_2O	0.29	KNO_3	0.632
U_3O_8	7.25	U Surrogate	0
BaO	0.11	$BaCO_3$	0.143
CdO	0.05	CdO	0.051
NiO	0.82	$Ni(OH)_2$	1.055
PbO	0.84	PbO	0.848
P_2O_5	2.16	$FePO_4{\cdot}xH_2O$	5.738
F	1.37	NaF	3.044
Carbonate	1.20#	Na_2CO_3	0.806
Nitrite	0.50#	$NaNO_2$	0.769
Nitrate	2.00#	$NaNO_3$	2.230
Organic Carbon	0.05#	$H_2C_2O_4{\cdot}2H_2O$	0.264
—	—	Water	279.400
—	—	—	—
TOTAL	100.0	TOTAL	495.825

* Target weights adjusted for assay information of starting materials
# Unit for volatile components is g/100 g of waste oxide
— indicates empty data field

Table 3.4. Composition and Properties of Aluminum Limited Waste and Glass Formulation HWI-Al-19 with 45% Waste Loading (wt%).

-	Al-Limited Waste*	Waste in Glass	Glass Forming Additives	Target Glass HWI-Al-19
Al_2O_3	53.27	23.97	-	23.97
B_2O_3	0.42	0.19	19.00	19.19
BaO	0.12	0.05	-	0.05
Bi_2O_3	2.54	1.14	-	1.14
CaO	2.39	1.08	4.50	5.58
CdO	0.05	0.02	-	0.02
Cr_2O_3	1.16	0.52		0.52
F	1.48	0.67	-	0.67
Fe_2O_3	13.11	5.90	-	5.90
K_2O	0.31	0.14	-	0.14
Li_2O	0.38	0.17	3.40	3.57
MgO	0.26	0.12	-	0.12
Na_2O	7.96	3.58	6.00	9.58
NiO	0.89	0.40	-	0.40
P_2O_5	2.34	1.05	-	1.05
PbO	0.91	0.41	-	0.41
SO_3	0.44	0.20	-	0.20
SiO_2	10.88	4.90	22.10	27.00
TiO_2	0.02	0.01	-	0.01
ZnO	0.18	0.08	-	0.08
ZrO_2	0.88	0.39	-	0.39
Sum	100.0	45.0	55.0	100.0

* Renormalized from Ref. [6] after removal of radioactive components.

Viscosity @1150°C, P			33
Conductivity @1150°C, S/cm			0.27
Crystal Content, As Melted			None
Crystal Content, 72 hr at 950°C			1.3
Crystal Content, CCC			1.9
TCLP			Pass
PCT, g/L	-	DWPF-EA	HWI-Al-19
	B	16.7	0.654
	Li	9.6	0.794
	Na	13.3	0.624

- Empty data field

Table 3.5. Composition of Melter Feed to Produce 100 kg of Target Glass HWI-Al-19 (Target Glass Yield = 500 g/L Feed) from the Al-Limited Waste Simulant Using $Al(OH)_3$ as the Aluminum Source.

Al-Limited Waste Simulant		Glass-Forming Additives	
Starting Materials	**Target Weight (kg)** *	**Starting Materials**	**Target Weight (kg)** *
$Al(OH)_3$	37.047	—	—
H_3BO_3	0.341	H_3BO_3	34.089
$BaCO_3$	0.070	—	—
Bi_2O_3	1.156	—	—
CaO	1.099	$CaSiO_3$ (Wollastonite)	9.798
CdO	0.025	—	—
Cr_2O_3	0.532	—	—
NaF	1.483	—	—
$Fe(OH)_3$ (13% Slurry)	48.539	—	—
KNO_3	0.308	—	—
Li_2CO_3	0.432	Li_2CO_3	8.625
MgO	0.121	—	—
NaOH	2.190	Na_2CO_3	10.364
$Ni(OH)_2$	0.514	—	—
$FePO_4{\cdot}xH_2O$	2.795	—	—
PbO	0.413	—	—
Na_2SO_4	0.358	—	—
SiO_2	4.945	SiO_2	17.276
TiO_2	0.010	—	—
ZnO	0.084	—	—
$Zr(OH)_4{\cdot}xH_2O$	1.020	—	—
H_2O	91.903	—	—
Na_2CO_3	0.314	—	—
$NaNO_2$	0.346	—	—
$NaNO_3$	0.984	—	—
$H_2C_2O_4{\cdot}2H_2O$	0.119	—	—
—[2]	—	—	—
Simulant Total	197.148	Additives Total	80.152
—	—	FEED TOTAL	277.300

* Target weights adjusted for assay information of starting materials
— Empty data field

Table 3.6. Composition of LAW Waste Stream and Contributions to Melter Feed and Product Glass.

UFP-VSL-00062A Contract Run UF Permeate Tank: 9,400 Data Points for Liquids [4]			Amount required to replace 6 wt% Na_2O in 100 kg glass		
Components	Units	Bulk Average	Moles	Grams	Oxides in Glass (wt%)
Na^+	mol/kg water	4.43	193.5	4452	Na_2O - 6.00
$Al(OH)_4$	mol/kg water	0.27	11.9	322	Al_2O_3 - 0.61
Na^+	mol/mol Al	16.23	-	-	-
^-OH	mol/mol Al	10.22	121.9	2072	-
NO_3	mol/mol Al	2.91	34.7	2152	-
$^-NO_2$	mol/mol Al	0.76	9.1	417	-
$^{-2}PO_4$	mol/mol Al	0.18	2.1	204	P_2O_5 - 0.15
$^{-2}SO_4$	mol/mol Al	0.14	1.7	160	SO_3 - 0.13
wt% water	%	79.7	-	43690	
Total				53469	6.89

- Empty data field

Table 3.7. Glass Processing and Product Quality Requirements.

Property	Requirement(s)
Volume % Crystals	< 1 Volume % at 950°C*
HLW Canister Centerline Cooling Heat Treatment	Report amount of crystals
PCT per ASTM C1285. Test conducted at glass to water ratio of 1 gram of glass (-100 +200 mesh) per 10 ml of water (90°C – 304L SS vessel)	B < 16.695 g/l Na < 13.346 g/l Li < 9.565 g/l Normalized mass loss less than that of reference glass DWPF-EA
TCLP per EPA Method SW-846-1311	Delisting Limits Ag < 3.07 mg/l As < 0.616 mg/l Ba < 100 mg/l Cd < 0.48 mg/l Cr < 4.95 mg/l Cu < 29200 mg/l Ni < 22.6 mg/l Pb < 5.00 mg/l Sb < 0.659 mg/l Se < 1.00 mg/l Tl < 0.282 mg/l Zn < 225 mg/l
Viscosity (poise) at 1100°C	10 to 150 P
Electrical Conductivity (S/cm) at 1100°C	0.2 to 0.7 S/cm
Glass Transition T_G (onset)	Report T_G

* In the present work, this requirement is relaxed in order to realize high loadings.

Table 4.1. DM100 Melter Test Matrix with Alminum Limited Waste.

Aluminum Source	Sodium Additive Source	Reductant	Feed Solids Content (g glass/liter)	Base Glass Formulation	Test	Glass Pool Temperature
Boehmite	Na_2CO_3	None	500	HWI-Al-19	1	1200°C
					2	1150°C
Hydroxide	LAW Waste Stream	Sugar	500	HWI-Al-19	3	1200°C
					4	1150°C
	LAW Waste Stream	Sugar	≈ 400	HWI-Al-19	5	1200°C
					6	1150°C
	LAW Waste Stream	Cellulose	500	HWI-Al-19	7	1200°C
					8	1150°C
	LAW Waste Stream	Cellulose	≈ 400	HWI-Al-19	9	1200°C
					10	1150°C
	Na_2CO_3	None	500	Optimized	11	1200°C
					12	1150°C

Table 4.2. DM1200 Tests Performed with Final HLW Bubbler Configuration.

Test	Feed	Glass Yield	Duration	Bubbling Rate	Glass Production Rate
Test 1 VSL-08R1360-1 [3]	Al-Limited Waste	500 g/l	48 hrs	124 lpm	1500 $kg/m^2/d$
Test 2 VSL-08R1360-1 [3]	Al-Limited Waste	500 g/l	48 hrs	71 lpm	1050 $kg/m^2/d$
Configuration Test 9A VSL-04R4800-4 [7]	AZ-101	400 g/l	145 hrs	64 lpm	1050 $kg/m^2/d$
Configuration Test 9B VSL-04R4800-4 [7]	AZ-101	400 g/l	72 hrs	134 lpm	1400 $kg/m^2/d$
Test 1B VSL-05R5800-1 [20]	AZ-102	340 g/l	114 hrs	65 lpm	900 $kg/m^2/d$
Test 2B VSL-05R5800-1 [20]	C-106/AY-102, High Waste Loading	340 g/l	105 hrs	90 lpm	1050 $kg/m^2/d$
MACT HLW 1 (400°C plenum) VSL-05R5830-1 [54]	C-106/AY-102, spiked	430 g/l	52 hrs	24 lpm	700 $kg/m^2/d$
MACT HLW 2A (345°C plenum) VSL-05R5830-1 [54]	C-106/AY-102, spiked	430 g/l	75 hrs	9 lpm	550 $kg/m^2/d$
MACT HLW 1-cont (400°C plenum) VSL-05R5830-1 [54]	C-106/AY-102, spiked	430 g/l	19 hrs	28 lpm	742 $kg/m^2/d$
MACT HLW 2B (500°C plenum) VSL-05R5830-1 [54]	C-106/AY-102, spiked	430 g/l	54 hrs	43 lpm	1072 $kg/m^2/d$

Table 5.1. DM1200 Vitrification System Data Collection Points and Data Acquisition Methods.

Component	Comments	Recording Method		Units
		Manual	Electronic	
Feed	Feed Tank Mass	30 min	2 min	kg
	Feed Total Cycle Time	30 min	NR	sec
	Speed of mixing blades	120 min	2 min	Hz
Melter	Electrode Current	120 min	NR	A
	Electrode Voltage	120 min	2 min	V
	Electrode Power	120 min	2 min	kW
	Resistance of Glass Pool	120 min	NR	ohms
	Plenum Temperature-8" From Ceiling	120 min	2 min	°C
	Plenum Thermowell-17" From Ceiling	120 min	2 min	°C
	Plenum Exposed-17" From Ceiling	120 min	2 min	°C
	Glass Pool Temperature-1" From Bottom (West)	120 min	2 min	°C
	Glass Pool Temperature-13" From Bottom (West)	120 min	2 min	°C
	Glass Pool Temperature-18" From Bottom (West)	120 min	2 min	°C
	Glass Pool Temperature-27" From Bottom (West)	120 min	2 min	°C
	Glass Pool Temperature-9" From Bottom (East)	120 min	2 min	°C
	Glass Pool Temperature-18" From Bottom (East)	120 min	2 min	°C
	Glass Pool Temperature-24" From Bottom (East)	120 min	2 min	°C
	Glass Pool Temperature-30" From Bottom (East)	120 min	2 min	°C
	T/C 02, 03, 12, 13 Average	120 min	2 min	°C
	Riser Temperature	120 min	2 min	°C
	Discharge Chamber Temperature #1	120 min	2 min	°C
	Discharge Chamber Temperature #2	120 min	2 min	°C
	Discharge Exhaust Flow Temperature	120 min	2 min	°C
	Bottom Electrode Temperature	120 min	2 min	°C
	East Electrode Temperature	120 min	2 min	°C
	West Electrode Temperature	120 min	2 min	°C
	Melter Pressure	*120 min*	2 min	In WC
	Glass Level	120 min	2 min	Inches
	Glass Density	120 min	2 min	g/cc

NR: not recorded in this manner.

Table 5.1. DM1200 Vitrification System Data Collection Points and Data Acquisition Methods (cont'd).

Component	Comments	Recording Method		Units
		Manual	Electronic	
Melter	Electrode Bubbler #1 Flow	120 min	NR	SCFH
	Electrode Bubbler #2 Flow	120 min	2 min	SCFH
	Electrode Bubbler #3 Flow	120 min	2 min	SCFH
	Electrode Bubbler #4 Flow	120 min	2 min	SCFH
	Electrode Bubbler #5 Flow	120 min	NR	SCFH
	Lance Bubbler 1 Flow*	NR	2 min	lpm
	Lance Bubbler 2 Flow*	NR	2 min	lpm
	Lance Bubbler 3 Flow*	NR	2 min	lpm
	Lance Bubbler 4 Flow*	NR	2 min	lpm
	Electrode Bubbler #1 Back Pressure	120 min	NR	PSI
	Electrode Bubbler #2 Back Pressure	120 min	NR	PSI
	Electrode Bubbler #3 Back Pressure	120 min	NR	PSI
	Electrode Bubbler #4 Back Pressure	120 min	NR	PSI
	Electrode Bubbler #5 Back Pressure	120 min	NR	PSI
	Back Pressure Lance Bubbler 1	NR	2 min	PSI
	Back Pressure Lance Bubbler 2	NR	2 min	PSI
	Back Pressure Lance Bubbler 3	NR	2 min	PSI
	Back Pressure Lance Bubbler 4	NR	2 min	PSI
	Return signal Lance Bubbler 1A flow	NR	2 min	lpm
	Return signal Lance Bubbler 2A flow	NR	2 min	lpm
	Return signal Lance Bubbler 3A flow	NR	2 min	lpm
	Return signal Lance Bubbler 4A flow	NR	2 min	lpm
	Return signal Lance Bubbler 1B flow	NR	2 min	lpm
	Return signal Lance Bubbler 2B flow	NR	2 min	lpm
	Return signal Lance Bubbler 3B flow	NR	2 min	lpm
	Return signal Lance Bubbler 4B flow	NR	2 min	lpm
	NE Corner Wall Purge Flow	120 min	NR	SCFH
	SE Corner Wall Purge Flow	120 min	NR	SCFH
	SW Corner Wall Purge Flow	120 min	NR	SCFH
	NW Corner Wall Purge Flow	120 min	NR	SCFH
	Dam Cooler Inlet Flow	120 min	NR	SCFH
	Argon Purge Flowmeter #1	120 min	NR	SCFH
	Argon Purge Flowmeter #2	120 min	NR	SCFH
	Argon Purge Flowmeter #3	120 min	NR	SCFH
	MM-OG-PR-003 Purge Air	120 min	NR	SCFH
	MM-OG-DPR-002 Low Side Purge Air	120 min	NR	SCFH
	MM-OG-DPR-002 High Side Purge Air	120 min	NR	SCFH
	MM-XXX-PR-200 Purge Air	120 min	NR	SCFH

* = Only two top-entering bubbler locations will be used for these tests. Flow value identified is a mathematical sum of other individual signals.
NR: not recorded in this manner.

Table 5.1. DM1200 Vitrification System Data Collection Points and Data Acquisition Methods (cont'd).

Component	Comments	Recording Method		Units
		Manual	Electronic	
Cooling Panel	Chilled Water Supply Temperature	NR	2 min	°C
	Chilled Water Return Temperature	NR	2 min	°C
	Cooling Panel N-1 Temperature	NR	2 min	°C
	Cooling Panel N-2 Temperature	NR	2 min	°C
	Cooling Panel N-3 Temperature	NR	2 min	°C
	Cooling Panel N-4 Temperature	NR	2 min	°C
	Cooling Panel N-5 Temperature	NR	2 min	°C
	Cooling Panel S-1 Temperature	NR	2 min	°C
	Cooling Panel S-2 Temperature	NR	2 min	°C
	Cooling Panel S-3 Temperature	NR	2 min	°C
	Cooling Panel S-4 Temperature	NR	2 min	°C
	Cooling Panel E-1 Temperature	NR	2 min	°C
	Cooling Panel E-2 Temperature	NR	2 min	°C
	Cooling Panel W-1 Temperature	NR	2 min	°C
	Cooling Panel W-2 Temperature	NR	2 min	°C
	Cooling Panel B-1 Temperature	NR	2 min	°C
	Cooling Panel B-2 Temperature	NR	2 min	°C
	Cooling Panel B-3 Temperature	NR	2 min	°C
	MCL-CW-P-401 Inlet Pressure	120 min	NR	PSI
	MCL-CW-P-401 Outlet Pressure	120 min	NR	PSI
	Primary CW Supply Temperature	120 min	NR	°F
	Primary CW Return Temperature	120 min	NR	°F
	Primary Chilled Water Return	120 min	NR	PSI
Film Cooler	Film Cooler Air Temperature	120 min	2 min	°C
	Film Cooler Outlet Temperature	120 min	2 min	°C
	Film Cooler Differential Pressure	120 min	2 min	In WC
	Film Cooler Air Flow Rate (Pitot tube)	120 min	NR	In WC
Control Air	Temperature	120 min	2 min	°C
	Differential Pressure	120 min	2 min	In WC
	Flow Rate	120 min	2 min	scfm
Transition Line	Transition Line Differential Pressure	120 min	2 min	In WC
Melter	Melter Pressure	120	2 min	In WC

NR: not recorded in this manner.

Table 5.1. DM1200 Vitrification System Data Collection Points and Data Acquisition Methods (cont'd).

Component	Comments	Recording Method		Units
		Manual	Electronic	
SBS	Gas Inlet Temperature	120 min	2 min	°C
	Gas Outlet Temperature	120 min	2 min	°C
	SBS jacket chilled water outlet temperature	120 min	2 min	°C
	Down-comer temperature @ 3"	120 min	2 min	°C
	Down-comer temperature @ 8"	120 min	2 min	°C
	Down-comer temperature @ 13"	120 min	2 min	°C
	Down-comer temperature @ 18"	120 min	2 min	°C
	Down-comer temperature @ 23"	120 min	2 min	°C
	Down-comer temperature @ 28"	120 min	2 min	°C
	Down-comer temperature @ 33"	120 min	2 min	°C
	Down-comer temperature @ 38"	120 min	2 min	°C
	Down-comer temperature @ 43"	120 min	2 min	°C
	Down-comer temperature @ 48"	120 min	2 min	°C
	Down-comer temperature @ 53"	120 min	2 min	°C
	Down-comer temperature @ 58"	120 min	2 min	°C
	Down-comer temperature @ 63"	120 min	2 min	°C
	Down-comer temperature @ 68"	120 min	2 min	°C
	SBS inlet gas pressure	120 min	2 min	In WC
	SBS off-gas outlet pressure transmitter	120 min	2 min	In WC
	SBS cooling cw flow rate	120 min	2 min	GPM
	chilled water inlet temperature	120 min	2 min	°C
	SBS cooling coil cw inlet temperature	120 min	2 min	°C
	SBS overflow tank level	120 min	NR	Gallons
	SBS overflow tank flow totalizer	120 min	NR	Gallons
	SBS cooling coil outlet temperature	120 min	2 min	°C
	SBS lance BD totalizer	120 min	NR	Gallons
	SBS overflow tk vent pressure	120 min	NR	In WC
	SBS-pw-p-502 outlet press. Indication	120 min	2 min	PSI
	chilled water supply temperature	120 min	NR	°C
	chilled water supply pressure	120 min	NR	PSI
	chilled water return pressure	120 min	NR	PSI
	chilled water return temperature	120 min	NR	°C
	SBS overflow tank high level switch	120 min	NR	Switch
	SBS overflow tank low level switch	120 min	NR	Switch
	SBS process water level indicator	120 min	NR	Gallons
	SBS-recirc pump discharge temperature	120 min	2 min	°C
	SBS-pw-t-501 FW fill flow totalizer	120 min	NR	Gallons
	SBS pw temperature outside submerged bed	120 min	2 min	°C
	SBS pw level transmitter	120 min	2 min	Gallons
	SBS recirc flow rate transmitter	120 min	2 min	GPM
	SBS down-comer annulus pressure indicator	120 min	NR	PSI

NR : not recorded this manner.

Table 5.1. DM1200 Vitrification System Data Collection Points and Data Acquisition Methods (cont'd).

Component	Comments	Recording Method		Units
		Manual	Electronic	
WESP	WESP Inlet Gas Temperature	120 min	2 min	°C
	WESP Outlet Gas Temperature	120 min	2 min	°C
	WESP Differential Pressure	120 min	2 min	In WC
	WESP Liquid Level	120 min	NR	Inches
	WESP Voltage	120 min	NR	kV
	WESP Current	120 min	NR	mA
	WE-PW-P-601 Discharge Pressure	120 min	NR	PSI
	WESP Spray Nozzle Inlet Pressure	120 min	NR	PSI
	WESP Recirc. Flowmeter	120 min	NR	LPM
	WESP Liquid Level	120 min	NR	Inches
	WESP Outlet Absolute Pressure	NR	2 min	Inches
	WESP Outlet Gas Flow Rate (Pitot)	NR	2 min	Inches
	Blow-Down Flow Totalizer	120 min	NR	Gallons
HEME #1	HEME #1 Differential Pressure	120 min	NR	In WC
	HEME #1 Gas Temperature	120 min	NR	°C
HEPA #1	HEPA #1 Differential Pressure	120 min	2 min	In WC
	HEPA #1 Outlet Gas Temperature	120 min	2 min	°C
Packed-Bed (Caustic) Scrubber	Caustic Scrubber Inlet Gas Temperature	120 min	2 min	°C
	Caustic Scrubber Differential Pressure	120 min	NR	In WC
	Caustic Scrubber Liquid Level	120 min	NR	Inches
	Caustic Scrubber Recirc. Flow rate	120 min	NR	GPM
	Caustic Scrubber pH	120 min	NR	pH units
	Sump Temperature	120 min	2 min	°C
	Blow-Down Flow Totalizer	120 min	NR	Gallons
HEME	HEME #2 Differential Pressure	120 min	NR	In WC
	HEME #2 Gas Temperature	120 min	NR	°C
	Blow-Down Flow Totalizer	120 min	NR	Gallons
Process Heater	Process Heater Outlet Gas Temp	120 min	2 min	°C
Catalyst Bed	TCO Heater Gas Inlet Temperature	NR	2 min	°C
	TCO Heater Gas Outlet Temperature	NR	2 min	°C
	SCR Gas Outlet Temperature	NR	2 min	°C
	Inlet Pressure	NR	2 min	In WC
	Outlet Pressure	NR	2 min	In WC
	TCO Differential Pressure	120 min	NR	In WC
	SCR Differential Pressure	120 min	NR	In WC
	Total Differential Pressure	120 min	NR	In WC
	NH3 Supply Filter Differential Pressure	120 min	NR	In WC
	NH3 Mass Flow Rate	120 min	NR	lbs/hr
	Dilution Air Flow	120 min	NR	Scfm
	Dilution Air Pressure	120 min	NR	psi
	Dilution Air Temperature	120 min	NR	°C
	NH3 Supply Pressure Regulator	120 min	NR	Psi
	NH3 Supply Pressure	120 min	NR	psi

NR: not recorded in this manner.

Table 5.1. DM1200 Vitrification System Data Collection Points and Data Acquisition Methods (cont'd).

Component	Comments	Recording Method		Units
		Manual	Electronic	
HEPA #2	HEPA #2 Differential Pressure	120 min	NR	In WC
Blowdown	SBS Overflow Blowdown Sample Tank Level	120 min	NR	Gallons
	SBS Overflow Blowdown Sample Tank Level	120 min	NR	Gallons
	WESP Blowdown Sample Tank Level	120 min	NR	Gallons
	Acid HEME Blowdown Sample Tank Level	120 min	NR	Gallons
	PBS Blowdown Sample Tank Level	120 min	NR	Gallons
	Caustic HEME Blowdown Sample Tank Level	120 min	NR	Gallons
	Caustic Storage Tank #1 Level	120 min	NR	Gallons
	Acid Storage Tank #2 Level	120 min	NR	Gallons
	Caustic Tank pH	120 min	2 min	pH units
	Acid Tank pH	120 min	2 min	pH units
	Acid Tank Vent Pressure	120 min	2 min	In. WC
	Neutralization Tank Vent Pressure	120 min	2 min	In. WC
	Neutralizing Storage Tank Level	120 min	NR	Gallons
	Neutralization Tank pH	120 min	2 min	pH units
Bypass HEPA	Bypass HEPA Inlet Temperature	120 min	2 min	°C

NR: not recorded in this manner.

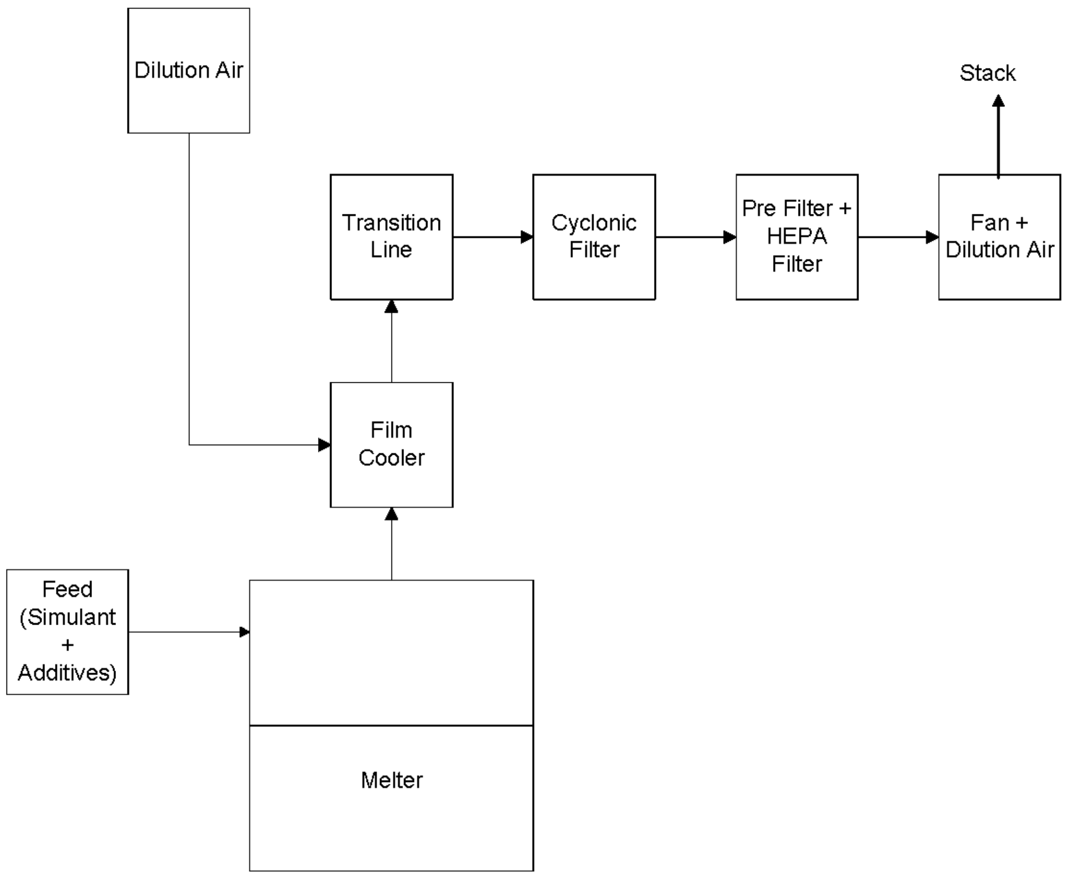

Figure 2.1. Schematic diagram of DuraMelter 100 vitrification system.

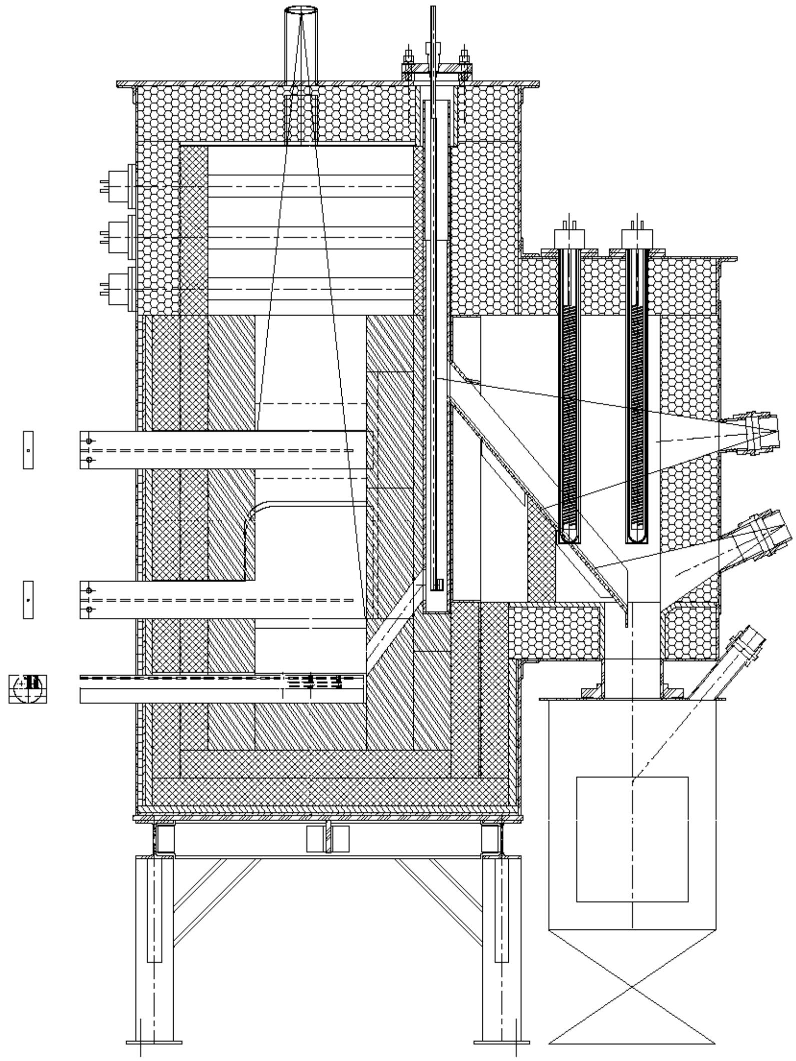

Figure 2.2a. Schematic diagram showing cross-section through the DM100-BL melter.

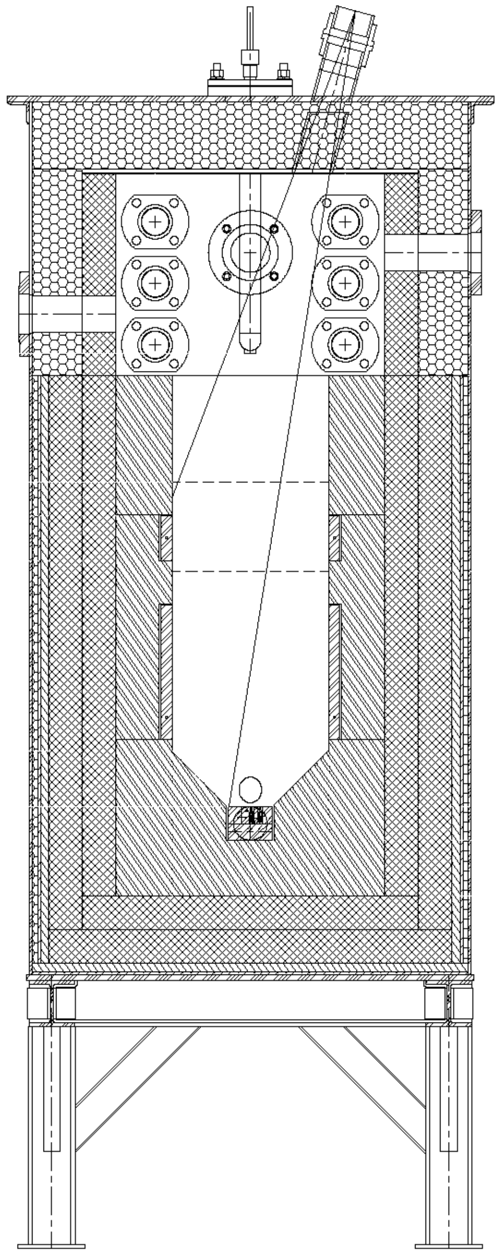

Figure 2.2b. Schematic diagram showing cross-section through the DM100-BL melter.

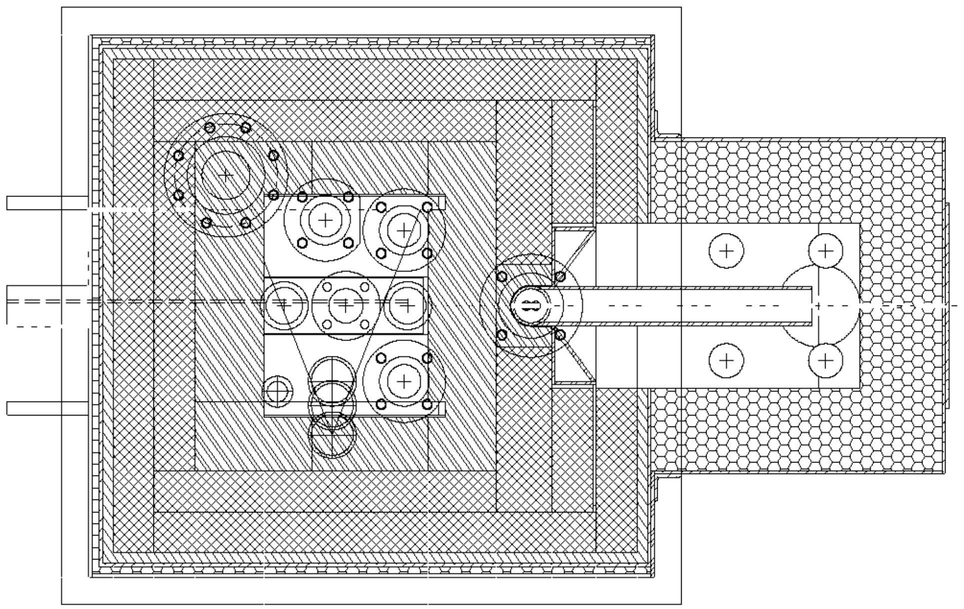

Figure 2.2c. Schematic diagram showing cross-section through the DM-100-BL-melter. Plan view showing locations of lid ports.

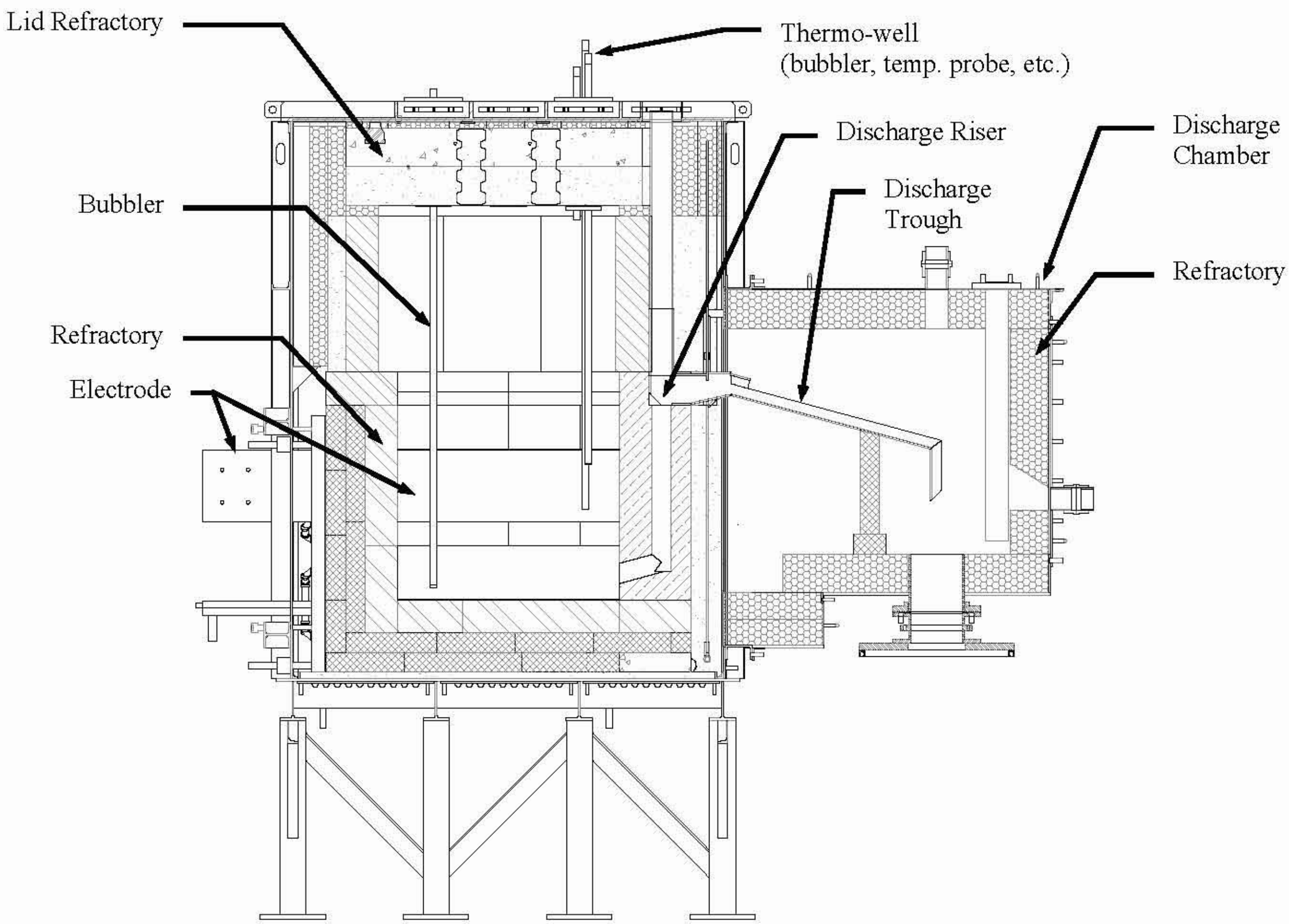

Figure 2.3. Cross-section of the DM1200 melter through the discharge chamber.

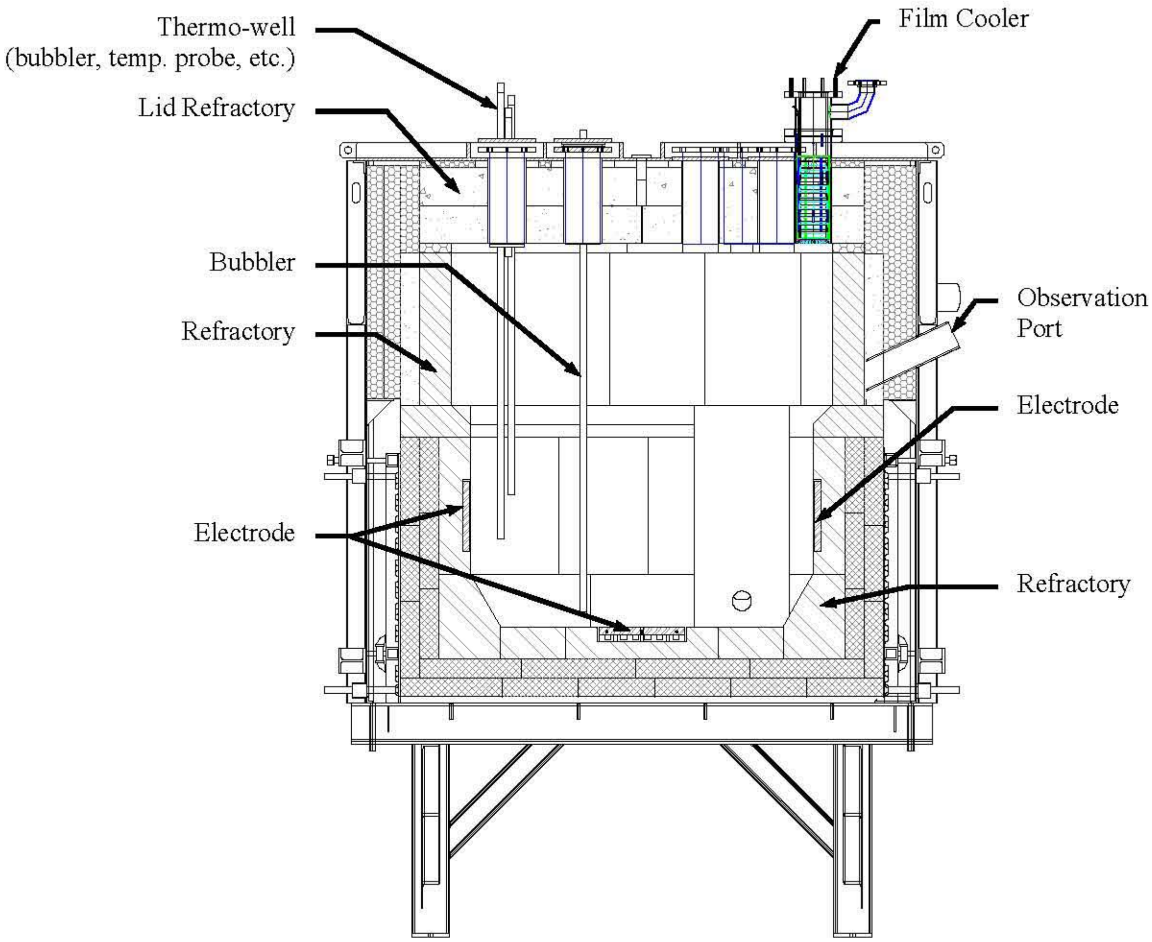

Figure 2.4. Cross-section through the DM1200 melter showing electrodes.

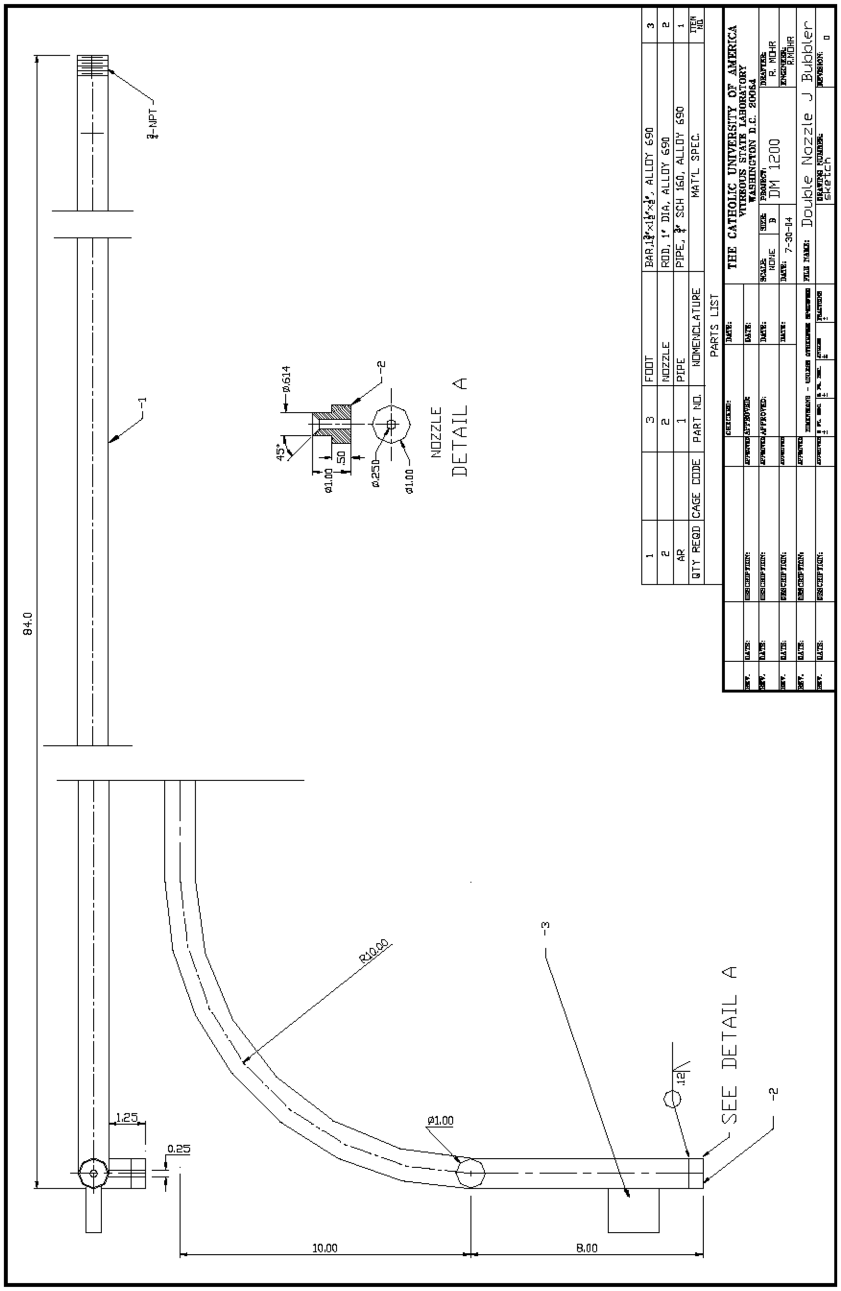

Figure 2.5. Specifications of double outlet "J" bubbler.

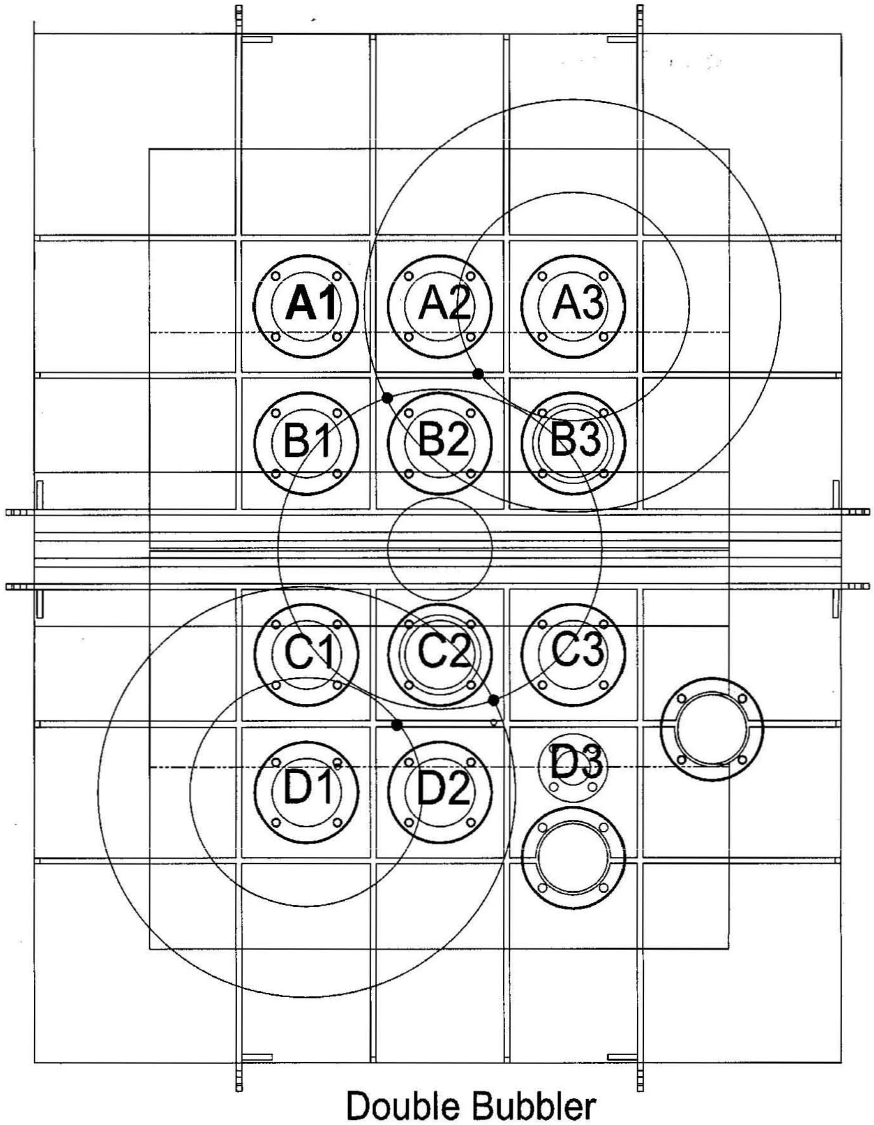

Figure 2.6. Placement of double outlet bubblers. Note: solid circles represent location of bubbler outlet.

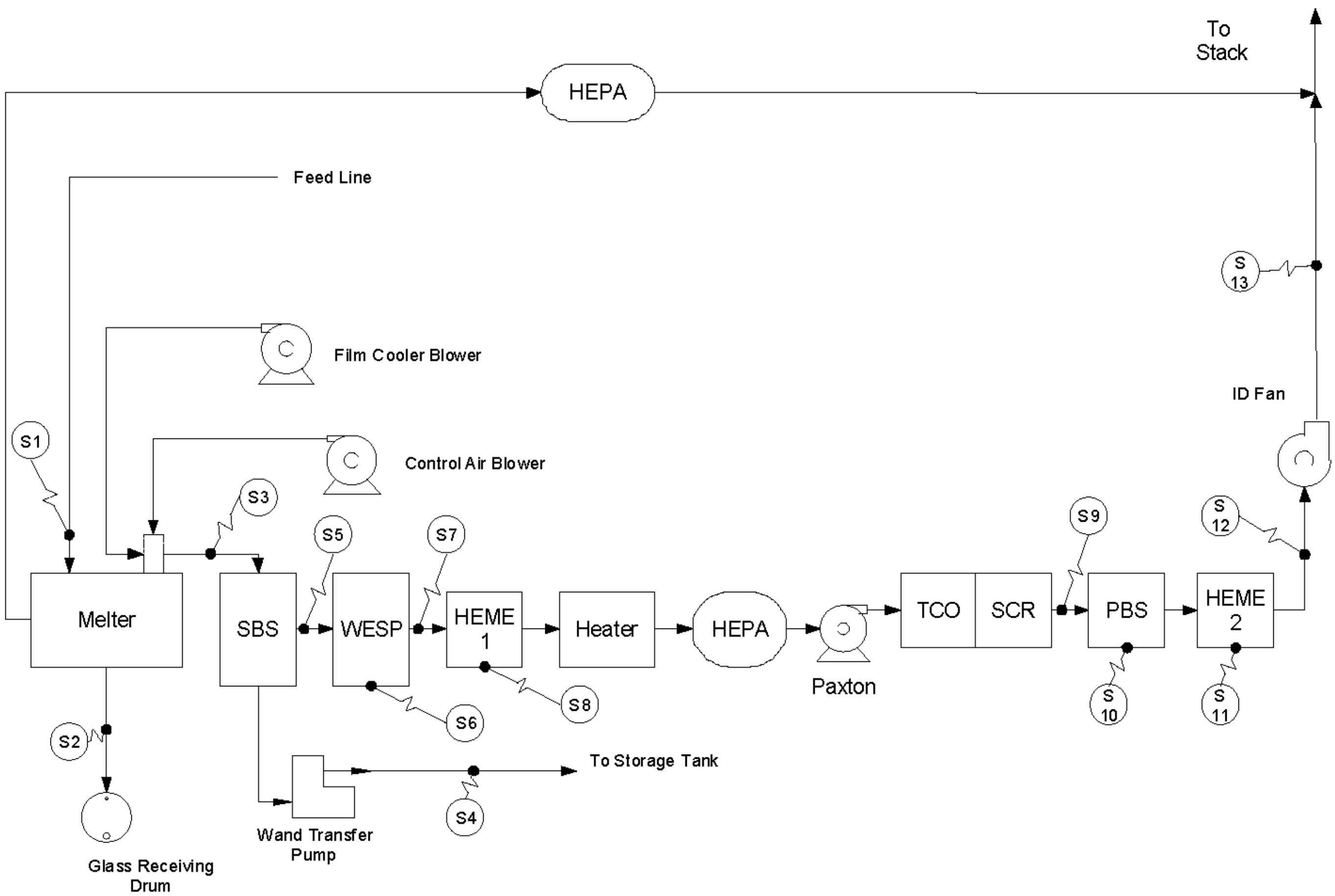

Figure 2.7. Schematic diagram of DM1200 off-gas system. "Sx" indicates sampling point; see Table 2.2 for identification.