ACC#248519 DP-MS-86-75

GLASS MAKING TECHNOLOGY FOR HIGH-LEVEL NUCLEAR WASTE

by

RECORDS ADMINISTRATION

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A paper proposed for presentation at the American Institute of Chemical Engineers Meeting Boston, MA August 24-27, 1986

and for publication in the proceedings

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INTRODUCTION

R. F. Taylor of Harwell, UK, has published an excellent review paper on "Chemical Engineering Problems of Radioactive Waste Fixation by Vitrification". The general subject is exhaustively treated there. Our paper, despite its ambitious title, addresses specific and unique chemical engineering aspects of the Defense Waste Processing Facility (DWPF) at the Savannah River Plant.

Previous articles and reports, 2-7 as well as other papers being presented at this meeting, give an overall view of DWPF processes and design and treat certain parts in more detail. This paper addresses the glass melter and those processes that are directly coupled to it. A somewhat disproportionate emphasis is given to sludge pretreatment, for the sake of completeness in this Session.

We have attempted to focus on those features of the DWPF that may be of general interest or even useful to the practicing chemical engineer.

Glassmaking can be reduced to a minimum of 3 unit operations: batching, melting, and forming. Nuclear waste vitrification also requires a major sidestream process, off-gas decontamination. Forming, so very important to commercial glass production, is in the DWPF merely a matter of pouring the molten glass from the melter into a large mold, or canister, with uncontrolled "natural" cooling. Thus the main divisions to be discussed are batching, glass melting, and off-gas treatment.

BATCHING

Batch feed preparation and delivery must provide a chemically balanced, homogeneous stream to the melter. Although the DWPF melter has a 64-hour glass residence time, mixing is very slow in the molten phase and nonexistent in the melting phase or "feed pile", where crucial chemical reactions occur. Therefore the melter is quite unforgiving of batching errors.

Wet vs. Dry Batch

Typically, industrial glass melters are fed a dry mixture of sand, chemicals, and cullet.⁸ Dry feed minimizes melter energy consumption; and energy efficiency is often the key to economic success. Nuclear waste, however, exists as dilute aqueous solutions and slurries.

Early waste vitrification processes, both in this country and abroad, dried and calcined the waste prior to blending with glass-forming chemicals or frit. This required a rotary drum, spray tower, or other type dryer. Dry powder conveying and blending

equipment is notoriously unreliable and is avoided, when possible, in remote cells. Radioactive powders are easily dispersed. In some cases, poor mixing of dried waste with frit resulted in serious melting and quality problems.

Wet slurry batching has proven to be preferable for nuclear waste vitrification in the DWPF and several similar projects world-wide. Chemical accuracy, homogeneity, sampling, and therefore quality assurance are superior. Mechanical equipment is limited to highly reliable agitators and centrifugal pumps. Radioactive dust is avoided. High-cost canyon space is saved.

Slurry feeding, as it is called, is not without penalty: the melter energy requirement is roughly doubled, to evaporate water. Additional "boosting" heaters must be used and/or the melter surface area must be much larger for the same glass production rate. For a typical slurry of 50 wt% solids and a typical melt temperature of 1150°C, melt flux is about 20-40 kg/hr per square meter of molten glass surface.

Direct feeding of water slurry at <100°C onto molten glass at >1000°C is not a safety (steam explosion) problem as once feared, due to the high viscosity of the glass. Some steam surging does occur but can be accommodated by the ventilation system. Provided that reasonable care is taken not to overfeed and flood the melter, slurry does not cause excessive damage to refractory walls.

Chemical Treatment

Nuclear high-level waste originates as a strong acid, the raffinate left after the extraction of recyclable fuel and products from dissolved fuel elements. At Savannah River as well as at some other nuclear fuel processing plants, this raffinate is promptly neutralized with NaOH for interim storage in tanks. Upon neutralization, hydroxides of iron, aluminum, and other metals including fission products and actinides are precipitated as a sludge.

Nuclear waste in almost any chemical form that can be delivered to a melter may be vitrified. However, some chemical adjustments are usually made, either to reduce the inert volume, or to remove some troublesome species, or to improve melter performance. A considerable amount of chemical pretreatment is done for the DWPF.

First, the bulk of soluble, nonradioactive salt is removed for disposal as a solid low-level waste. This greatly reduces glass volume. It also removes the need for chemical denitration of melter feed — or at least mitigates the nitrate complications seen in other systems such as enhanced Ru volatility and NO_x scrubbers. The salt decontamination process is described by Doherty⁹ and the low-level waste process and form by Langton et al., ¹⁰ in papers being presented at this meeting. Soluble radioactive species (principally Cs-137) are concentrated and processed as described by Doherty. ⁹ Significant chemical engineering aspects of the

processing of sludge and of combined sludge and salt-concentrate into DWPF melter feed are described below.

Sludge solids are leached to remove soluble, nonradioactive components by batch washing in a tank. The noteworthy aspect of this treatment is the size of the batch tank: 5 million liters (1.3 million gallons). This may not be a chemical industry record, but it is an unprecedented scale for remote processing of radioactive material. The compelling economic reason for using these large tanks is their availability. With the advent of DWPF waste solidification, several new 26-m dia underground tanks that would otherwise have been needed for liquid waste storage have become available. A large capital saving was realized by using these tanks and gravity separation for sludge washing, instead of new tankage and centrifuges in a shielded building as originally planned. The very large batch size affords another significant advantage for DWPF: long term feed constancy. A single batch of sludge slurry will feed the DWPF for 2 years.

In practice, sludges from several selected storage tanks will be mobilized (slurried) using large pumps with submerged discharge nozzles, and then pumped to a leach/wash tank. Sludge with a high aluminum content will first be leached with hot NaOH. Aluminum removal reduces both the viscosity and the volume of glass. Other sludge may then be added, and the entire batch washed about 20 times. Each wash-settle-decant cycle takes 1 month; nearly 2 years are required to process a batch. A large number of relatively

small washes was selected to approximate continuous washing and thereby minimize the total volume of wash water that must eventually be evaporated and/or decontaminated. Washing of 1-2 million liter batches of sludge slurry will proceed simultaneously in 2 tanks. The products are combined in a single DWPF feed tank. Four 15,000 l/min submerged centrifugal pumps are required to suspend and homogenize a 4-million-liter batch of 15% sludge slurry.

In the shielded canyon building of the DWPF, the washed sludge slurry is treated with formic acid to release gases such as CO₂, NO, and O₂ which could otherwise cause excessive foaming in the melter. Equally important, formic acid reduces Hg⁺² to Hg° which is stripped out and recovered as a byproduct.* The formic acid also reduces the slurry yield stress by dissolving some species and reducing the agglomerate size of others. The cesium/potassium concentrate from the salt precipitate hydrolysis process⁹ is added to the sludge.

A 40,000-liter batch reactor/evaporator (Figure 1) is used to treat the sludge with formic acid, blend in the cesium concentrate, and strip out the mercury. The noteworthy chemical engineering feature of this vessel is the coil/agitator configuration.

Remotely replaceable coils were specified because of coil failure

^{*}This is for environmental protection only. Being volatile, mercury will not become part of the glass matrix.

experience in the Purex plant at Savannah River. Replaceability was accomplished with a tight, 1.2 meter (4-foot) diameter nest of 3 coils suspended from a large flange in the tank top. The agitator that operates in the volume within these coils (approximately 10% of the tank) suspends and homogenizes, throughout the whole tank, non-Newtonian slurries of up to 50 wt% solids with yield stresses up to 150 dyne/cm². This is a 130-rpm, 75 kW (100 hp) agitator with a four-blade flat bottom turbine and a special, multi-angle pitched top turbine. Both impellers are 0.92 m in diameter. Slurry flow is generally downward within the coil nest, radially outward near the bottom, and up at the walls; although with thin slurries, considerably more radial and tangential motion is observed.

Formic acid is added slowly to hot (90°C) slurry to control the release rate of reaction gases. The slurry is then boiled to complete the chemical reactions and to strip out mercury. Water and mercury are co-condensed and separated in a decanter. Water is recycled to the evaporator, and the mercury is pumped to a hot cell for purification by washing and distillation.

Mercury oxides comprise 1-3% of the sludge solids, apparently distributed as finely-divided particles. Mercury is chemically reduced to the metal by formic acid, but does not readily coalesce and settle out from the thick sludge slurry. The late David Janes found that, for >0.5 wt% Hg in formated sludge, boiling evaporates mercury and water in a constant proportion of 1:750. This compares

to the equilibrium vapor mass ratio of 1:250. Below about 0.5 wt% Hg, the mercury stripping efficiency decreases markedly. Janes' unpublished work is summarized on Figure 2. Mercury stripping normally proceeds for 28-36 hours but, as is readily apparent from Figure 2, complete removal is not achieved. This can be accepted, because the balance will be condensed from the melter off-gas and returned to the waste tank farm for eventual recycle to the DWPF.

Glassformers

The borosilicate glass matrix can be added to the melter feed either as individual batch chemicals (e.g. borax, soda, and silica) or as premelted glass beads or frit. DWPF elected to use frit to simplify storage and delivery systems and minimize batching errors. A single frit formula can accomodate the chemical variations expected in DWPF waste feed over a period of several years. Batch chemicals, either soluble or insoluble, could also be used. Some studies have shown significant melt rate enhancement when only one or two components are added as chemicals and the rest as frit.

careful attention must be paid to frit particle size and shape. Slurries of frit alone and of frit plus waste sludge must be pumpable and reasonably stable (non-settling). Frit and waste should not easily separate from one another. Other factors include abrasion (frit angularity), melting behavior, and direct cost. For DWPF, crushed or ground frit sized to -80, +200 mesh (74 to 175 µm) is optimal. These specifications are influenced by the fact that a portion of the frit is used as an abrasive to clean (decontaminate)

the surface of product glass canisters. Spherical beads have handling advantages but are not as efficient for canister decontamination, and are more expensive.

Purchased frit is shipped and stored in 2300-kg containers that are sealed to keep out water vapor, which can cause lumping. At the time of use, frit is slurried with water for transport into the DWPF process tanks. A small amount of formic acid is added to keep the frit slurry below pH 8. Alkali-containing frit slurries tend to increase the pH and thicken. Frit slurry is fed to melter feed batch tanks at 60 wt% solids. For decontamination of product canisters, 8% frit slurry is used, boosted with 800-kPa (100 psig) air at the blast nozzles.7

Pumping

Transfers of sludge, frit, and sludge/frit slurries from tank to tank in the DWPF are accomplished with commercially-available or slightly modified centrifugal slurry pumps. Special systems were developed for two unusual pumping requirements: melter feed delivery, and mercury transfer.

The DWPF melter is fed through the lid, at two points to ensure good distribution on the melt surface. The feed is an abrasive, non-Newtonian slurry, approximately 50% solids with a yield stress up to 150 dynes/cm². Two independent feed systems, normally delivering about 1.5 l/min each, were specified. Accurate flowrate control is required. Lines must be small (ca. 1-cm) to maintain enough velocity to prevent solids settling. Plugging of

these small lines by oversize sludge/frit lumps and the inevitable "trash" must be prevented. The hot end of the feed line (inside the melter) must be cooled to prevent pluggage by drying. A number of safety and other interlocks automatically stop feed, and feed lines must then be promptly and automatically flushed with water. All equipment must be radiation-resistant, reasonably long-lived, and remotely replaceable.

The system developed to satisfy these criteria is shown in Figure 3. The pump itself is a relatively standard, 400 1/min centrifugal slurry pump very similar to those used elsewhere in the DWPF for batch transfers. A pressure head is generated in a recirculation loop, and a small sidestream is taken off to feed the melter. Efforts to find a small direct delivery pump have been unfruitful. Peristaltic and diaphragm pumps are in general not radiation-tolerant. Commercially-available gear and vane pumps and other positive-displacement devices including "progressive cavity" pumps failed tests with simulated slurry, usually by binding or excessive wear from frit. Airlifts, although successful for other radwastes, plugged excessively with simulated DWPF melter feed. Low-flow centrifugal pumps do not generate sufficient head.

Flow in the small feed line is measured with a ceramic-lined, magnetic-type flowmeter, and is controlled by varying the pump speed. A bar-type screen, installed parallel to the main recirculation flow at the feed line takeoff tee, keeps lumps out of the small line and is self-cleaning. Without this final screening,

pluggage occurs daily; with it, pluggage is rare. The feed tube inside the melter is water-jacketed.

The mercury pump developed for DWPF is based on a "water-lift" principle similar to airlifting of other liquids. The pump is shown schematically in Figure 4. The critical parameters are lift line diameter and water flow rate. Tests have shown that mercury can be lifted 12 m in a 1.6-cm diameter line using 10 1/min water at 2000 kPa (265 psig).

GLASS MELTING

Feed material, after delivery to the melter, floats on top of the molten glass which has a specific gravity of about 2.5. Water is driven off the "feed cap" and vitrification gases are released from the waste as the batch is gradually heated to the glass pool temperature of 1150°C. The ground glass (frit) which has been added to the radioactive waste melts and dissolves over 70 known chemical species in the waste stream. Waste glass is produced at 100 kg/hr.

The melter (Figure 5) is a refractory-lined cylindrical vessel with a water-cooled stainless steel shell. A high-density, fused-cast, chrome-alumina refractory contains the molten glass, which, in addition to dissolving the different chemical species (mostly metal oxides) in the waste, attacks metals and refractories. Wall material loss, based on worst case conditions, may be as high as 5 cm per year. The initial refractory lining will be 30 cm thick. Based on loss of refractory material, the melter will have at least

a 2 to 4 year life expectancy before structural or containment failure.

To achieve the glass production rates desired, and to ensure combustion of vitrification gases, plenum heaters will be used in the DWPF melter. Unlike other nuclear waste glass melters which have a "cool" vapor space above the glass pool, heaters will maintain the vapor space in the DWPF melter at 625-800°C. A unique feature of these heaters is that, instead of inserting a standard high temperature heating element inside a sheath, the metal "sheath" (tube) is itself the heating element. Eight nickel/ chrome/iron tubes are suspended horizontally above the glass pool; and, with appropriate power supplies and connectors, up to 7100 amperes of current flow through the tubes causing direct resistance (I²R) heating. Due to the 183 cm span the tubes traverse inside the melter, the operating temperature of the tubes must be kept cooler than a standard high temperature heater to avoid metal creep. Also, the high-amperage load requires a close-coupled water-cooled transformer which makes the system energy-inefficient.

One very unique aspect of the hot plenum operation of the glass melter is the ability to burn organics which will accompany the radioactive waste feed. Soluble radioactive waste species are concentrated via an organic precipitation process. Most of the organics are removed from the waste species (described by Doherty⁹). The residual organics in the melter feed stream readily vaporize. In addition, nonvolatile organics in the melter feed including

formate and phenylborate salts will be pyrolyzed, releasing CO and H₂. These, along with the volatile organics, must be burned within the melter plenum to avoid the formation of explosive mixtures in the downstream off-gas treatment equipment. Thus the melter operates as both a glass production vessel and as an incinerator. It operates at a slight vacuum. Up to 40 kg/hr of air will leak in, and 130 kg/hr (150% of maximum stoichiometric combustion requirement) is added positively.

Power to keep the molten glass at 1150°C and to provide the energy to melt the incoming feed is supplied by direct "in-situ" joule heating. Two pairs of electrodes (Figure 5) are arranged in an over-under configuration and equipped such that both use the same phase electrical circuit. Typically, molten glass at 1150°C has a resistivity of 2 ohm-cm. The electrodes operate at 70 v and supply 160 kW to melt the feed and make up for heat losses through the walls.

Temperature control is achieved through the automatic adjustment of current flowing between the electrodes. Silicon controlled rectifiers (SCR's) control the power input to the primary side of a multi-tap transformer based on feed back from the temperature control unit. Use of the SCR's on the primary side of the transformer eliminates the potential for sending a DC current rather than an AC current through the melter. The DC would cause plating on one set of electrodes and dissolve the other set. Glass

corrosion of the electrodes is slowed via use of a nickel/chrome/iron alloy.

Replaceable thermowells, mounted so that they touch the electrically active glass yet do not allow the current to flow to the grounded melter shell, will contain several Type B (platinum/ platinum-rhodium) thermocouples. Type B thermocouples were selected for their long term resistance to drift or failure at elevated temperatures. Other process control features added to the melter include a level-sensing, dip-tube bubbler, and two television camera systems which monitor the amount of coverage of the feed cap. Pumping excessive quantities of feed into the melter can result in the feed pile bridging across the entire melter surface. Since vitrification gases are released beneath the feed pile, a bridged surface can cause large pressure surges as the pile is breached and resealed, or collapses. The television cameras allow direct determination of the amount of feed pile coverage to prevent the unwanted pressure surges. Moderate surging is normal because the floating feed pile may either trap a bubble of gas beneath it, or the standing pool of boiling water on top of the feed pile may suddenly run off or break through.

Two unique heater systems have been designed for the passageways that carry the molten glass from the main chamber and discharge it into the product canisters. Both heaters use nickel/ chrome/iron alloy for both the inner flow-through pipe and the strip heaters around it (Figure 6). Typically, flowing glass carries enough thermal energy with it as it leaves the pool to need only a small amount of energy input to remain at 1050°C, the desired minimum temperature. The discharge heaters are sized to account for those periods when glass is not being poured and the glass in the discharge tubes must be kept above the liquidus (crystal-formation) temperature. Any pluggages which form in the discharge tubes would require replacement of the entire melter, since in the remote environment, no access is available to ream the tubes.

Another unique feature of the DWPF melter system is the motive system for starting and stopping glass flow from the melter into the product canister. A flange, connected to the melter pour spout with a metal bellows unit, will lower onto the nozzle of the product canister. Special "K" ring metal seals contained in a gasket-type insert create an adequately leak-tight seal. Through connections on the bellows housing, a vacuum is drawn on the product canister. By controlling the amount of vacuum relative to the plenum above the glass pool in the melter, glass can be drawn into the canister. Starting and stopping glass flow is done by adjustment of the relative vacuums of the canister and melter. When a canister is full, flow is stopped. After a cooling period of approximately 1/2 hour, the bellows flange is lifted, and a turntable will rotate the full canister away from and an empty canister under the pour spout.

The preceding description is intended only to acquaint the interested chemical engineer with certain functional design features of the DWPF melter. Details of mass and energy transport, reaction kinetics, material stresses, etc., are beyond our present scope. A few general observations are in order, however. The molten glass is quite viscous, ca. 50 poise. Boundary layers are large (several cm). Convection is slow (cm/hr) but necessary to homogenize the glass during its 64-hr residence time. Tracer tests with models and research scale melters indicate that glass product is 95% adjusted to a step change in feed composition after 3 volume throughputs. The feed pile is very complex, with high vertical gradients of temperature (>50°C/cm) and chemical and physical properties (from boiling water slurry on top, to a dry solid calcining interior, to semi-molten glass on the bottom, over a distance of about 15 cm). The plenum zone is likewise complex as different gases at different temperatures from different sources are mixed, heated, and reacted. Heat transfer through the melter walls can be characterized by very high temperature gradients in the refractory due to the water-cooled shell, complex geometry at the nonperpendicular intersection of two cylinders, periodic glass flow interruptions, and a significant long-term change due to refractory erosion.

Much theorectical and modelling work has been done on the DWPF melter concept (example, reference 5). On the other hand, this and other waste glass melters will provide challenges to chemical

engineers for years to come as performance is optimized and design improvements are made in replacement models.

OFF-GAS TREATMENT

One area which has required a great deal of development effort has been in handling of off-gases generated during the vitrification process. Ideally only gases, including air, water from the incoming feed stream, mercury, combustion products, oxygen from waste calcination, NO_{X} , SO_{X} , and halides will enter and be treated in the off-gas system. However, entrained feed solids, glass splatter, and some semi-volatile salt species also enter the off-gas system.

Initially the glass splatter presented the greatest difficulty since it would typically block the off-gas discharge port on the melter within several days of continuous operation. The pluggage, being of glass deposits, would require several hundred pounds of mechanical force to clear. Development of a reamer capable of this force, for use in a remote, crowded, and very hostile environment, was an undesireable option with a low probability of success.

Instead, a special port insert, an off-gas "film cooler", was developed to prevent this type of deposit from sticking to the walls of the exit pipe. The device injects an air/steam mixture (through circumferential slots in the pipe wall) parallel to the off-gas flow. The net effect of this boundary layer is that the glass splatter is cooled to the point at which the particles have little tendency to stick to the port. Some deposits still do

attach to the pipe walls, but a brush reamer with only a few pounds of pressure is quite capable of keeping the off-gas port open.

The injected gases also cool the bulk off-gas to <400°C, mitigating attack of the Ni-Cr-Fe alloy line by halogen and sulfur salts. The line is kept above the dew point of water, HCl, H₂SO₄, etc. Some salts do condense, including CsCl and Hg₂Cl₂, forming very fine (sub-micron) aerosols and agglomerates.

Entrained feed solids, cooled glass-splatter particles, and condensation aerosols are kept from depositing in the main off-gas line by maintaining the gas velocity above 1.5 m/sec. A water jet ejector quenches the gas stream to <60°C. Most of the large entrainment particles (>10 micron diameter) are removed from the off-gas with the jet ejector. Cesium (the most radioactive particle present) forms very fine aerosols, with diameters typically less than 1 micron. To avoid frequent change-out of filters, a knock-out system is required to reduce the cesium concentration in the off-gas stream. The key to removal of cesium is a collapsingwake steam jet atomized scrubber. Water is injected into an expanding supersonic steam jet producing small droplets which contact and capture particles in the off-gas stream. A small diameter pipe upstream of a centrifugal separator allows the droplets to agglomerate to many times their original size and facilitates simple centrifugal (cyclone) separation. Two atomized scrubbers in series reduce the cesium concentration in the off-gas stream by a factor of fifty. To reduce the cesium (radioactivity) in the final gas discharged to the atmosphere by an additional factor of 16 million, a wet filter, a dry filter, and a sand filter are all used.

YIELD

Overall, the process of vitrifying waste into a stable glass form is very efficient for most compounds with typically >95% yield of waste solids ending up in the glass. The balance consists of a decontaminated mercury byproduct, decontaminated gaseous releases, and recycled waste. Cesium, which forms semi-volatile species, has a typical glass yield of >90%. These values are all first-pass figures. The radioactivity that does not end up in the glass in the first pass, is recycled to the waste tanks. Thus the actual yield of stabilizing the radioactive wastes in glass is greater than 99.9%.

ACKNOWLEDGMENTS

This article represents the work of many people, over a period of several years. The authors would particularly like to acknowledge an organization and place known as "TNX", the chemical engineering semiworks of the Savannah River Laboratory. The information contained in this article was developed during the course of work under Contract No. DE-ACO9-76SR00001 with the U.S. Department of Energy.

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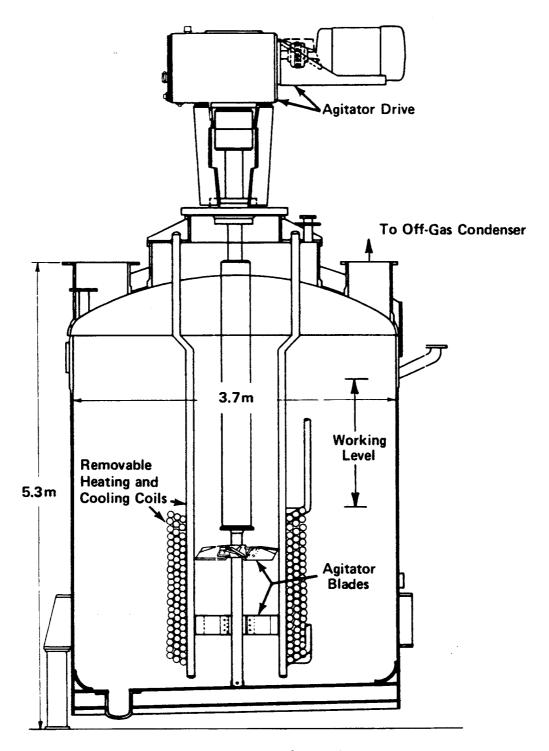
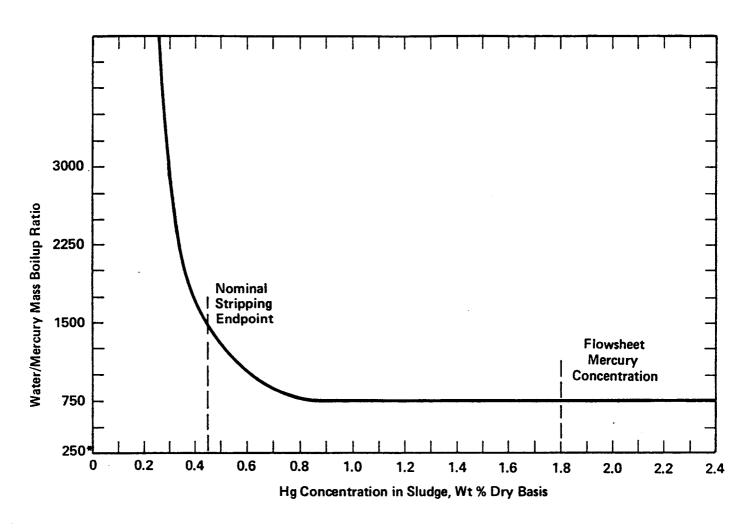


FIGURE 1. DWPF Slurry Processor



* Theoretical ratio based on vapor pressures at 100°C

FIGURE 2. Steam Stripping Efficiency vs.
Mercury Concentration

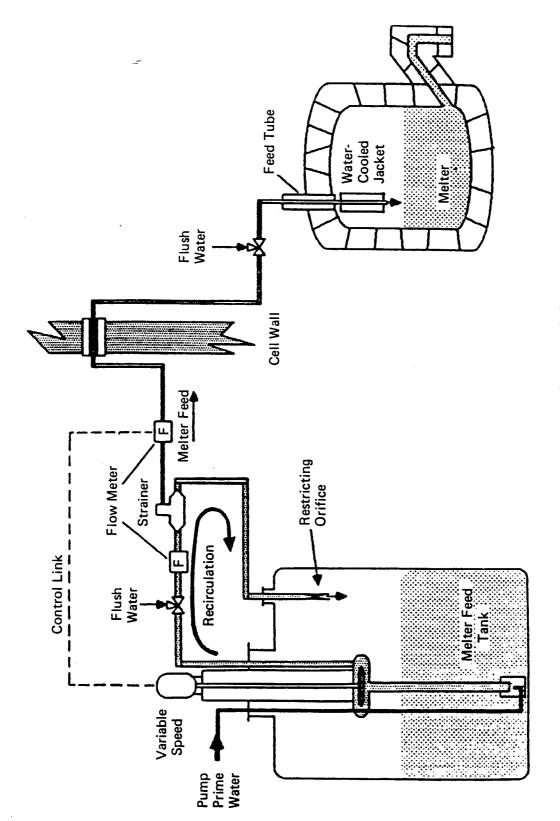


FIGURE 3. DWPF Melter Feed System

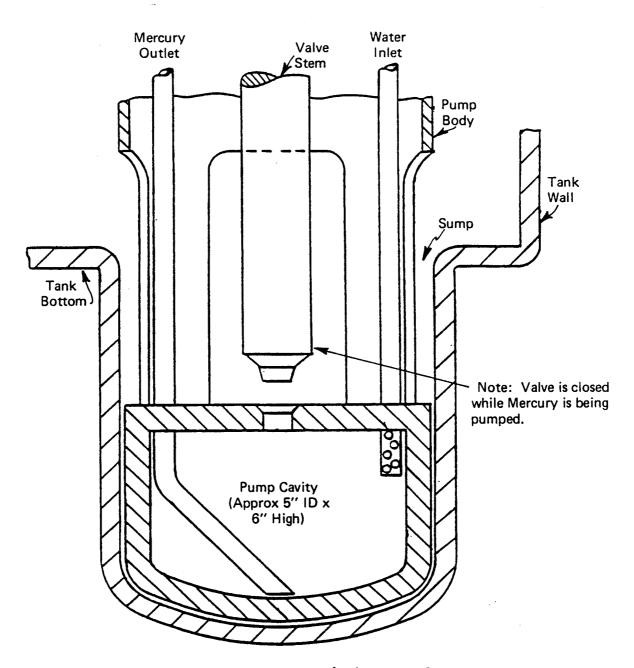


FIGURE 4. Mercury Displacement Pump

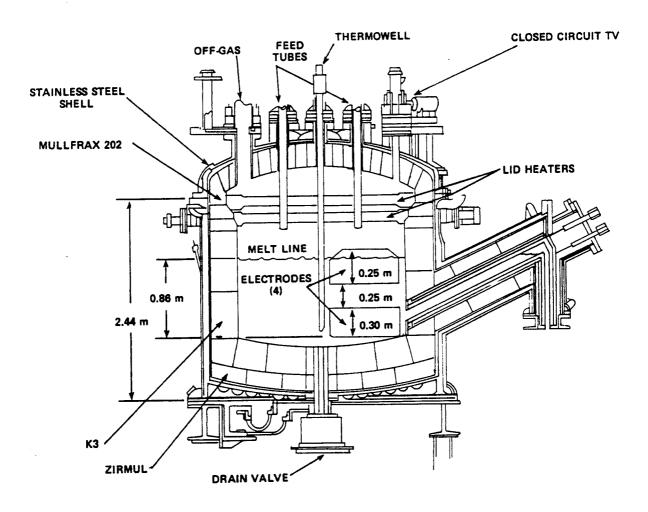


FIGURE 5. DWPF Melter

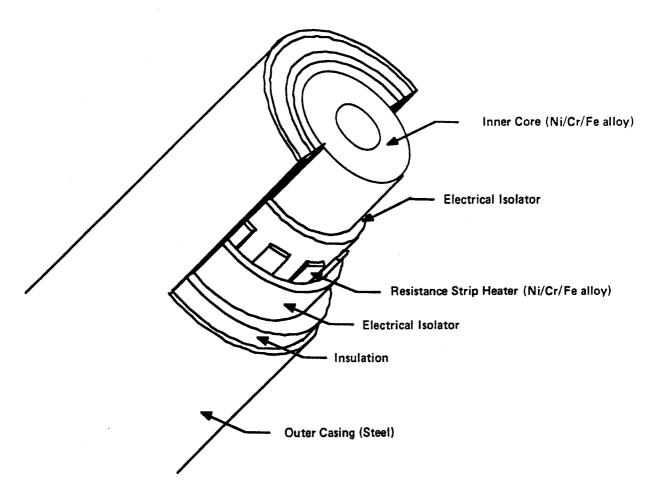


FIGURE 6. Riser and Pour Spout Heaters
Conceptual Cutaway View